The evaluation of the degradability of AddiFlex was done by Dr. I. Jakubowicz at the SP Swedish National Testing and Research Institute in Boras, Sweden. Dr. Jakubowicz's report about "Evaluation of degradability of AddiFlex" has been summarized below.

The results of the size exclusion chromatography measurements had shown that the material degrades fast at temperatures of 50° C, 60° C and 70° C. The rate of termooxidativ degradation was depending on temperature, oxygen content and the content of prooxidant in the material. Temperature seems to be the most important factor. The molecular weight values of both materials were under 5000 after 2 weeks at 70° C, and after 8 weeks at 60° C. The content of prooxidant was also important. The material containing 20 % prooxidant showed about 30 to 50 % lower molecular mass values then corresponding values of the material 10 % prooxidant. The oxygen content seemed to have the slightest influence. No significant influence was observed for AddiFlex with 10 % prooxidant and only little influence for AddiFlex with 20 % prooxidant.

According the EN 13432 Standard the germination rate and the biomass production had to reach at least 90 % of the control, to fulfil the requirements. The germination rates with wheat and mustard in the substrate mixtures A, B, C and D reached an amount between 101.1 % and 110.2 %, when compared with the substrate mixtures without test material (blank). The biomass production with wheat and mustard in the substrate mixtures A, B, C and D reached an amount between 97.6 % and 125.6 %, when compared with the substrate mixtures without test material (blank). That means, that the plant growth requirements according to the EN 13432 Standard have been fulfilled.

6 Remark

The results from the mineralization test, from the analysis of heavy metals, from the analysis of halogenated compounds and from the plant growth test were done at EMPA and were archived under the report number 414000. Beside these results, also the disintegration analysis, which was done by Dr. U. Galli in an industrial composting plant in Bellach, Switzerland and the evaluation of the degradability of these foils, which was done by Dr. I. Jakubowicz at the SP Swedish National Testing and Research Institute in Boras, Sweden, have been summarized in this report.

St. Gallen, 2002-02-07/KJP

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The mineralization in soil columns was tested. The mineralization of the materials were examined in microbial activated soil in soil columns at an incubation temperature of 60.0° C. The mineralization rate after 180 days of incubation was 57.9 % and 62 % respectively. After 200 days of incubation the mineralization rate of AddiFlex with 10 % prooxidant was 60.3 % and the mineralization rate of AddiFlex with 20 % prooxidant was 65.1 %.

According to the DIN-Pre-Standard 54900 (1998) (Testing the compostability of polymeric materials; Test no. 2 Evaluation of the complete biodegradability of polymeric materials in laboratory tests) and the ASTM Standard D 6400-99 (Standard specification for compostable plastics) a biodegradable material will fulfil the requirements, when at least 60 % of the carbon is mineralized to carbon dioxide and assimilated into biomass within 180 days.

This goal has been reached by the product AddiFlex with 20 % prooxidant after 180 days of incubation. The mineralization of AddiFlex with 10 % prooxidant was a little bit slower. After 180 days of incubation 57.9 % of the carbon were mineralized. A mineralization rate of < 60 % was reached after 200 days of incubation. However, after this incubation period the mineralization of both materials was still continuing. Therefore, it can predicted that the both materials can be completely mineralized to carbon dioxide.

The mineralization process is faster at higher temperatures. In composting plants the industrialised rotting process occurred at temperatures of 80° C. Therefore it can be estimated that under such optimal conditions the materials will degrade two times faster.

The disintegration test under real conditions was done by Dr. Galli in an industrial composting plant in Bellach, Switzerland. Dr. Galli's report about "Biodegradable AddiFlex modified PE-film degradation" has been summarized below.

According to the DIN-Pre-Standard 54900 and the EN 13432 Standard at least 90 % of the polymer fraction had to be disintegrated (residue in the sieve fraction of a 2 mm sieve) after 12 weeks of incubation. 5 kg of polymer foils (AddiFlex with 10 % and 20 % prooxidant) were cut into pieces of 100 x 200 mm and placed into the hot spot of the first 3 m of a windrow with freshly shredded green waste material. The amount of polymer foil corresponded to a portion of about 0.23 % of the total dry weight of the green waste material. After twelve weeks of incubation the amount of visible polymer film fractions was 0.0029 % of the total dry weight of the green waste material. That means that 99.99 % of the added polymer film materials were disintegrated.

