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# Biodegradability and Compostability of Polymers—Test Methods and Criteria for Evaluation

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The treatment of solid waste in controlled composting facilities is an important possibility for reducing garbage. Natural and synthetic polymeric materials can be used for many purposes, for example, as packaging materials, where compostability is required. A prerequisite for official regulations and the decision as to which materials may be composted is investigations on their biodegradability and the quality of the compost produced. Several standardization groups at the ISO, CEN, and DIN are developing definitions, test methods, and classification systems for differentiating compostable from noncompostable materials. The concept which will be standardized and used in Germany is described in detail. It includes characterization of the test material, determination of the biodegradability using laboratory tests such as simple aquatic batch tests and a controlled aerobic composting test, investigation of the disintegration of the test material in industrial or bench-scale composting facilities, and finally, chemical and ecotoxicological analysis of the compost produced.

**KEY WORDS:** Compost; aerobic biodegradability; paper; poly-$\beta$-hydroxybutyrate/valerate; cellulose powder.

## INTRODUCTION

The treatment of solid waste in controlled composting facilities or anaerobic digesters is a valuable method for treating and recycling organic waste material [2, 17]. It is an important possibility for meeting, for example, the German legislative targets of making use of garbage as laid down in laws on waste treatment, management, and recycling. Another example is the composting of packagings, which has specifically been mentioned in German [9] and European [7] directives on packaging and packaging waste. Therefore typical input materials for composting plants are today, and will especially in future be, not only green waste and biowaste from households, but also products based on paper and biodegradable plastics [3, 8, 16, 21]. An example is the direct disposal of packaging materials with alimentary residues and the subsequent treatment in composting facilities.

A prerequisite for the decision as to which materials may be composted is investigations on their biodegradability and the quality of the compost produced. Unequivocal definitions, suitable test methods, and sensible evaluation criteria including limit values and practicable classification systems are important conditions for differentiating compostable from noncompostable materials [21]. The German Standardization Organization DIN as well as the European CEN (European Committee for Standardization) and recently also the ISO (International Standardization Organization) are drawing up concepts and selecting and developing methods, with the aim of obtaining standards for identifying biodegradable and compostable polymeric materials which can commonly be used and the results of which are accepted by all parties concerned.

The aim of this paper is to give an overview of the present activities to obtain information on the biodegradability and compostability of polymeric materials.

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Sufficient biodegradability is an important prerequisite but not the only requirement for deciding whether a material is compostable or anaerobically treatable. Suitable materials should have no negative effect on the biological treatment processes. The end product of this treatment, the compost, must meet the already existing strict criteria for compost quality. The strategy and the test methods presented here will be used in Germany and for packagings under European Union regulations.

## OFFICIAL REGULATIONS AND STANDARDS

In Germany the recycling and treatment of solid waste and garbage are regulated by laws on waste treatment [10] and on waste recycling and treatment [11]. A special technical guideline gives details on how to treat municipal waste [12] and another describes the quality and use of compost [13] and a federal law on composting is in preparation. Because of these regulations the biological treatment (composting or anaerobic digesting) of suitable materials is possible and will be more and more a requirement. Methods used to identify biodegradable and compostable polymers will be standardized in Germany in the near-future. This DIN standard, which includes the conception and individual test methods, is under development [6]; the principle steps of this conception are indicated in Fig. 1. The conception involves physical and chemical characterization of the material, e.g., indication of all ingredients, dry substance and voltatile substance, and amount of total organic carbon. Assessment of the complete biodegradability (mineralization) follows, using laboratory methods such as a modification of the $CO_2$ evolution test or the respirometric test and/or the controlled aerobic composting test. Disintegration of the polymeric material into forms to be used later, e.g., films of a certain thickness, has to be tested in pilot and/or industrial composting plants to confirm the total removal of the final products, e.g., films or formed articles. As in the previous laboratory steps the biodegradability of the material has been established, it is sufficient to determine, under technical conditions, the disintegration only. Finally, the quality of the compost produced is evaluated using analytical and ecotoxicological standard methods. This includes, e.g., analysis of heavy metals, total dry solids, growth inhibition test with higher plants, earthworm test, and daphnia test using an aquatic eluate.

