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Testing the Performance and the Disintegration of Biodegradable Bags for the Collection of Organic Wastes

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Summary: The biodegradability of five different biodegradable garbage bags were analyzed according to the DIN-Standard draft 54'900 „Measurement of the compostability of polymers". The tests have to prove that a „biodegradable polymer" can be degraded under controlled composting conditions. Five different types of bags were tested. The bags were made from cornstarch, polycaprolactone and Kraft paper. To claim compostability the material has to biodegrade and to disintegrate in a composting system, to mineralize completely to carbon dioxide and water, and to fulfill several quality criteria such as a limited amount of heavy metals, no toxic organic compounds and no organic non-biodegradable additives. The analysis of the heavy metal content showed that the polymers themself contained very low amounts of heavy metals. However, the printing with green and blue colors with copper pigments was increasing the copper content in all products. The mineralization experiments showed that all five materials disintegrated during the rotting process in standardized compost and all five tested products also fulfilled the mineralization rate of 60 % within six months.

Standards for testing the compostability of biodegradable polymers

The compostability of biodegradable polymers can be tested according to the DIN-Standard draft 54'900, respectively according to the ISO/CD-Standard 15'986. 2.

According to the ISO/CD-Standard each validated method, which is suitable to prove the compostability of a product is allowed to be used, whereas according to the DIN-Standard draft the testing procedure has to follow the prescribed methods.

Both Standards are describing methods in order to test the compostability of biodegradable polymers. The tests should prove that a polymer could be biodegraded during a controlled composting process and that the quality of the produced compost has not altered, when biodegradable polymers had been mineralized.

These Standards are consisting of different tests. There is a chemical examination, a further test, which should prove the ultimate degradation of the polymers (mineralization), and a disintegration test under real conditions in a composting plant or under lab conditions.

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 a chemical analysis is required. The heavy metal content should not exceed certain values. Further the product

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should contain no toxic organic compounds such as chlorinated compounds like PCB and dioxins.

Chemical analysis

The concentration of heavy metals are not allowed to exceed certain values. The limited concentrations of heavy metals in the biodegradable polymers should prevent that additional toxic compounds are incorporated in the recycled product (compost).

Table 1: The following amounts of heavy metals are tolerated in the biodegradable polymers by the DIN-Standard draft 54'900 (1997) "Testing of the compostability of polymeric materials":

Heavy metal	Limited amount [mg/kg]	Heavy metal	Limited amount [mg/kg]
Lead	30	Nickel	15
Cadmium	0.3	Zinc	100
Chrome	30	Mercury	0.3
Copper	22.5		

Ultimate degradation

Procedure 1:

This procedure describes the biological degradation of polymers by aerobic living microorganisms in a liquid test system. The mineralization of a polymer is characterized in this procedure by the consumption of oxygen. An inoculated synthetic media in a closed respirometer is used. The produced carbon dioxide is trapped by a suitable absorber. The oxygen consumption can be estimated by measuring the pressure reduction in the closed respirometer.

Procedure 2:

This procedure describes the biological degradation of polymers by aerobic living microorganisms in a liquid test system. The mineralization of a polymer is characterized in this procedure by the production of carbon dioxide. The liquid media is aerated with carbon dioxide free air. The produced carbon dioxide can be trapped in a sodium hydroxide solution and then analyzed by a titration process.

Procedure 3:

The test compound is mixed with microbial activated soil, packed in glass columns and incubated under continuos aeration. The degradation rate is calculated according the organic carbon in the test compound. The carbon dioxide, which is produced during the biodegradation process out of the polymer, can be trapped in a sodium hydroxide solution. The amount of carbon dioxide can be calculated according to the amount of sodium carbonate.

The measurement of the mineralization of the polymers occurs not under optimal conditions, as it would happen under natural conditions. However, these procedures allow to distinguish between polymers that are biodegradable and polymers that aren't.

Disintegration

Procedure 1:

Testing the disintegration under laboratory conditions.

The rotting process takes part in big containers in incubation chambers, which can be heated up, to 60° C. The biodegradable polymers were mixed with shredded organic waste. The material has to be turned over once a week. And at these occasions the humidity and the pH-value had to be adjusted if necessary. After 6 and 12 weeks of incubation the samples were visually examined, and the disintegration rate estimated.
If ever possible temperature and oxygen content were measured continuously during the whole rotting process.

Procedure 2:

Testing the degradability under praxis conditions in a composting plant.

The test is made in a composting plant of low technical Standard. That means no aeration of the piles. The samples were mixed with shredded organic waste and transferred in net tissue sacks. The sacks, which contain the polymer material, were inspected at different times during the rotting process.

The material is disintegrated when over 90 % of the polymer samples had disappeared at the end of the rotting period. The material don't has to be completely mineralized at this time, however, it should be transformed to smaller particles, which are not anymore visible. The mineralization to carbon dioxide and water is examined in other test procedures (liquid medium or soil column test).

The ISO/CD-Standard 15'986.2 is asking for one of these degradation tests (laboratory scale or real conditions on a composting plant). The DIN-Standard draft 54'900 is asking for both examinations (disintegration under real conditions on a composting facility and under laboratory conditions) in order to fulfil the requirements.