The amount of heavy metals (lead, chrome, nickel, zinc, cadmium, copper and mercury) and the halogenated biphenyls (PCB) were analysed.

All analysed heavy metals were below the tolerated values of the DIN-Pre-Standard 54900.

All values of the congeners of polychlorinated biphenyls 28, 52, 101, 138, 153 and 180 were under the detection limit of 0.02 mg/kg dry weight.

The physical-chemical parameters had been measured and were in accordance to the values obtained in the past.

Plant growth test with mustard as test species

	Substrate mixture with 50 % soil without test material (blank)	Substrate mixture C with 50 % soil with test material (AddiFlex with 10 % prooxidant)	Substrate mixture D with 50 % soil with test material (AddiFlex with 20 % prooxidant)
Germination (amount)	83 / 95 / 87 88 ± 6.11	84 / 96 / 89 90 ± 6.03	92 / 99 / 98 96 ± 3.79
Germination rate (%)[*6]		102.3 %	109.1 %
Biomass (g)	22.01 / 25.79 / 27.66 25.15 ± 2.88	29.29 / 31.43 / 26.46 29.04 ± 2.49	28.22 / 31.95 / 33.93 31.37 ± 2.90
Biomass (%)[*6]		115.5 %	124.7 %

[*6] Calculated as per cent of the corresponding values obtained with the substrate mixture with soil without test material (blank) and substrate mixture with soil with test material.

5 Summary

The compostability of the polymer foil AddiFlex with 10 % and 20 % prooxidant had been tested according to the DIN-Pre-Standard 54900 (1998), respectively according the to the EN 13432 Standard.

According to the EN 13432 Standard each validated method, which is suitable to prove the compostability of a product is allowed to be used, whereas according the DIN-Pre-Standard 54900 the testing procedure has to follow the described methods.

Both Standards are describing methods in order to test the compostability of biodegradable polymers. The Standards are consisting of different tests. There is a chemical examination (analysis of heavy metals), a further test, which should prove the ultimate degradation of the polymers (mineralization), a disintegration test under real conditions in a composting plant and ecotoxicity test (plant growth test).

In both Standards the composition of the polymers has to be declared. A polymer should not contain any organic compounds, which are not biodegradable. Further the product should contain no toxic organic compounds such as chlorinated compounds like polychlorinated biphenyls (PCB).

Plant growth test with wheat as test species

	Substrate mixture with 50 % soil without test material (blank)	Substrate mixture C with 50 % soil with test material (Addi-Flex with 10 % prooxidant)	Substrate mixture D with 50 % soil with test material (Addi-Flex with 20 % prooxidant)
Germination (amount)	83 / 95 / 86 88 ± 6.25	84 / 96 / 89 90 ± 6.03	93 / 98 / 99 97 ± 3.22
Germination rate (%)[*6]		102.3 %	110.2 %
Biomass (g)	22.01 / 25.79 / 27.39 25.06 ± 2.77	29.23 / 31.43 / 26.46 29.04 ± 2.49	28.50 / 31.63 / 34.26 31.47 ± 2.88
Biomass (%)[*6]		115.9 %	125.6 %

[*6] Calculated as per cent of the corresponding values obtained with the substrate mixture with soil without test material (blank) and substrate mixture with soil with test material.

Plant growth test with mustard as test species

	Substrate mixture with 25 % soil without test material (blank)	Substrate mixture A with 25 % soil with test material (Addi-Flex with 10 % prooxidant)	Substrate mixture B with 25 % soil with test material (Addi-Flex with 20 % prooxidant)
Germination (amount)	91 / 91 / 95 92 ± 2.31	90 / 93 / 95 93 ± 2.52	100 / 98 / 99 99 ± 1.00
Germination rate (%)[*6]		101.1 %	107.6 %
Biomass (g)	22.79 / 29.32 / 23.15 25.08 ± 3.67	25.63 / 33.44 / 25.63 28.23 ± 4.51	23.54 / 28.12 / 21.79 24.48 ± 3.27
Biomass (%)[*6]		112.6 %	97.6 %

lecular weight values of both materials were under 5000 after 2 weeks at 70° C, and after 8 weeks at 60° C. The content of prooxidant was also important. The material containing 20 % prooxidant showed about 30 to 50 % lower molecular mass values then corresponding values of the material 10 % prooxidant. The oxygen content seemed to have the slightest influence. No significant influence was observed for AddiFlex with 10 % prooxidant and only little influence for AddiFlex with 20 % prooxidant.

