BIODEGRADABILITY TESTING FOR SUSTAINABLE ADHESIVE DEVELOPMENT

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Abstract

Sustainability has been a major driver for consumer behavior and product innovation globally. There is a growing interest in biodegradable adhesives that are designed to break down naturally over time, reducing their environmental impact. Biodegradability is a key requirement for biodegradable and compostable products. In this talk, we will discuss key testing methods for biodegradability and their applications in support of sustainable adhesive development.

Background

Sustainability in packaging has become a vital focus in efforts to reduce the environmental impact of waste. Sustainable packaging involves using materials that are recyclable, renewable, or biodegradable, aiming to lessen the ecological footprint compared to traditional packaging. This is increasingly important as packaging waste significantly contributes to pollution, often persisting in landfills and oceans for centuries, harming ecosystems and wildlife. To reverse this trend, many countries now mandate packaging to be biodegradable or compostable, especially in the area where recycling is difficult to do. Some of these requirements are summarized in **Table 1**.

Table 1. Biodegradability Requirements for Various Product Type

Product Type	**Key requirement**	**Standards/Certification** [1-4]
Food Packaging	Compostability	ASTM D6400, EN 13432, OK compost Home/Industrial, BPI Certification (US),
Adhesives	Compostability	ASTM D6400 (compostable), TÜV AU 203
Disposable Food Service Ware	Industrial compostability	BPI Certification (US), Seedling Logo (EU)
Personal Care Product	Rapid biodegradation (often ≥60% within 28 days)	OECD 301, ISO 14851/14852, Ecocert, COSMOS

Biodegradability testing methods and standards are critical for assessing how materials break down in various environments, ensuring they meet environmental safety and sustainability goals. Below is an overview of the key testing methods, standards, and insights from recent literature, focusing on their principles, applications, and limitations.

Key Biodegradability Testing Methods

Biodegradability testing typically measures the extent to which microorganisms can decompose a material into simpler compounds like carbon dioxide (CO_2), water, and biomass. Common methods include:

1.Aerobic Respirometry: Measures oxygen consumption or CO_2 production as microorganisms degrade a material under aerobic (oxygen-rich) conditions.

Examples:

- OECD 301B (CO_2 Evolution Test): Assesses "ready biodegradability" over 28 days by measuring CO_2 output. A material is deemed readily biodegradable if 60% of its organic carbon converts to CO_2 within a 10-day window.
- ISO 14855: Evaluates aerobic biodegradation under controlled composting conditions at 58°C, requiring 90% biodegradation within 6 months for compostability claims.

Application: Widely used for liquids, plastics, and packaging in lab-simulated environments like compost or wastewater.

2.Anaerobic Digestion Tests: Measures biogas (methane and CO_2) production under oxygen-free conditions, simulating landfills or anaerobic digesters.
Examples:

- ASTM D5511: Tests biodegradation in high-solids anaerobic conditions (e.g., bioreactor landfills) over 45 days.
- ISO 15985: Assesses anaerobic biodegradation in aqueous systems by biogas measurement.

Application: Relevant for materials expected to degrade in landfills or sludge digesters.

3.Soil Burial Tests : Monitors weight loss, physical changes, or CO_2 evolution when materials are buried in soil.
Examples:

- ASTM D5988: Evaluates aerobic biodegradation in soil, with no specific threshold for certification.
- ISO 17556: Measures oxygen demand or CO_2 evolved in soil over extended periods.

Application: Used for agricultural films or products likely to end up in natural soil environments.

4. Marine Environment Tests : Assesses biodegradation in seawater, often at the sediment interface, using oxygen demand or CO_2 production.
Examples:

- ISO 19679: Tests aerobic biodegradation in marine sediment under lab conditions.
- ASTM D6691: Measures biodegradation of plastics in marine environments.

Application: Critical for materials like fishing gear or packaging that may pollute oceans.

Major Standards

Several organizations have developed standardized protocols to ensure consistency and reliability in biodegradability testing:
OECD (Organization for Economic Cooperation and Development):

- Focuses on "ready" and "inherent" biodegradability (e.g., OECD 301 series, OECD 302B). These are tiered tests used in regulatory frameworks like REACH in Europe, emphasizing aquatic environments.

ISO (International Organization for Standardization):

- Covers diverse environments (e.g., ISO 14855 for composting, ISO 17556 for soil, ISO 19679 for marine). ISO standards often align with certification requirements like EN 13432 for compostable packaging in Europe.