Ecological tests and testing the compost quality

The ecological examinations are done at the end of the rotting process. A compost will pass the ecological test, if summer barley grown on a rotted compost, which contained at the beginning of the rotting process 10 % biodegradable polymer, will reach at least 90 % of the yield, which summer barley has reached on the same reference compost, which contained no biodegradable polymer.

Testing the performance and the disintegration of biodegradable bags for the collection of organic wastes

Introduction

In a project, where the performance and the disintegration of different types of biodegradable bags in high and low Standard composting plants were examined, our institute was responsible for the chemical analysis of heavy metals and the testing of the disintegration and mineralization of the biodegradable bags in the laboratory. The testing was done according to the DIN-Standard draft 54'900 (1997) "Testing of the compostability of polymeric materials". These testing procedures allow to evaluate the biodegradability of polymers under conditions of a controlled composting process.

Totally five different types of biodegradable bags made of biodegradable polymers were examined. Two materials were starch based products, two materials were polycaprolactone based products and the fifth material was made of paper and cellophane.

Results

Analysis of the heavy metals

The compost, produced from organic waste, will be recycled as substrate and fertiliser for plants. Therefore, the concentrations of heavy metals are not allowed to exceed certain values (Table 1).

The limited concentrations of heavy metals in the biodegradable polymers should prevent that additional toxic compounds are incorporated in the recycled product (compost). During the rotting process a substantial amount of the biodegradable polymer will be mineralized, so that the concentration of heavy metal will increase. Therefore, the limited values for certain heavy metals in the biodegradable polymers are very low. The concentrations are about three to four times lower than the tolerated values in the compost itself.

Table 2: The analysis of the five different bags made of biodegradable polymers has lead to the following results:

Heavy metal	Waste bag based on polycaprolactone [mg/kg]	Waste bag based on starch (without print) [mg/kg]	Waste bag based on polycaprolactone [mg/kg]
Lead	1.5 ± 0.3	< 1.0	< 1.1
Cadmium	< 0.2	< 0.2	< 0.2
Chrome	< 2.5	<2.0	< 2.2
Copper	**27 ± 13**	< 2.0	**61 ± 2**
Nickel	5.7 ± 1.4	< 4.0	< 5.0
Zinc	7.5 ± 2.2	< 4.0	< 5.0
Mercury	0.14 ± 0.10	< 0.08	0.13 ± 0.09

Heavy metal	Waste bag based on starch [mg/kg]	Waste bag based o paper and cellophane [mg/kg]
Lead	< 2.1	< 1.1
Cadmium	< 0.2	< 0.2
Chrome	< 2.1	2.5 ± 0.3
Copper	19 ± 9	11 ± 2
Nickel	< 4.0	< 5.0
Zinc	< 5.0	8.3 ± 0.5
Mercury	< 0.10	0.10 ± 0.01

The tolerated amount for the very toxic heavy metals such as cadmium and mercury was 0.3 mg/kg polymer. The content of cadmium resp. mercury was in all bags lower than this tolerated amount. All biodegradable bags with green color prints contained higher amounts of copper (Table 2). In two of the tested materials the copper concentration exceeded the tolerated amount.

Heavy metals can enter in the products by different ways. One of the reasons for a higher metal content in the endproducts is the use of already enriched raw materials. This is true for natural based and petrochemical based polymers. Plant materials can contaminate natural based polymers. It's important to cultivate such plants on soils, which didn't contain large amounts of heavy metals or other persisting agricultural chemicals. During the growth of the plants, such chemicals and heavy metals will accumulate in the plant cells. Contaminated soils are therefore not suitable for the production of raw material for biodegradable polymers.

But also the synthesis of the polymers is a source, where polymers can get contaminated by heavy metals. For example: A polymer can be contaminated by heavy metals, which are used as catalyst. And a third contamination source for contaminating the polymers with heavy metals is the printing with colors that are metal based.

Further analysis showed that the heavy metal concentration in the biodegradable polymers never exceeded the tolerated amounts, however, the analysis of the green prints showed that the green color was copper based. Depending upon the amount of green parts and the weight of the biodegradable bags the copper content could easily exceed the limited values. The producer of biodegradable bags should be aware, that not only the polymers itself must fulfil the DIN-Standard draft 54'900 but also the color for the prints. Green and blue copper-based colors should not be used for the printing biodegradable materials in future.

Disintegration in composting reactors

The materials that were tested for disintegration were mixed with fresh shredded garden waste and transferred into compost reactors. The disintegration procedure in our lab reactors was the same process that occurred in composting piles. The reactors were actively aerated during the rotting process.

The temperature course during the rotting process was observed and at certain times, after 2, 4, 8 and 12 weeks of incubation, the polymer samples were visually examined. The material should completely disintegrate during a rotting period in a standardized compost matrix, so that the biodegradable polymer should no longer be recognizable.