Another example where the biological treatment of waste materials is possible is the field of packagings. To specify the possibility of biological treatment in the European packaging directive [7], which will be one of several possibilities for treating packaging waste, a working group of CEN concerned with the degradability of packagings (CEN TC261/SC4/WG2) has proposed definitions and will suggest a test concept for a European standard. This concept corresponds in principle to the DIN concept for polymeric materials [6], and the test methods included are also comparable. The CEN group has adopted a controlled aerobic composting test on a laboratory scale and tested in a ring test [18]. Future work will be directed toward drawing up an anaerobic composting test. The European directive and the European standards will have consequences for national regulations such as the German packaging directive [9] and for national standards.

The basis of the work to regulate the compostability of materials is expressed by definitions. Some im-

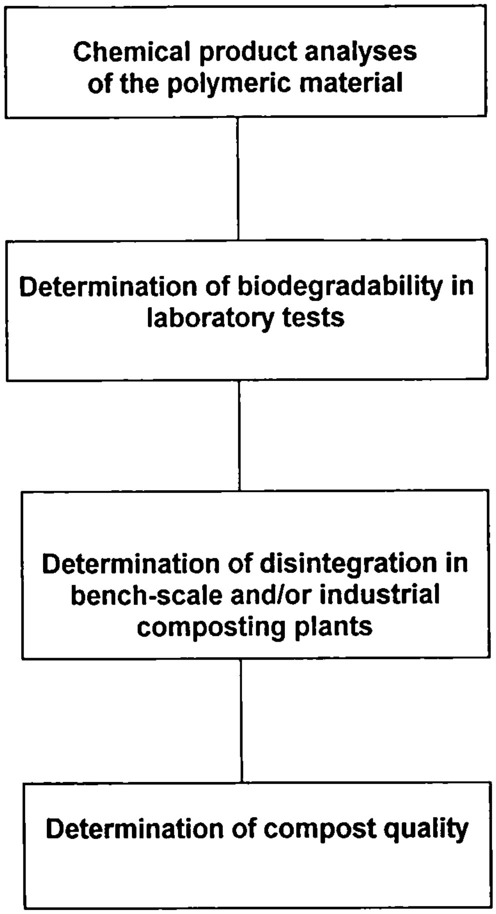


Fig. 1. DIN/CEN test concept to determine compostability.

Table I. Example of Definitions on Biodegradability and Compostability (CEN Definitions)

| | |
|---|---|
| Degradation | Degradation is an irreversible process leading to a significant change in the structure of a material, typically characterized by a loss of properties (e.g., integrity, molecular weight or structure, mechanical strength) and/or fragmentation. Degradation is affected by environmental conditions and proceeds over a period of time comprising one or more steps. |
| Degradable | A material is called degradable with respect to specific environmental conditions if it undergoes a degradation to a specific extent within a given time measured by specific standard test methods. |
| Biodegradation | Biodegradation is a degradation caused by biological activity, especially by enzymatic action, leading to a significant change in the chemical structure of a material. |
| Compost | Compost is an organic soil conditioner obtained by biodegradation of a mixture consisting principally of various vegetable residues, occasionally with other organic material and having a limited mineral content. |
| Compost quality | Compost quality is defined by the relevant national or EU standards. |
| Compostability | Compostability is a property of a packaging to be biodegraded in a composting process. To claim compostability it must have been demonstrated that a packaging can be biodegraded in a composting system as can be shown by standard methods. The end product must meet the relevant compost quality criteria. |

portant definitions suggested by the CEN working group are presented as an example in Table I. It is important to state in such a definition that biodegradability is an essential prerequisite for compostability and that complete biodegradability has to be proved but that other criteria such as the compost quality have to be fulfilled too.

Another international standardization group elaborating test methods for polymeric materials is ISO TC 61 "Plastics" [15]. Here, too, tests on the biodegradability of plastic material in aquatic, compost, and anaerobic environments will be standardized. It is very important to harmonize these different activities and strategies to come to the same test methods not only for producers of polymeric materials such as BASF but also for users of such materials, those running composting facilities, and authorities.