ASTM (American Society for Testing and Materials):

- Includes specific methods for plastics (e.g., ASTM D6400 for compostability, ASTM D5338 for composting conditions). ASTM D6400 requires 90%

disintegration and biodegradation within 180 days under industrial composting conditions.

EN (European Norms):

- EN 13432: A widely recognized standard for compostable packaging, requiring 90% biodegradation within 6 months, 90% disintegration within 12 weeks, and no ecotoxicity in the resulting compost.

Traditional methods can take months or years, particularly for slowly degrading materials, which hinders research and development (R&D) timelines. Accelerated testing methods address this by simulating conditions that speed up degradation, enabling faster decision-making and certification. This is especially relevant given increasing global environmental regulations, such as the EU's focus on biodegradable agricultural polymers and the U.S. EPA guidelines for landfill conditions.

Sewerage Sludge in house test to assess biodegradability

A key challenge in achieving fully sustainable packaging is the adhesives, particularly in multi-layer packaging. Conventional adhesives, typically made from synthetic polymers, are not biodegradable, causing packaging to remain intact long after its intended use. This has driven the need to develop biodegradable adhesives that allow packaging to decompose naturally, reducing its environmental persistence. These adhesives must match the performance of traditional options while being cost-effective and scalable for industrial use. Advancing biodegradable adhesives is a critical step toward a circular economy and mitigating the global packaging waste crisis.

As Henkel pursued the development of compostable adhesive, we needed in house test methods that can help screen biodegradability of raw materials and prototype formulation quickly and reliably. Among a suite of biodegradability testing methods established, the most robust and "adhesive friendly" method we used for adhesive development is based on ISO 14852 using sewerage sludge digestion.

Following lab procedure was adapted from ISO 14852 standard [1]. As shown in Figure 1, the inlet air is first stripped off CO_2 using 10M KOH solution. The CO_2 free air is injected to the test bottle (2L) filled with DI water, buffer solutions and three inorganic nutrient solutions. This is necessary to keep the pH constant in the system during the test, even at high concentrations of the test material. The medium contains about 2400 mg/l of phosphorus and 50 mg/l of nitrogen and is therefore suitable for concentrations in the test material of up to 2 000 mg/l of organic carbon.

Procedure:

1. Fresh activated sludge is obtained from a local sewage-treatment plant, decanted, and added to the test medium at 2.5% (v/v).
2. Test samples (liquid, particles, or films) are introduced with a total organic carbon (TOC) of 250 mg.
3. Sodium benzoate serves as the reference material.
4. Bland sample is included to capture background CO_2 evolution.
5. Evolved CO_2 is absorbed by NaOH and measured via titration or TOC analysis.

