

## Review

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# Certifications and testing methods for biodegradable plastics

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**Abstract:** This paper offers a comprehensive review of previous studies and articles on international standards and certification criteria for biodegradable plastics. It highlights key insights into the biodegradation environment and certification processes for these materials. As various countries and organizations intensify research efforts on biodegradable plastics, certification standards for biodegradability are evolving and expanding. This trend is expected to play a pivotal role in shaping international standards. Nonetheless, several challenges persist, including the absence of universally recognized testing methods, inconsistencies between real-world and laboratory biodegradation conditions, and a lack of clear definitions and standardized criteria. Above all, establishing international standards is critical to advancing biodegradable plastics as a viable alternative to conventional plastics.

**Keywords:** biodegradable; bioplastic; degradability; testing method; pollution prevention

## 1 Introduction

Plastics, such as disposable items and packaging materials, are closely intertwined with our daily lives. Plastics possess a variety of physicochemical properties, including high

strength, toughness, excellent rigidity, insulation, and lightness (Ilyas et al. 2018; Nayanathara Thathsarani Pilapitiya and Ratnayake 2024; Pan et al. 2020). Additionally, they are cost effective and easy to mold and process (Jiang et al. 2018; Liu et al. 2015). For these reasons, plastics are used across a wide range of industries, including agriculture, manufacturing, and healthcare (Gautam et al. 2007; Nayanathara Thathsarani Pilapitiya and Ratnayake 2024; Pan et al. 2020). Worldwide, the annual consumption of plastics has increased to more than 300 million tons and is expected to reach 500 million tons (Mellinas et al. 2016; Mohamed et al. 2020; Thakur et al. 2018). However, as consumption increases, conventional plastics pose serious environmental challenges because of their nonrenewable nature, unsustainable usage, and inability to biodegrade naturally. Research into biodegradable plastics as alternatives to conventional plastics began in the 1800s when Alexander Parks established the first artificial plastic based on cellulose. As oil prices increased in the 1970s and dependence on oil increased, research began in earnest, and environmental pollution in the 21st century became a major social issue; degradable plastics have become a notable international research direction and have become increasingly popular. There have been a series of reports on the preparation, characterization, performance detection, and degradability testing of degradable plastics (Atef et al. 2014; Giannakas et al. 2016; Liu et al. 2018; Shujun et al. 2006; Xie et al. 2020).

Plastic is a polymer compound formed by monomer polymerization (Chamas et al. 2020; Ghosh and Jones 2021; MacLeod et al. 2021). The long chain of carbon molecules in its structure is very firm and not easy to break, which is the main reason why ordinary plastic is difficult to decompose (Abdelmoez et al. 2021; Yashchuk et al. 2012). Degradable plastics can degrade more quickly than ordinary plastics, but under less demanding conditions and with lower requirements (Emadian et al. 2017; Haider et al. 2019; Karan et al. 2019). The principle behind biodegradable plastics is to make it easier for long chains of carbon molecules to break apart, facilitating the breakdown of polymers into smaller pieces, which can then be broken down into carbon dioxide and water, causing less harm to the environment (Chen et al. 2021; Shen et al. 2019). If biodegradability tests yield results

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that meet the required standards, certificates are issued for the corresponding materials and products. These tests are conducted across various environments (industrial composting, soil, marine, and aqueous systems) following international standards and guidelines to ensure the biodegradability of plastics. This underscores the critical importance of international standards and guidelines in the continued development of biodegradable plastics. This paper focuses on the types of biodegradable plastics and their certification criteria and discusses potential improvements through an expanded review of these standards.

## 2 Types of biodegradable plastics

Biodegradation refers to the process of organic matter being eroded, destroyed, and absorbed by microorganisms (Faris et al. 2014; Siddiqui et al. 2022). Biodegradable plastics can be degraded by enzymes released by microorganisms in the environment, such as bacteria, fungi and algae, as well as by natural degradation reactions under natural conditions, such as natural hydrolysis and natural oxidation (Faris et al. 2014; Kyrikou and Briassoulis 2007; Menossi et al. 2021; Siddiqui et al. 2022).

Biodegradable plastics are polymers that have properties that meet the requirements of selected applications and can be completely or partially degraded into environmentally harmless substances, such as carbon dioxide, methane, water, inorganic compounds, and biomass, under certain conditions (Adamcová and Vavrková 2014; Daglen and Tyler 2010; Markowicz and Szymańska-Pulikowska 2019). Biodegradable plastics are a source of carbon and energy for microorganisms; in the presence of oxygen, biodegradation reactions can be expressed by equation (1), and in the absence of oxygen, they can be expressed by equation (2), (Jayasekara et al. 2005; Leja and Lewandowicz 2010; Niranjana and Prashantha 2018; Shen et al. 2019):

$$CT = CR + CB + CO_2 \quad (1)$$

$$CT = CR + CH_4 + CB + CO_2 \quad (2)$$

Where CT is the total carbon content of the degradable plastics, CR is a residue of plastic or a byproduct generated in the process of decomposition, and CB is the biomass produced by microorganisms through reproduction and growth.