## TEST METHODS ON BIODEGRADABILITY

### Screening Tests

Official regulations, guidelines, and test methods are required to define unequivocally the compostability of a material. Companies such as BASF which develop biodegradable polymeric materials need, in addition, simple short-term tests to decide if a certain chemical structure has a degrading potential before biodegradability is confirmed and quantified in standard methods. Such screening tests do not confirm biodegradability, are legally uncertain, and will therefore not be used in standards or regulations. For the screening of biodegradable structures, we use unspecific screening tests such as enzymatic tests or determinations of the decay of thin films in a compost environment. In the first case a suitable test material is incubated with special enzymes such as lipases or esterases and the enzymatic degradation of a nonsoluble test material is followed by the production of soluble dissolved organic carbon (DOC) or a pH change, made visible by an indicator. In the second case materials can be tested from which thin films can be formed. These films are fixed in frames used for diapositive slides and incubated in compost, and the disintegration is followed by visual inspections. No differentiation between abiotic and biotic processes is, however, possible and no quantitative test results are obtained.

### Aquatic Batch Tests

In the DIN and CEN conception fairly simple aquatic batch tests are used in the first step to obtain information on the inherent and complete biodegradability of a test material. Such tests are modified versions of the known $CO_2$ evolution test (ISO 9439) [14], the manometric respirometry test (ISO 9408) [14], or the two-phase closed bottle test (ISO 10708) [14], which are not designed for the testing of polymeric material. These tests, which will also become ISO standards, are based on measurements of oxygen demand (ISO draft 14851) [15, 19] or carbon conversion (ISO draft 14852 [15, 20] and comparison with the theoretically expected values. Both techniques are unequivocal indicators of biodegradation processes. The disadvantage is that the percentage biodegradation in both cases does not include the amount of carbon converted to new cell biomass, which is not in turn metabolized to carbon dioxide during the test or requires no biochemical oxygen. Test results based on these analytical parameters and expressed as a percentage often have a gap between about 50 and 100%. Better information on complete biodegradation may be obtained with these methods by performing carbon balances if possible [19, 20]. This is normally not done when aquatic standard tests are used for biodegradation studies and needs much experience which is not yet sufficiently available. One prerequisite is, for example, the availability of precise biomass determinations. Other modifications of the original aquatic standard methods will improve the tests if used for screening the biodegradability of polymeric materials in composting environments. Examples are the use of suspensions of compost as inocula to introduce microorganisms from a compost environment into test vessels and the use of optimized inorganic media with a high buffering capacity and more nutrients to allow higher concentrations of the test compounds.

## Controlled Aerobic Composting Tests

All aquatic tests, however, do not represent the real conditions of a composting environment, in which fungi, molds, and actinomycetes are very active, microorganisms are fixed on the substrate, and relatively high temperatures (50–70°C) prevail. These conditions are, for many polymeric materials, important prerequisites for successful biodegradation in a biological treatment plant and are different from those in aquatic systems. For these reasons a controlled aerobic laboratory composting test is required. A method developed by De Baere *et al.* [4] and already available as ASTM Standard D 5338 [1] was modified, especially by introducing a constant temperature during the whole test period instead of a temperature profile (CEN TC261/SC4 N42). The test will become an ISO standard as well (ISO draft 14855) [15].

The test is based on the measurement of biogenetically produced carbon dioxide and, as additional information, the weight loss of the test material. A mixture of mature compost derived from a properly operating aerobic composting plant and the test material is introduced into closed vessels, for example, glass flasks with a volume of about 2–3 L, and incubated at 58°C under aeration and moisture control for up to about 45 days. In parallel, blank vessels with compost only and a known reference substance are investigated. Airflow carefully must be dosed or recorded and carbon dioxide measured in the exhaust air using suitable methods (e.g., infrared analyses, gas chromatography, or discontinuous determination by absorption in a sodium hydroxide solution and determination of the dissolved inorganic carbon). The amount of biologically produced carbon dioxide deriving from the degradation of the test material is compared to the theoretical maximum amount. Biodegradation curves are recorded as test results and the biodegradation percentage is calculated at the end of the test. As additional information, the weight loss of the test material and the disintegration of a compact material can also be determined at the end of the test.