4.6 Plant growth test

Ecotoxic effects on two higher plants (wheat and mustard) was determined by comparing soil from the soil columns, which contained no test material (blank) and soil, which contained test material. The plant growth test had been done according the European Standard EN 13432 (Packaging – Requirements for packaging recoverable through composting and biodegradation) and the Standard OECD 208 (Terrestrial plants, growth test).

Mixtures of plant substrate with 25 % and 50 % (v/v) of soil from the soil columns were prepared. The mixtures were filled in pots and 100 seeds of wheat and mustard were added on the top of the soil mixtures and lightly covered with the soil mixtures. The test was performed in three parallels for each mixture.

After three weeks of incubation in a glass house at an average temperature of 20° C the germination rate (number of grown plants) and the plant biomass of the soil mixtures (soil with test material and soil without test material (blank)) were evaluated and compared in all mixtures. Germination and biomass were calculated as per cent of the corresponding values obtained with the substrate mixture with soil, which contained no test material and substrate mixture with soil, which contained test material.

Plant growth test with wheat as test species

	Substrate mixture with 25 % soil without test material (blank)	Substrate mixture A with 25 % soil with test material (AddiFlex with 10 % prooxidant)	Substrate mixture B with 25 % soil with test material (AddiFlex with 20 % prooxidant)
Germination (amount)	91 / 90 / 95 92 ± 2.65	91 / 93 / 95 93 ± 2.00	99 / 98 / 99 99 ± 0.58
Germination rate (%)[*6]		101.1 %	107.6 %
Biomass (g)	22.79 / 29.02 / 23.15 24.99 ± 3.50	25.89 / 33.44 / 25.63 28.32 ± 4.44	23.54 / 28.12 / 21.99 24.55 ± 3.19
Biomass (%)[*6]		113.3 %	98.2 %

Physical-chemical parameters of the soil from the soil columns:

Parameter	AddiFlex with 10 % prooxidant	AddiFlex with 20 % prooxidant
Density [g/l][*3]	0.547	0.591
Total dry solids [%][*4]	39.8	38.7
Volatile solids [%][*5]	40.9	40.6
Conductivity mS/cm	9.41	9.30
pH	5.5	5.7
Nitrate [mg/l]	5000	5000
Nitrite [mg/l]	0	0
Ammonium [mg/l]	100	100
Potassium [mg/l]	450	450
Phosphate [mg/l]	25	25
Calcium [mg/l]	1150	1250
Magnesium [mg/l]	0	0

[*3] Fresh soil (soil, which was in the soil columns)

[*4] In the fresh soil (residue after drying the fresh soil for 6 hours at 100° C)

[*5] In the dry soil (weight loss after drying the dried soil for 6 hours at 650° C)

4.5 Evaluation of the degradability of AddiFlex

The evaluation of the degradability of AddiFlex was done by Dr. I. Jakubowicz at the SP Swedish National Testing and Research Institute in Boras, Sweden. Dr. Jakubowicz's report about "Evaluation of degradability of AddiFlex" has been summarized in this test report.

Polyolefins can be made oxobiodegradable by the use of prooxidant additives leading to chain scission and hydrophilic surface modification. These predegraded, low molecular mass oxidation products can be further mineralized by microorganisms.

At the SP Swedish National Testing and Research Institute in Boras, Sweden the thermooxidation of AddiFlex with 10 % and 20 % prooxidant was tested at three temperatures (50° C, 60° C and 70° C). Beside the temperature the oxygen content was also varied. The incubation was done in an air atmosphere and in an atmosphere of 5 % and 10 % oxygen.

The effects of the thermooxidation were evaluated by size exclusion chromatography (SEC) measurements. The number-average molecular weight (Mn) as well as the weight-average molecular weight (Mw) had been measured.

The results of the size exclusion chromatography measurements had shown that the material degrades fast at temperatures of 50° C, 60° C and 70° C. The rate of termooxidativ degradation was depending on temperature, oxygen content and the content of prooxidant in the material. Temperature seems to be the most important factor. The mo-

4.3.2 Analysis of halogenated compounds

The results of the analysis were in the table below.

Halogenated contaminants	Amount found in the polymer foil AddiFlex
Polychlorinated biphenyls (PCB)	The values of the congeners 28, 52, 101, 138, 153 and 180 were all under the detection limit of 0.02 mg/kg dry weight.