Generally, the biodegradation of biodegradable plastics involves three steps (Chen et al. 2021): (1) Microorganisms adhere to the surface of plastics and produce water-soluble intermediate degradation products. Adherence and degradation products are related to plastic properties (such as

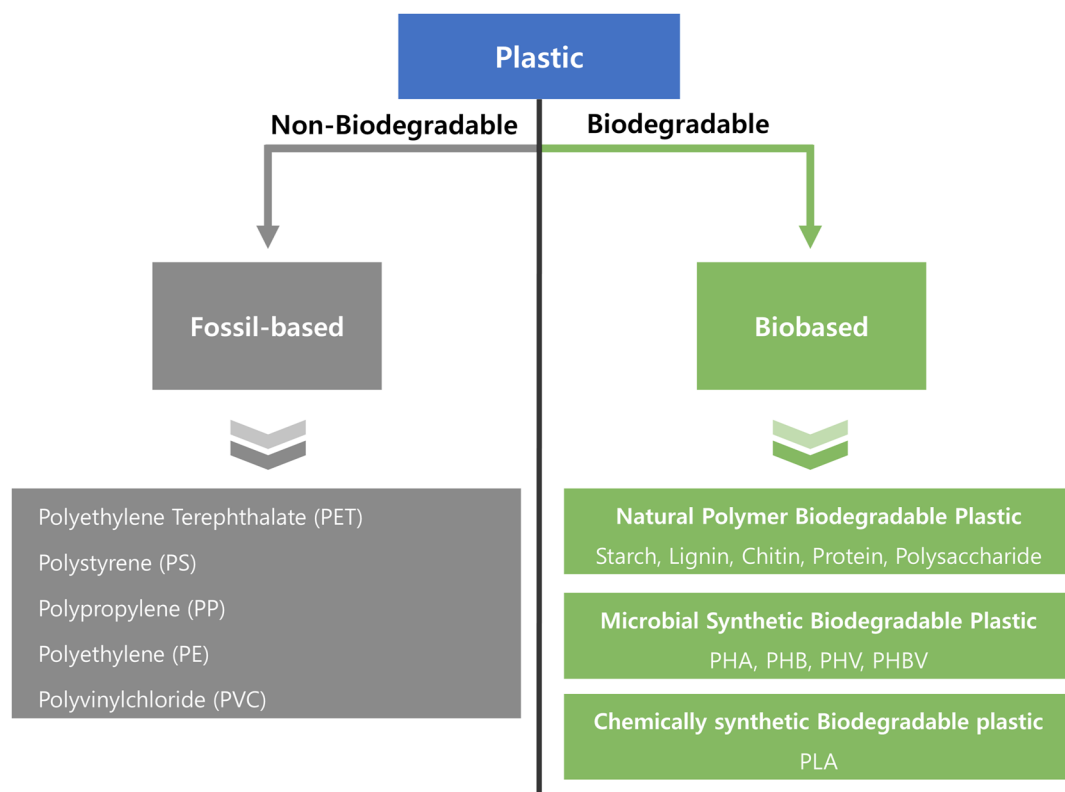
surface roughness, crystallinity, molecular weight, and functional groups), microbial species, and external conditions (such as temperature and humidity). (2) Microorganisms secrete specific enzymes (such as extracellular enzymes and intracellular enzymes), adsorb on the surface of polymer materials, digest polymer chains, and then degrade the polymer materials into fragments of oligomers, dimers, and monomers with low quantum fractions through various reactions, such as hydrolysis and oxidation. (3) These low-molecular-weight monomers enter the cell membrane of microorganisms, are absorbed and utilized by the microorganisms, are eventually converted into carbon dioxide, water, methane, etc., and are subsequently released. Biodegradable plastics have been widely studied in recent years, and various types of products, such as garbage bags, plastic bags, compost bags, and agricultural mulch films, have gradually begun to be commercialized (Faris et al. 2014; Siddiqui et al. 2022). Biodegradable plastics can be divided into three main categories: natural polymers, microbial synthetic polymers, and chemically synthetic polymers (Arikan and Bilgen 2019; Ashok et al. 2018; Rao et al. 2022). The three categories of biodegradable plastics described above are depicted in Figure 1.

### 2.1 Natural polymers

Natural polymers, such as starch (Bates et al. 1977), cellulose (Crépy et al. 2009), lignin (Wang et al. 2016), chitin (López-Ibáñez and Beiras 2022; Pillai et al. 2009; Sambasivan et al. 2009), protein (Swain et al. 2004), and polysaccharide (Behera et al. 2022; Wang et al. 2003) polymers, are abundant inexpensive, fully degradable, safe to use, nontoxic, and widely applicable. However, natural polymer materials still have several disadvantages. For example, they are easily corroded by microorganisms in the soil, and their plastic properties cannot meet the standard of use. It is difficult to combine the degradability of bioplastics with suitable plastic properties. In addition, relying entirely on natural materials to synthesize materials with suitable plastic performance is relatively difficult.

### 2.2 Microbial synthetic polymers

Microbial synthetic polymers are polymer materials produced by microorganisms through the fermentation of various carbon sources and have been studied mainly for microbial polyester production (Volkenburgh and White 1993). The product is characterized by complete biodegradability. Microbial synthetic polymer materials are easy to



**Figure 1:** Classification of fossil fuel-based plastics and biodegradable plastics.

process and form, but they need to be improved in terms of heat resistance and film formation and are expensive; therefore, they are mainly used in human implant materials or drug release applications. Some common examples of microbial synthetic polymers include PHA, PHB, PHV, and the copolymer of PHB and PHV known as PHBV (Bátori et al. 2018). Among them, polyhydroxybutyrate (PHB) and polyhydroxyalkanoate (PHA) are the bioplastics most commonly produced by microorganisms. PHAs are classified into three main types on the basis of the length of their carbon chains: short-chain length PHAs, medium-chain length PHAs, and long-chain length PHAs. Short-chain length PHAs typically contain three to five carbon monomers, medium-chain length PHAs contain 6 to 14 carbon monomers, and long-chain length PHAs contain more than 14 carbon monomers (Raza et al. 2018).

The properties of the PHA depend on the length of the carbon chain. The short carbon chain length included in the PHA increases thermoplasticity and increases flexibility and elasticity as the carbon chain length increases. However, they exhibit lower crystallinity and reduced mechanical properties (Li et al. 2016). PHAs are soluble in chloroform and other chlorinated solvents, but they are insoluble in water. These biodegradable materials can decompose in various environments, including marine, soil, home, and

industrial settings, making them highly regarded as among the most biodegradable plastics available. However, compared with other biodegradable plastics, PHAs have certain limitations restricting their widespread use because of their higher production costs and weaker properties. As they are produced through the cultivation and fermentation of microorganisms, their commercial application is still under active research and development.