During the rotting process the biodegradable polymers in the compost matrix did not alter the temperature course. Therefore the materials should not contain any compounds that could alter the microbial degradation process. After two days of incubation the temperature reached over 60° C. The mesophilic phase with temperatures of over 60° C lasted for approximately eight days. During the further disintegration process the temperature was sinking at a rate of two to three degrees per day. After four weeks of rotting, the temperature was still 30° C.

During the cooling phase we observed smaller temperature elevation. Because different microbial populations were involved in the degradation of the organic materials, it could be assumed that the temperature elevation during the degradation period occurred, because a certain special microbial population was degrading a special type of carbon compound.

The disintegration of the polymers was depending on the type of polymer and from product itself. Both starch-based products and one of the polycaprolactone-based materials were already completely disintegrated after two weeks of incubation. The second polycaprolactone based polymer was after two weeks of incubation covered with mould, partly disintegrated but still present. After further two weeks of incubation also this material was completely disintegrated. The microbial attack of the paper and cellophane-based bag started only after an incubation period of four weeks. During the further rotting process the material was slowly further disintegrated, and after twelve weeks of incubation the product had nearly completely disappeared. Especially the outer bag made of paper was entirely disintegrated, whereas small parts of the inner bag made of cellophane were still visible.

Mineralization in soil columns and in an aquatic test system

Materials made from synthetic polymers or modified naturally occurring materials were only biodegradable, if their degradation by microorganism under aerobic conditions was leading to carbon dioxide, water, naturally occurring organic compounds and biomass. During the mineralization process the carbon in the biodegradable polymers is oxidized by microorganisms to carbon dioxide.

Mineralization in soil columns:

The degradation of the biodegradable compounds occurred in soil columns. The mineralization was monitored by measuring the produced carbon dioxide.

A precise amount of the biodegradable polymers was mixed with manure compost, which itself contains very low amounts of easily degradable carbon compounds and filled in glass columns. The soil microorganisms degrade the organic compound of the polymers 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 than washed in a sodium hydroxide solution. The produced carbon dioxide was trapped as sodium carbonate in the absorbing solution.

Once a week an aliquot of the sodium hydroxide solution was taken and the carbonate content measured by titration.

Mineralization in aquatic test systems:

The mineralization in an aquatic test system occurred in a similar way as the mineralization in the soil columns. The degradation of the polymer was monitored by measuring the produced carbon dioxide. The test is suitable for water-soluble and water insoluble compounds. A certain amount of test compound was added to the mineral salt solution of the test system. A compost extract, fresh sewage sludge or an adapted microbial population could be used as inoculum. The system was actively aerated. The gas at the outlet of the test system was transporting the carbon dioxide produced by the microorganisms out of the reactor bottles. The gas was than washed in a sodium hydroxide solution. The produced carbon dioxide was trapped as sodium carbonate in the absorbing solution.

Once a week an aliquot of the sodium hydroxide solution was taken and the carbonate content measured.

The carbon content of the test compounds was analyzed 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 that carbon can be estimated and the degree of mineralization of the test compound calculated out of the produced carbon dioxide in the mineralization tests.

In the mineralization tests both starch-based products were showing right at the beginning high degradation rates with carbon dioxide production. Already after seven days of incubation over 10 % of the carbon in the polymer was oxidized to carbon dioxide. After a further week already 25 % of the theoretical amount of carbon dioxide was produced. And after 4 weeks of incubation 44 % and 30 % respectively of the estimated carbon dioxide amount had been produced. During the further incubation the carbon dioxide production continued and reached after seven weeks about 50 % and after three months the produced carbon dioxide was over 60 % of the theoretical amount. The other part of the carbon must be assimilated into the biomass. During the degradation of easy degradable compounds such as starch or sugars a big part of the substrate carbon can be assimilated to biomass. We therefore can conclude that the mineralization of these compounds was terminated at this time.

According to the DIN-Standard draft 54'900 (1997) "Testing of the compostability of polymeric materials" a degradation rate of 60 % of the carbon of the test material within 180 days is sufficient to fulfil the Standard limits.

The transformation of polycaprolactone based polymers in the soil columns was slower as the one of the starch based polymers. The degradation was slow but continuous. For the transformation of 60 % of the carbon in the product to carbon dioxide and biomass an incubation period of 180 days was required.

In the aquatic test system with sewage sludge as inoculum the degradation was much faster. However, the two types of polymers degraded with different rates. After a lag time of one week, one polycaprolactone-based polymer showed a fast degradation and after five weeks of incubation over 60 % of the polymer carbon was oxidized to carbon dioxide. The mineralization of the second product was slower. The lag time lasted three weeks until disintegration occurred. Then the compound disintegrated with similar rates as the other polycaprolactone-based compound. And after 6 weeks of incubation over 60 % of the polymer carbon had been oxidized to carbon dioxide.

The mineralization of the bags made out of paper and cellophane in the soil columns was slow but continuos. Round 8 % of the theoretical amount of carbon dioxide were produced per week. After six weeks of incubation around 40 % of the carbon was mineralized and after an incubation time of six months 60 % of the estimated carbon dioxide was produced. The slower mineralization of this product in the soil columns occurred similar to the disintegration in the compost reactors.