4.4 Quality test of the soil after the mineralization test

After the mineralization test the soil (from the soil columns), which contained 90 % plant substrate of peat basis and 10 % mature compost, the physical-chemical parameters had been determined.

The measurements were done according the following test procedures:

Easy soluble salts and nutrients such as nitrate, ammonium, phosphate, potassium, magnesium and calcium had been extracted according the procedure “Referenzmethoden der Eidg. Landwirtschaftlichen Forschungsanstalten, Volume 1; Bodenuntersuchung zur Düngeberatung; Volumenextraktion mit Wasser (1:2) Code H2OGH-Ex.

200 ml distilled water were transferred in a 500 ml glass bottle (Schott) and then soil from the soil columns were added until the water/soil-suspension reached a volume of 300 ml. Then the suspension was shaken on a shaker for 1 hour.

The pH of the soil suspension was measured with a pH electrode prior filtration. Then the suspension was filtrated through a paper filter.

The conductivity and the ions were measured in the clear extract. For the conductivity measurement a conductivity electrode was used. The measurement of the ions was done with half quantitative test methods, which were listed below.

The following tests had been used:

Nitrate, nitrite:	Merckoquant No. 1.10020
Ammonium:	Merckoquant No. 1.10024
Potassium	Merckoquant No. 1.10042
Phosphate	Merckoquant No. 1.10428
Calcium	Sigma No. 587-A
Magnesium	Aquamerck No. 1.11131

The volatile solids in the dried soil were measured after incineration at 650° C for 6 hours.

that was important to get pathogen free compost, the temperatures decreased and reached values of about 40° C after twelve weeks.

Polymer foils (AddiFlex with 10 % and 20 % prooxidant, 5 kg of each type) were cut into pieces of 100 x 200 mm and placed into the hot spot of the first 3 m of a windrow with freshly shredded green waste material. In order to facilitate the recovery of the specimens the polymer foils had not been shredded at the beginning. The amount of polymer foil corresponded to a portion of about 0.23 % of the total dry weight of the green waste material.

After 2, 6 and 12 weeks of incubation the following results were obtained:

There were no visual differences between the disintegration behaviour of the two polymer foils AddiFlex with 10 % and 20 % prooxidant.

After 2 weeks of disintegration the polymer films showed degradation due to their experienced brittleness. Larger pieces were still visible. After six weeks of incubation the visible polymer film samples became significantly smaller. After twelve weeks of incubation the amount of visible polymer film fractions was 0.0029 % of the total dry weight of the green waste material.

4.3 Analysis of heavy metals and halogenated compounds

4.3.1 Analysis of heavy metals

The amount of heavy metals was analysed and was listed in the table below.

Heavy metal	Amount found in the polymer foil AddiFlex [mg/kg]	Tolerated values [mg/kg] (DIN-Pre-Standard 54900)
Lead	< 2.3	30
Chrome	< 1.0	30
Nickel	< 0.5	15
Zinc	20 ± 1	100
Cadmium	< 0.1	0.3
Copper	< 2.0	22.5
Mercury	< 0.23	0.3

All analysed heavy metals in the polymer foil AddiFlex were below the tolerated values of the DIN-Pre-Standard 54900.

During an incubation period of 200 days 65 % of the carbon in the polymer material AddiFlex with 20 % prooxidant was transformed to carbon dioxide.

Incubation time (days)	Positive Standard (Glucose) % CO_2 production*[1]	AddiFlex with 20 % prooxidant % CO_2 production*[1]	AddiFlex with 20 % prooxidant % CO_2 production*[2]
0	0	0.0	0.0
6	50.4	6.2	7.7
13	68.2	10.3	12.8
22	72.2	15.1	18.9
32	74.2	18.5	23.1
43	77.3	22.0	27.5
54	79.5	25.3	31.6
62	80.1	26.7	33.3
81		30.7	38.3
95		33.7	42.1
109		37.0	46.2
123		39.5	49.3
137		42.0	52.4
150		45.0	56.2
165		47.1	58.9
180		49.6	62.0
200		52.1	65.1

*[1] % CO_2 production; in comparison to the theoretical amount

*[2] % CO_2 production; corrected values under the assumption that glucose is completely mineralized to carbon dioxide and biomass

Only the dissimilated carbon (produced CO_2) was measured, the assimilated carbon (produced biomass) was not measured.