## 2.3 Chemically synthesized polymers

Compared with natural polymers and microbial synthetic polymers, chemically synthesized polymers have more advantages. On the one hand, chemical methods can be applied to synthesize biodegradable polymers that are similar in structure to natural polymers; on the other hand, these methods can also include copolymerization, in which a biodegradable monomer can be combined with other monomers to yield a polymer material with excellent biodegradation performance (Ray and Bousmina 2005). Among the plastic varieties, studies have focused mainly on polycarbon dioxide/epoxy compounds (Fei et al. 2004; Nanda et al. 2011; Niu et al. 2009), synthetic acid ester plastics (Lee 1996; Mihara et al. 2008; Thellen et al. 2008), starch-based

synthetic plastics (Rivero et al. 2009), synthetic polyester plastics (Cao and Kuboyama 2010; Dziadek et al. 2017; Filippi et al. 2020; Gong et al. 2009), polybutylene succinate (PBS), polycaprolactone (PCL), polybutylene adipate terephthalate (PBAT), and polylactic acid (PLA); the physical properties of these compounds have been shown to be improved by blending, and these materials have been used in food packaging, films, containers, etc. (Nofar et al. 2020; Ravati and Favis 2013a,b). However, in the case of such biopolymers, there is no clear standard for separation (Ross et al. 2017; Soroudi and Jakubowicz 2013; Spierling et al. 2020), and it is highly likely that these plastics will be mixed with other plastic waste after consumption and discarded. In the case of PLA, several papers (Gere and Czigany 2018, 2019; La Mantia et al. 2012; Torres-Huerta et al. 2014; You et al. 2018) have reported the effects of contamination in the waste disposal process. Chemically synthetic biodegradable polymers include polylactic acid (PLA), which is transparent, strong, and favorable for reusability. Moreover, it can be processed through various methods, such as extrusion and injection molding, as a thermoplastic resin. Recently, it has been applied in DDSs (drug delivery systems), vascular grafts, and other medical implants. PCL (polycaprolactone) is a thermoplastic aliphatic polyester with high durability and hardness. It is commonly used in mulching films and plastic bags.

Polyvinyl alcohol (PVA) is a polymer produced by hydrolyzing polyvinyl acetate and is widely used as an industrial raw material. PBS (polybutylene succinate) is a polymer synthesized by the copolymerization of succinic acid or adipic acid with 1,4-butanediol. Biodegradable plastics stand out for their high heat resistance and compatibility with natural fibers, allowing them to be easily combined with other polymers. Additionally, PGA (polyglycolic acid) is another example.

### 3 Standards and guidelines

The development of degradable plastics to replace common nondegradable plastics has been a research hotspot in recent years. Owing to the combined efforts of the government and the scientific and entrepreneurial sectors, the development of degradable plastics has made continuous progress (Chiellini and Corti 2008), and these materials have been applied to various industrial materials, such as degradable agricultural mulch, degradable disposable tableware, degradable packaging materials, degradable daily plastics, and degradable surgical sutures in the pharmaceutical industry (Kopitar et al. 2022; Liu et al. 2017; Siwek et al. 2019; Yang et al. 2020). With the increasing demand and

production of degradable products, establishing a sufficiently sensitive testing technology and certification system for degradable plastics to regulate the market for degradable plastics, ensure the degradability of plastics, and, ultimately, protect the environment is particularly important and urgent (Bano et al. 2017; Chinaglia et al. 2018; Luckachan and Pillai 2011; Scott 2010; Shah et al. 2008; Vroman and Tighzert 2009). The degradation of polymers by any factor, including UV radiation, visible light, moisture, heat, and microorganisms, can be evaluated by measuring changes in physical appearance, molecular weight, amount of carbon dioxide evolved, and enumeration of microbial growth on the polymer surface after exposure to natural environments such as soil, compost, fresh water/marine water, and sewage sludge (Ammala et al. 2011; Fotopoulou and Karapanagioti 2019; Laycock et al. 2017).

#### 3.1 ASTM

In the United States, the recognized authority for establishing definitions, test methods, and standards is the American Society for Testing and Materials (ASTM) (ASTM International 2022) through its Standards Institute (Agamuthu and Faizura 2005; Kyrikou and Briassoulis 2007; Menossi et al. 2021). As one of the leading countries in the research and development of degradable plastics, the United States has several organizations engaged in related research units, such as the Plastic Degradation Research Consortium (PDRC) and the National Renewable Energy Laboratory (NREL). The aims of these methods are the synthesis of biodegradable materials, processing technology, degradation experiments, and the development of methods to test the technical standard system (Bano et al. 2017; Chinaglia et al. 2018; Shah et al. 2008). The basic focuses for the development of standard methods in the United States are broadly divided into the test environment, test method, and environmental impact evaluation (Shin 1999). Several standard test methods are currently available for evaluating the stability of polymers, and they are listed in Table 1.