The method was tested in a ring test by the CEN working group with the aim of making it a European standard. Test materials for the ring test were (1) poly-$\beta$-hydroxybutyrate, poly-$\beta$-hydroxyvalerate copolymers (Biopol) as a powder, (2) packaging paper in small pieces, and (3) microcrystalline cellulose powder (Avicel) as a reference material. A summary of the test results based on $CO_2$ evolution and expressed as a percentage of the theoretical is presented in Table II. Figure 2 is an example of the biodegradation curves. For more details on the test method, see Pagga *et al.* [18]. The results of the ring test and the experience gained by the participants showed that the method is suitable and can be performed without too many problems if the technical and analytical equipment is available. Preliminary comparisons with bench-scale and technical-scale composting tests as well as daily hands-on experience in monitoring, consulting, and operating full-scale composting plants have demonstrated that the laboratory-controlled aerobic composting test yields comparable results which can therefore be considered to be relevant and predictive [5].

**Table II.** Results of Controlled Aerobic Composting Tests (CEN Ring Test)

| | Cellulose powder (Avicel) | Poly-$\beta$-hydroxy butyrate/valerate copolymer (Biopol) | Packaging paper |
|---|---|---|---|
| Number of test results from 9 laboratories | 21 | 26 | 25 |
| Mean biodegradation degree, $CO_2$ of $ThCO_2$ (%) | 84 | 88 | 80 |
| Standard deviation | 9 | 13 | 9 |
| Range of 95% of all values | 66–102 | 61–115 | 61–99 |
| Test duration until the plateau phase was reached (days) | 30–40 | 20–30 | 30–40 |

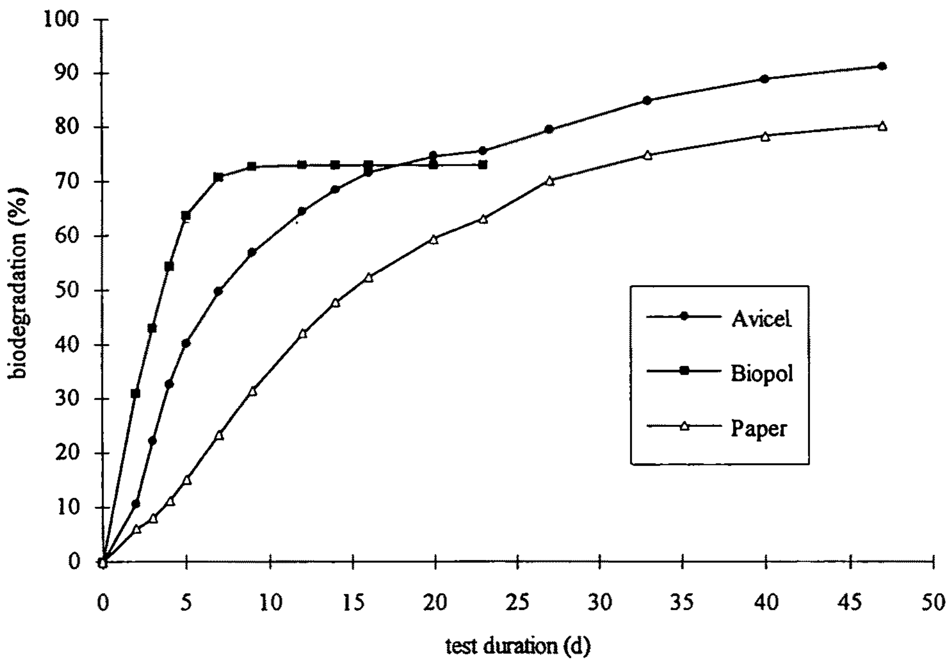


Fig. 2. Controlled aerobic composting test: examples of biodegradation curves.

## FURTHER INVESTIGATIONS AND TEST METHODS

For determining the disintegration of polymeric materials in semitechnical (bench-scale) or technical composting facilities, no standard method yet exists. Such guidelines and suggestions and methods for characterizing the polymeric material to be composted are being developed by the DIN and CEN working groups [6].

Chemical analyses of the compost produced have to be performed in Germany according to the German technical guideline on the quality and use of compost [13]. These investigations include, for example, water content, volatile substances, amount of salt, and amounts of the plant nutrients nitrogen, phosphorus, potassium, magnesium, and calcium. Organic and inorganic hazardous substances have to be checked, for example, polychlorinated biphenyls, dioxine, and heavy metals. In addition, ecotoxicological investigations are required to prevent toxic effects during the composting process and when the produced compost is used. Mixtures of soil and compost are tested for growth inhibition with higher plants or toxicity tests with earthworms. Aquatic eluates are tested, for example, with daphnia. More experience is necessary, however, before the most suitable methods can be suggested or limit values established.

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