4.2 Disintegration of the polymerfoil AddiFlex with 10 % and 20 % prooxidant under real conditions in a composting plant

The disintegration test under real conditions was done by Dr. U. Galli in an industrial composting plant in Bellach, Switzerland. Dr. Galli's report about "Biodegradable Addi-Flex modified PE-film degradation" has been summarized in this report.

In the composting plant in Bellach, municipal green waste is composted after shredding in open-air windrows. The windrows were turned three times per week. The duration of the process was lasting up to four months for compost destined for horticulture (screened at 20 mm) or potting mixes (screened at 10 mm). In the first four to six weeks temperatures of 65 to 75° C were reached in the centre of the windrows. After this heat phase

3 Validation

The mineralization test in the soil columns occurred according to our EMPA-Standard Procedures.

4 Results

4.1 Mineralization in the soil columns

During an incubation period of 200 days 60 % of the carbon in the polymer material AddiFlex with 10 % prooxidant was transformed to carbon dioxide.

Incubation time (days)	Positive Standard (Glucose) % CO_2 production*[1]	AddiFlex with 10 % prooxidant % CO_2 production*[1]	AddiFlex with 10 % prooxidant % CO_2 production*[2]
0	0	0.0	0.0
6	50.4	4.4	5.5
13	68.2	10.8	13.4
22	72.2	15.4	19.3
32	74.2	18.9	23.5
43	77.3	22.4	28.0
54	79.5	25.9	32.3
62	80.1	27.0	33.7
81		30.9	38.6
95		34.3	42.8
109		36.3	45.4
123		38.3	47.9
137		40.7	50.9
150		42.3	52.9
165		44.2	55.2
180		46.3	57.9
200		47.3	60.3

*[1] % CO_2 production; in comparison to the theoretical amount

*[2] % CO_2 production; corrected values under the assumption that glucose is completely mineralized to carbon dioxide and biomass

1 Order

Measuring the mineralization of two biodegradable polymer foils with the specification "AddiFlex" with 10 % and 20 % prooxidant respectively in soil columns.

2 Material and Methods

2.1 Test objects

We got approximately 25 grams biodegradable polymer foils with the specification "AddiFlex" with 10 % and 20 % prooxidant respectively. The polymer foils had been preaged in an oven at 70° C for 4 weeks. These preaged materials were used for the following mineralization test in our laboratory.

2.2 Mineralization in the soil columns

The mineralization of the biodegradable polymer foils occurred in soil columns at an incubation temperature of 60° C. The mineralization was monitored by measuring the produced carbon dioxide. A precise amount of the test materials were mixed with microbial activated soil, which itself contained very low amounts of easy degradable carbon compounds. This mixture was filled in glass columns. The microorganisms in the soil were degrading the organic component (test materials) and were producing new biomass. The soil columns were continuously aerated with oxygen. The gas at the outlet of the columns was transporting the carbon dioxide, produced by the microorganisms, out of the glass columns. The gas was then washed in an adsorbing solution. The produced carbon dioxide was trapped as sodium carbonate in a sodium hydroxide solution. Periodically an aliquot of the sodium hydroxide solution was taken and the carbonate content measured by titration.

The carbon contents of the test compounds were analysed before starting the mineralization test.

Once the carbon content of the test compound is known, the theoretical amount of carbon dioxide that could be produced out of the carbon in the test material can be estimated. The degree of mineralization of the test compound can be calculated out of the produced carbon dioxide.

2.3 Chemical analysis

The amount of different heavy metals and halogenated compounds (PHB) had been analysed.

2.4 Procedure

Mineralization test	from	2001-05-17	to	2001-12-03	(200 days)
Plant growth test	from	2001-12-14	to	2002-01-04	(21 days)

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Test report
Nr. 422809

Wir forschen und prüfen für Sie

Test Assignment:	Measuring the mineralization of two biodegradable polymer foils
Test Object:	Two biodegradable polymer foils with the specification "AddiFlex" with 10 % and 20 % prooxidant respectively
Client Ref.Nr.:	Mr. L. Johansson
Order dated of:	2000-08-17
Test Object received:	2001-05-07
Test performed:	2001-05-17 to 2002-01-04
Number of Pages:	12
Attachments:	-

St Gallen, February 07. 2002
Project Leader:

Leader section
Microbial ecology on
building materials

SWISS TESTING

STS 057
Microbial ecology on
building materials

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