#### 3.2 ISO

The International Organization for Standardization (ISO) (ISO 2022), a worldwide federation of national standard bodies (Sawada 1998), has developed standard testing methods for evaluating degradable plastics in specific environments (Zumstein et al. 2019). For example, ISO 14851 and ISO 14852 are used to determine the biodegradability of polymeric materials in aqueous media via activated sludge,

**Table 1:** List of published standards for degradation evaluation by ASTM.

| Standard       | Description  |
|----------------|--|
| ASTM D3045-92  | Standard practice for hear ageing of plastics without load   |
| ASTM D3826-98  | Standard practice for determining degradation end point in degradable polyethylene and polypropylene using a tensile test  |
| ASTM D4329-05  | Standard practice for fluorescent UV exposure of plastics  |
| ASTM D5071-99  | Standard practice for exposure of photodegradation plastics in a xenon arc apparatus   |
| ASTM D5208-01  | Standard practice for fluorescent ultraviolet (UV) exposure of photodegradable plastics  |
| ASTM D5209-92  | Standard test method for determining aerobic biodegradation of plastic materials in the presence of municipal sewage sludge  |
| ASTM D5210-92  | Standard test method for determining anaerobic biodegradation of plastic materials in the presence of municipal sewage sludge  |
| ASTM D5247-92  | Standard test method for determining the anaerobic biodegradability of degradable plastics by specific microorganisms  |
| ASTM D5271-02  | Standard test method for determining the anaerobic biodegradation of plastics materials in an activated-sludge-wastewater-treatment system                                 |
| ASTM D5272-92  | Standard practice for outdoor exposure testing of photodegradable plastics   |
| ASTM D5338-15  | Standard test method for determining aerobic biodegradation of plastic materials under controlled composting conditions, incorporating thermophilic temperatures           |
| ASTM D5338-98  | Standard test method for determining aerobic biodegradation of plastic materials under controlled composting conditions  |
| ASTM D5510-94  | Standard practice for hear ageing of oxidatively degradable plastics   |
| ASTM D5511-18  | Standard test method for determining anaerobic biodegradation of plastic materials under high-solids anaerobic-digestion conditions  |
| ASTM D5526-94  | Standard test method for determining anaerobic biodegradation of plastic materials under accelerated landfill conditions   |
| ASTM D5864-11  | Standard test method for determining aerobic aquatic biodegradation of lubricants and their components   |
| ASTM D5951-96  | Standard practice for preparing residual solids obtained after 2002 biodegradability standard methods for plastics in solid waste for toxicity and compost quality testing |
| ASTM D5988-18  | Standard test method for determining aerobic biodegradation of plastic materials in soil   |
| ASTM D5988-96  | Standard test method for determining aerobic biodegradation in soil of plastic materials or residual plastic materials after composting                                    |
| ASTM D6002-96  | Standard guide for assessing the compostability of environmentally degradable plastics   |
| ASTM D6003-96  | Standard test method for determining weight loss from plastic materials exposed to simulated municipal solid-waste (MSW) aerobic compost environment                       |
| ASTM D6139-11  | Standard test method for determining the aerobic aquatic biodegradation of lubricants and their components using the Gledhill shake flask                                  |
| ASTM D6340-98  | Standard test method for determining aerobic biodegradation of plastic materials in an aqueous or compost environment  |
| ASTM D6400-99  | Standard specifications for compostable plastics   |
| ASTM D6954-04  | Standard guide for exposing and testing plastics that degrade in the environment by a combination of oxidation and biodegradation  |
| AMST D6691-01  | Standard test method for determining aerobic biodegradation of plastic materials in the marine environment by a defined microbial consortium                               |
| ASTM D6691-17  | Standard test method for determining aerobic biodegradation of plastic materials in the marine environment by a defined microbial consortium or natural sea water inoculum |
| AMST D6692-01  | Standard test method for determining biodegradability of radiolabeled polymeric materials in seawater  |
| ASTM D6776-02  | Standard test method for determining anaerobic biodegradability of radiolabeled plastic materials in a laboratory-scale simulated landfill environment                     |
| ASTM D6852-02  | Standard guide for the determination of bio-based content, resources consumption, and environmental profile of materials and products                                      |
| ASTM D6866-04  | Standard test methods for determining the bio-based content of natural range materials using radiocarbon and isotope ratio mass spectrometry analysis                      |
| ASTM D6868-03  | Standard specification for biodegradable plastics used as coatings on paper and other compostable substrates   |
| ASTM D6954-18  | Standard guide for exposing and testing plastics that degrade in the environment by a combination of oxidation and biodegradation  |
| ASTM D7026-04  | Standard guide for sampling and reporting of results for the determination of bio-based content of materials via carbon isotope analysis                                   |
| AMST D7081-05  | Standard specifications for nonfloating degradable plastics in the marine environment  |
| ASTM D7444-18a | Standard practice for heat and humidity aging of oxidatively degradable plastics   |
| ASTM D7473-21  | Standard test method for weight attrition of nonfloating plastic materials by open system aquarium incubations   |
| ASTM D7475-11  | Standard test method for determining the aerobic degradation and anaerobic biodegradation of plastics materials under accelerated bioreactor landfill conditions           |
| ASTM D7991-15  | Standard test method for determining aerobic biodegradation of plastics buried in sandy marine sediment under controlled laboratory conditions                             |



compost, or soil. ISO 14851 determines the biological oxygen demand, whereas ISO 14852 measures evolved carbon dioxide (Jayasekara et al. 2005; Leja and Lewandowicz 2010; Niranjana and Prashantha 2018). Over the past few decades, ISOs have created many standards for the development of plastics, focusing on the degradability of plastics (Goel et al. 2021), and some of these standards are listed in Table 2.

### 3.3 CEN

The CEN technical committee TC 249 (Plastics) in the Comité Européen de Normalization (CEN) – the Committee for Standardization (Briassoulis and Degli Innocenti 2017; European Committee for Standardization (CEN) 2022) – proposed a series of degradability testing standards and technical regulations for degradable plastics (Filiciotto and Rothenberg 2021). This standard is a reference point for all European producers, authorities, facility managers, and customers (Kyrikou and Briassoulis 2007). Many CEN standards are based on ISO standards, and all EU member states are required to adopt CEN standards and specifications (Krzan et al. 2006). There are currently 35 active standards, as shown in Table 3.