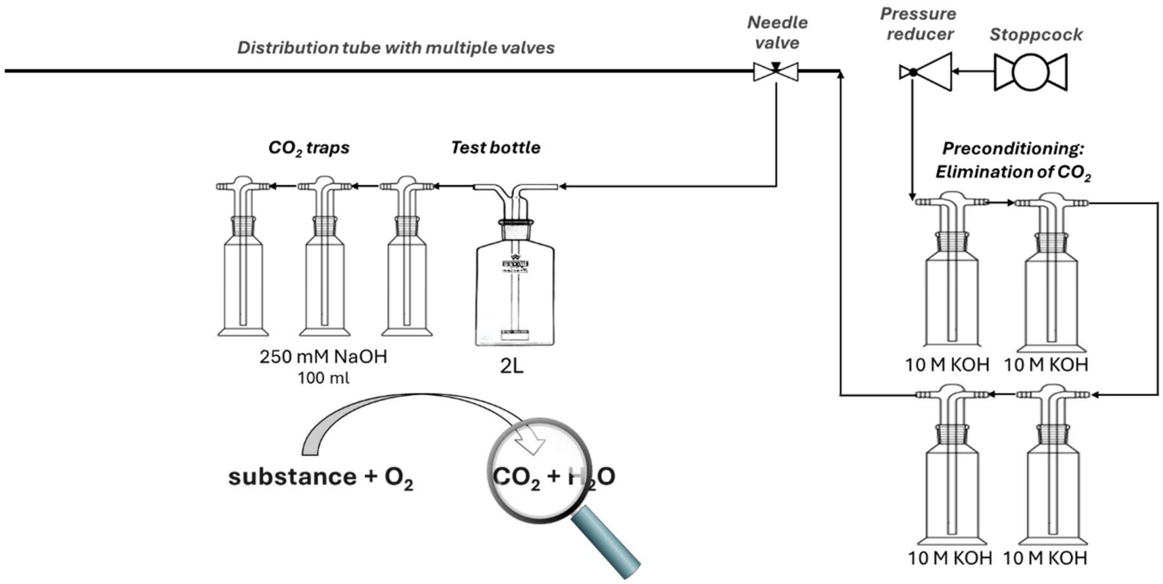

Figure 1. ISO 14852 sewerage sludge biodegradability test based on CO_2 evolution

The degree of biodegradation can be calculated by

$$D_t = \frac{\sum(CO_2)_T - \sum(CO_2)_B}{ThCO_2} \times 100$$

where

$\sum(CO_2)_T$	is the amount of carbon dioxide evolved in flask F_T between the start of the test and time t, expressed in milligrams;
$\sum(CO_2)_B$	is the amount of carbon dioxide evolved in the blank flask F_B between the start of the test and time t, expressed in milligrams;
$ThCO_2$	is the theoretical amount of carbon dioxide evolved by the test material, expressed in milligrams.

This method provides biodegradability data of many raw materials and adhesive formulations in development quickly and help us to select the better candidate. As shown in Figure 2, adhesive 1 has superior biodegradability than adhesive 2 after 49 days of measurement. The test method is very robust to accommodate broad selection of test objects, from liquid adhesive, hot melt coatings, to various constructions (PSA labels, paper straws, cold seal and laminated films).

Another important use of the in-house sewerage sludge biodegradability test is to predict the biodegradability in soil that is required by compostable certification scheme. Typical home composability certification requires test material to pass >90% biodegradability in soil within 6 (industrial) or 12 months (home). These tests take long time and costly. As we accumulate more in house sewerage biodegradability data along with in soil test data, we can build rough correlations between results obtained by two test methods. As indicated in Table 2, we use 60% ISO 14852 as cut off criterion to prescreen formulas that is designed for compostable certification.

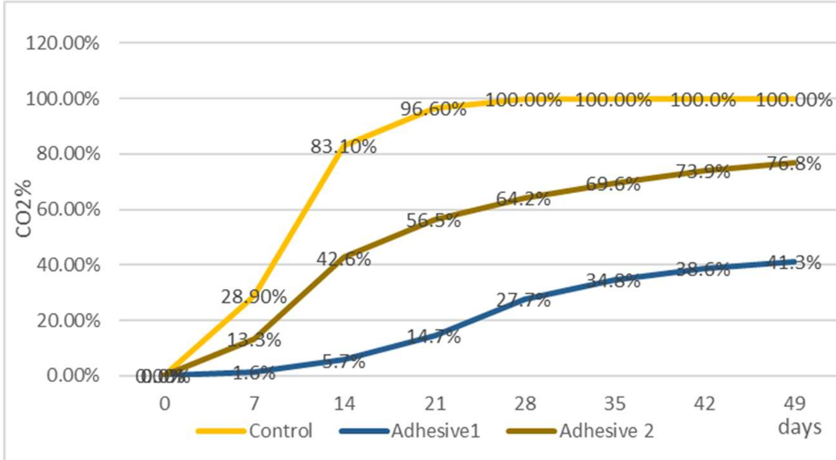

Figure 2. Biodegradability of adhesives measured by in house ISO 14852

Table 2. Comparison of Biodegradability Data: Sewerage sludge method vs. in Soil method

Biodegradability %	Raw material A	Raw Material B	Coated PSA label
ISO 14852 (56-64 days)	30%	50%	65%
EN13432 (180-360 days)	60-65%	90%	90%

Future work

We are establishing soil biodegradability testing based on ASTM D5988 in our sustainability testing laboratory in Henkel Bridgewater Innovation Center. Although the sewerage sludge test provides rapid results, compostable certifications still require biodegradability measured in soil.

Currently, we are focusing on running standard reference materials in soil from various locations/microbial communities, variations in organic material and composting conditions (e.g., temperature, C/N ratio, moisture, organic components). This work will help us better understand test variations and ways to improve standard and its utility in predicting compost behaviors in real life.

Literature

1. ISO 14852-2018.
2. ASTM D5988
3. EN13432
4. BPI industrial compostability certification scheme

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