### 3.4 Other published standards

In addition to ASTM, ISO, and CEN, other national organizations and research centers, as well as industries, consumers, and other interested groups, have developed relative standards and degradable technologies (Briassoulis and Dejean 2010). Different biodegradability specifications and standards have been established for different materials in different media and with different parameters to quantify their degradability. Since the publishing of the first guidelines on chemical testing by the Organization of Economic Cooperation and Development (OECD) in the 1980s, the guidelines have been sporadically updated and are freely available online, but they are not recognized worldwide (Filiciotto and Rothenberg 2021). The German Institute committee responsible for standardization is DIN FNK 103.3 (Krzan et al. 2006), the French standard specification NF U52-001 (2005) determines the biodegradability of agricultural films in soil (Rivero et al. 2009), and the Italian Normalization Organization (UNI) has also published relative standards such as UNI 11462:2012 and UNI 11495:2013 (Briassoulis and Degli Innocenti 2017). South Korea's KSM3100-1 certification standard has been in effect since June 1992 and has recently transitioned to the KS M ISO 14855 certification standard (Yu et al. 2018). Additionally, starting in 2024, in response to single-use item

regulations, biodegradation testing standards, including KS M ISO 17556 for general soil, will be implemented by the Ministry of Environment and the Korea Institute of Environment and Industry.

## 4 Environmental conditions for the biodegradation test

The biodegradability of biodegradable plastics has been evaluated under various environmental conditions, such as industrial composting and soil, marine, and freshwater environments. To certify the biodegradability of biodegradable plastics, testing is conducted on the basis of international standards. The biodegradability certification process for biodegradable plastics is shown in Figure 2, and the biodegradability of such plastics under different environmental conditions according to these standardized tests is compared in Table 4. These tests are useful for evaluating biodegradable plastics and confirming their biodegradability.

### 4.1 Industrial composting conditions

Biodegradation under industrial composting conditions was evaluated via ISO 14855 and ASTM D5338. Both standards involve monitoring and controlling the temperature, aeration, and humidity in a controlled environment to measure the extent and rate of aerobic biodegradation over 6 months at a constant temperature of  $58 \pm 2^\circ\text{C}$ . The  $\text{CO}_2$  generated during the biodegradation process is measured via gas chromatography, infrared analyzers, or similar methods. The comparison test material in ISO 14855 is cellulose with a particle size less than  $20\ \mu\text{m}$ , whereas in ASTM D5338, cellulose is used. The validity criteria included a biodegradation extent of 70 % or more within 45 days and a difference in the biodegradation rate of less than 20 % between different containers at the end of the test. In addition, volatile solids of 50–150 mg of  $\text{CO}_2$  per gram of 1 g of volatile solid should be produced 10 days after incubation in the blank.

### 4.2 Soil conditions

Biodegradation under general soil conditions was tested via ISO 17556 and ASTM D5988. The test measures the degree and proportion of aerobic biodegradation of plastic material in contact with soil through humidity control. The test duration was 6 months, and the temperature ranged from  $20\text{--}28^\circ\text{C}$ ,

**Table 2:** List of updated standards for degradation evaluation by ISO.

| Standard          | Description  |
|-------------------|--|
| ISO 7827:2010     | Water quality – evaluation of the “ready,” “ultimate” aerobic biodegradability of organic compounds in an aqueous medium – method by analysis of dissolved organic carbon (DOC)  |
| ISO 9408:1999     | Water quality – evaluation of ultimate aerobic biodegradability of organic compounds in aqueous medium by determination of oxygen demand in a closed respirometer; (this standard was last reviewed and confirmed in 2018; therefore, this version remains current)                                  |
| ISO 9439:1999     | Water quality – evaluation of ultimate aerobic biodegradability of organic compounds in aqueous medium – carbon dioxide evolution test; (this standard was last reviewed and confirmed in 2020; therefore, this version remains current)   |
| ISO 9887:1992     | Water quality – evaluation of the aerobic biodegradability of organic compounds in an aqueous medium – semi-continuous activated sludge method (SCAS); (this standard was last reviewed and confirmed in 2019; therefore, this version remains current)  |
| ISO 9888:1999     | Water quality – evaluation of ultimate aerobic biodegradability of organic compounds in aqueous medium – static test (Zahn-Wellens method); (this standard was last reviewed and confirmed in 2020; therefore, this version remains current)   |
| ISO 10634:2018    | Water quality – preparation and treatment of poorly water-soluble organic compounds for the subsequent evaluation of their biodegradability in an aqueous medium   |
| ISO 10707:1994    | Water quality – evaluation in an aqueous medium of the “ultimate” aerobic biodegradability of organic compounds – method by analysis of biochemical oxygen demand (closed bottle test); (this standard was last reviewed and confirmed in 2020; therefore, this version remains current)             |
| ISO 10708:1997    | Water quality – evaluation in an aqueous medium of the ultimate aerobic biodegradability of organic compounds – determination of biochemical oxygen demand in a two-phase closed bottle test; (this standard was last reviewed and confirmed in 2019; therefore, this version remains current)       |
| ISO 11733:2004    | Water quality – determination of the elimination and biodegradability of organic compounds in an aqueous medium – activated sludge simulation test; (this standard was last reviewed and confirmed in 2019; therefore, this version remains current)   |
| ISO 11734:1995    | Water quality – evaluation of the “ultimate” anaerobic biodegradability of organic compounds in digested sludge – method by measurement of the biogas production; (this standard was last reviewed and confirmed in 2017; therefore, this version remains current)                                   |
| ISO 14592-1:2002  | Water quality – evaluation of the aerobic biodegradability of organic compounds at low concentrations – part 1: Shake-flask batch test with surface water or surface water/sediment suspensions; (this standard was last reviewed and confirmed in 2019; therefore, this version remains current)    |
| ISO 14592-2:2002  | Water quality – evaluation of the aerobic biodegradability of organic compounds at low concentrations – part 2: Continuous flow river model with attached biomass; (this standard was last reviewed and confirmed in 2019; therefore, this version remains current)                                  |
| ISO 16221:2001    | Water quality – guidance for determination of biodegradability in the marine environment; (this standard was last reviewed and confirmed in 2017; therefore, this version remains current)   |
| ISO/TR 15462:2006 | Water quality – selection of tests for biodegradability; (this standard was last reviewed and confirmed in 2017; therefore, this version remains current)  |
| ISO 10210:2012    | Plastics – methods for the preparation of samples for biodegradation testing of plastic materials; (this standard was last reviewed and confirmed in 2017; therefore, this version remains current)  |
| ISO 13975:2019    | Plastics – determination of the ultimate anaerobic biodegradation of plastic materials in controlled slurry digestion systems – method by measurement of biogas production   |
| ISO 14851:2019    | Determination of the ultimate aerobic biodegradability of plastic materials in an aqueous medium – method by measuring the oxygen demand in a closed respirometer  |
| ISO 14852:2018    | Determination of the ultimate aerobic biodegradability of plastic materials in an aqueous medium – method by analysis of evolved carbon dioxide  |
| ISO 14853:2016    | Determination of the ultimate aerobic biodegradability of plastic materials in an aqueous system – method by measurement of biogas production  |
| ISO 14855-1:2012  | Determination of the ultimate aerobic biodegradability of plastic materials under controlled composting conditions – method by analysis of evolved carbon dioxide – part 1: General method; (this standard was last reviewed and confirmed in 2017; therefore, this version remains current)         |
| ISO 14855-2:2018  | Determination of the ultimate aerobic biodegradability of plastic materials under controlled composting conditions – method by analysis of evolved carbon dioxide – part 2: Gravimetric measurement of carbon dioxide evolved in a laboratory-scale test   |
| ISO 14593:1999    | Water quality – evaluation of ultimate aerobic biodegradability of organic compounds in aqueous medium – method by analysis of inorganic carbon in sealed vessels (CO <sub>2</sub> headspace test); (this standard was last reviewed and confirmed in 2020; therefore, this version remains current) |
| ISO 15473:2002    | Soil quality – guidance on laboratory testing for biodegradation of organic chemicals in soil under anaerobic conditions; (this standard was last reviewed and confirmed in 2020; therefore, this version remains current)   |
| ISO 15985:2014    | Plastics – determination of the ultimate anaerobic biodegradation under high-solids anaerobic-digestion conditions – method by analysis of released biogas; (this standard was last reviewed and confirmed in 2019; therefore, this version remains current)   |
| ISO 17088:2021    | Plastics – organic recycling – specifications for compostable plastics   |

**Table 2:** (continued)

| Standard         | Description  |
|------------------|--|
| ISO 17556:2019   | Plastics – determination of the ultimate aerobic biodegradability of plastic materials in soil by measuring the oxygen demand in a respirometer or the amount of carbon dioxide evolved                                |
| ISO 18830:2016   | Plastics – determination of aerobic biodegradation of nonfloating plastic materials in a seawater/sandy sediment interface – method by measuring the oxygen demand in closed respirometer                              |
| ISO 19679:2020   | Plastics – determination of aerobic biodegradation of nonfloating plastic materials in a seawater/sediment interface – method by analysis of evolved carbon dioxide  |
| ISO 20200:2015   | Plastics – determination of the degree of disintegration of plastic materials under simulated composting conditions in a laboratory-scale test   |
| ISO 22403:2020   | Plastics – assessment of the intrinsic biodegradability of materials exposed to marine inoculate under mesophilic aerobic laboratory conditions – test methods and requirements  |
| ISO 22404:2019   | Plastics – determination of the aerobic biodegradation of nonfloating materials exposed to marine sediment – method by analysis of evolved carbon dioxide  |
| ISO 22766:2020   | Plastics – determination of the degree of disintegration of plastic materials in marine habitats under real field conditions   |
| ISO 23832:2021   | Plastics – test method for determination of degradation rate and disintegration degree of plastic materials exposed to marine environmental matrices under laboratory conditions                                       |
| ISO 23977-1:2020 | Plastics – determination of the aerobic biodegradation of plastic materials exposed to seawater – part 1: Method by analysis of evolved carbon dioxide   |
| ISO 23977-2:2020 | Plastics – determination of the aerobic biodegradation of plastic materials exposed to seawater – part 2: Method by measuring the oxygen demand in closed respirometer   |
| ISO/DIS 5148     | Plastics – determination of specific aerobic biodegradation rate of solid plastic materials and disappearance time (DT50) under mesophilic laboratory test conditions (under development)                              |
| ISO/FDIS 23517   | Plastics – soil biodegradable materials for mulch films for use in agriculture and horticulture – requirements and test methods regarding biodegradation, ecotoxicity, and control of constituents (under development) |
| ISO 19095-6      | Plastics – evaluation of the adhesion interface performance in plastic-metal assemblies – part 6: Accelerated degradation test (under development)   |
| ISO/WD 5430      | Plastics – marine ecotoxicity testing scheme for biodegradable plastic materials – test methods and requirements (under development)   |
| ISO/CD 5412      | Biodegradable plastic shopping bags for composting (under development)   |

**Table 3:** List of updated standards for degradation evaluation by CEN.

| Standard          | Description   |
|-------------------|---|
| CEN/TR 15351:2006 | Plastics – guide for vocabulary in the field of degradable and biodegradable polymers and plastics items  |
| CEN/TR 17219:2018 | Plastics – biodegradable thermoplastic mulch films for use in agriculture and horticulture – guide for the quantification of alteration of films  |
| CEN/TS 17158:2018 | Composites made from cellulose-based materials and thermoplastics (usually called 3 wood polymer composites (WPC) or natural fiber composites (NFC)) – determination of particle size of lignocellulosic material |
| EN 13432:2000     | Packaging – requirements for packaging recoverable through composting and biodegradation  |
| EN 14995:2006     | Plastics – evaluation of compostability – test scheme and specifications  |
| EN 14045:2003     | Plastics – determination of the ultimate aerobic biodegradability of plastic materials in soil by measuring the oxygen demand in a closed respirometer or the amount of carbon dioxide evolved                    |
| EN 16620:2015     | Plastics – assessment of functional properties of polymers, including biodegradable polymers  |
| EN 16785-1:2015   | Plastics – determination of the degree of disintegration of plastic materials under defined composting conditions in a pilot-scale test – part 1: Method by analysis of evolved carbon dioxide                    |
| EN 16785-2:2018   | Plastics – determination of the degree of disintegration of plastic materials in soil under controlled laboratory conditions – part 2: Method by measuring the oxygen demand                                      |
| EN 17033:2018     | Plastics – biodegradable mulch films for use in agriculture and horticulture – requirements and test methods  |
| EN 17228:2019     | Plastics – bio-based polymers, plastics, and plastics products – terminology, characteristics, and communication  |
| EN 17417:2020     | Determination of the ultimate biodegradation of plastics materials in an aqueous system under anoxic (denitrifying) conditions – method by measurement of pressure increase                                       |
| EN ISO 10210:2017 | Plastics – methods for the preparation of samples for biodegradation testing of plastic materials   |



Table 3: (continued)

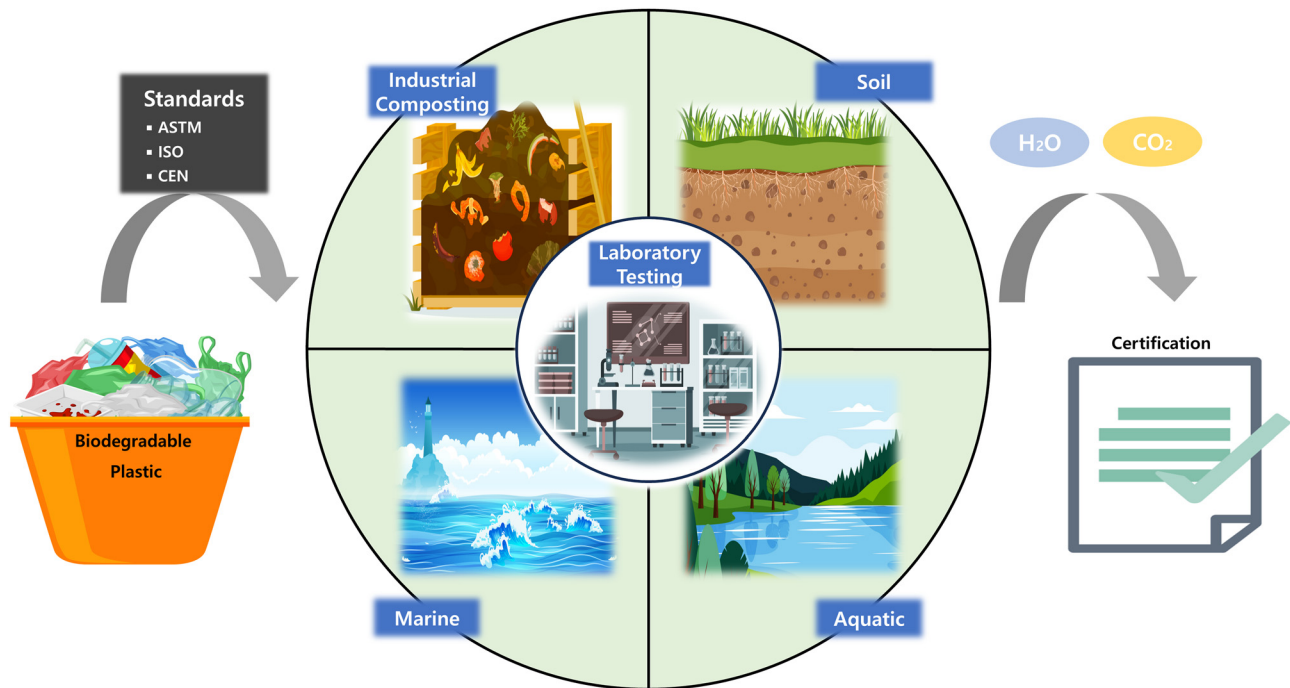
| Standard                      | Description  |
|-------------------------------|--|
| EN ISO 14851:2019             | Determination of the ultimate aerobic biodegradability of plastic materials in an aqueous medium – method by measuring the oxygen demand in a closed respirometer  |
| EN ISO 14852:2018             | Determination of the ultimate aerobic biodegradability of plastic materials in an aqueous medium – method by analysis of evolved carbon dioxide  |
| EN ISO 14853:2017             | Plastics – determination of the ultimate anaerobic biodegradation of plastic materials in an aqueous system – method by measurement of biogas production   |
| EN ISO 14855-1:2012           | Determination of the ultimate aerobic biodegradability of plastic materials under controlled composting conditions – method by analysis of evolved carbon dioxide – part 1: General method   |
| EN ISO 14855-2:2018           | Determination of the ultimate aerobic biodegradability of plastic materials under controlled composting conditions – method by analysis of evolved carbon dioxide – part 2: Gravimetric measurement of carbon dioxide evolved in a laboratory-scale test |
| EN ISO 15985:2017             | Plastics – determination of the ultimate anaerobic biodegradation under high-solids anaerobic-digestion conditions – method by analysis of released biogas   |
| EN ISO 16929:2021             | Plastics – determination of the degree of disintegration of plastic materials under defined composting conditions in a pilot-scale test  |
| EN ISO 17556:2019             | Plastics – determination of the ultimate aerobic biodegradability of plastic materials in soil by measuring the oxygen demand in a respirometer or the amount of carbon dioxide evolved  |
| EN ISO 18830:2017             | Plastics – determination of aerobic biodegradation of nonfloating plastic materials in a seawater/sandy sediment interface – method by measuring the oxygen demand in closed respirometer  |
| EN ISO 19679:2020             | Plastics – determination of aerobic biodegradation of nonfloating plastic materials in a seawater/sediment interface – method by analysis of evolved carbon dioxide  |
| EN ISO 20200:2015             | Plastics – determination of the degree of disintegration of plastic materials under simulated composting conditions in a laboratory-scale test   |
| FprEN ISO 14852 <sup>a</sup>  | Determination of the ultimate aerobic biodegradability of plastic materials in an aqueous medium – method by analysis of evolved carbon dioxide  |
| prEN 15534-2 <sup>a</sup>     | Composites made from cellulose-based materials and thermoplastics (usually called wood-polymer composites (WPC) or natural fiber composites (NFC)) – part 2: Characterization of compounds   |
| prEN 15534-3 <sup>a</sup>     | Composites made from cellulose-based materials and thermoplastics (usually called wood-polymer composites (WPC) or natural fiber composites (NFC)) – part 5: Specifications for cladding profiles and tiles  |
| prEN ISO 22403 <sup>a</sup>   | Plastics – assessment of the intrinsic biodegradability of materials exposed to marine inocula under mesophilic aerobic laboratory conditions – test methods and requirements  |
| prEN ISO 22404 <sup>a</sup>   | Plastics – determination of the aerobic biodegradation of nonfloating materials exposed to marine sediment – method by analysis of evolved carbon dioxide  |
| prEN ISO 22526-1 <sup>a</sup> | Plastics – carbon and environmental footprint of bio-based plastics – part 1: General principles   |
| prEN ISO 22526-2 <sup>a</sup> | Plastics – carbon and environmental footprint of bio-based plastics – part 2: Material carbon footprint, amount (mass) of CO <sub>2</sub> removed from the air and incorporated into polymer molecule  |
| prEN ISO 22526-3 <sup>a</sup> | Plastics – carbon and environmental footprint of bio-based plastics – part 3: Process carbon footprint, requirements, and guidelines for quantification  |
| prEN ISO 22766 <sup>a</sup>   | Plastics – determination of the degree of disintegration of plastic materials in marine habitats under real field conditions   |
| prEN ISO 23977-1 <sup>a</sup> | Plastics – determination of the aerobic biodegradation of plastic materials exposed to seawater – part 1: Method by analysis of evolved carbon dioxide   |
| prEN ISO 23977-2 <sup>a</sup> | Plastics – determination of the aerobic biodegradation of plastic materials exposed to seawater – part 2: Method by measuring the oxygen demand in closed respirometer   |

<sup>a</sup>Standards still being approved and not published yet.

with a tolerance of  $\pm 2^\circ\text{C}$ . ISO 17556 utilizes amorphous cellulose powder or PHB as the reference test material, with validity criteria of 60 % or more biodegradation extent for the reference material after 180 days and BOD or emitted CO<sub>2</sub>. ASTM D5988 uses cellulose or starch as the reference test material, with validity criteria of 70 % or more biodegradation within 6 months and CO<sub>2</sub> emission from the control reactor being 20 % or less than the mean value at the end of the test.

### 4.3 Marine conditions

Biodegradation under marine conditions was tested via ISO 19679 and ASTM D6691. ISO 19679 measures the extent and rate of anaerobic biodegradation of plastic materials precipitated in marine sand deposits at the marine–water interface. The test duration was under 24 months at a constant temperature of 15–25 °C with a tolerance of  $\pm 2^\circ\text{C}$ . ISO 19679 uses an amorphous cellulose filter and has validity



**Figure 2:** Biodegradable plastic certification process based on the degradation environment.

**Table 4:** Biodegradability of biodegradable plastics according to certification criteria for biodegradation according to environmental conditions.

| Environmental conditions | Test standard | Biodegradability of biodegradable plastic        | Reference  |
|--------------------------|---------------|--|--|
| Industrial composting    | ISO 14855     | PBS (powder) – 71.9 % after 90 days              | Zhao et al. (2005)                                 |
|                          |               | PCL (powder) – 90 % after 47 days                | Funabashi et al. (2007)                            |
|                          |               | PBAT (film) – 90 % after 130 days                | Jacquel et al. (2015)                              |
|                          |               | PLA (powder) – 90 % after 20 days                | Kunioka et al. (2006)                              |
|                          |               | PHB (film) – 79.7 % after 110 days               | Weng et al. (2011)                                 |
|                          |               | Cellulose (powder) – 97 % after 180 days         | Madhu et al. (2019)                                |
|                          | ASTM D5338    | PBS (powder) – 20 % after 45 days                | Yang et al. (2005)                                 |
|                          |               | PCL (film) – 95 % after 110 days                 | (Dilawar and Eskicioglu 2022; Kalita et al. 2020b) |
|                          |               | PBAT (pellet) – 92 % after 180 days              | Muniyasamy et al. (2013)                           |
|                          |               | PLA (film) – 90 % after 140 days                 | (Dilawar and Eskicioglu 2022; Kalita et al. 2020b) |
| Soil                     | ISO 17556     | PHB (film) – 80 % after 28 days                  | Tabasi and Ajji (2015)                             |
|                          |               | Cellulose (film) – 90 % after 28 days            | Tabasi and Ajji (2015)                             |
|                          |               | PBS (powder) – 90 % after 212 days               | Šerá et al. (2020)                                 |
|                          |               | PCL (film) – 17 % after 90 days                  | Nevoralová et al. (2020)                           |
|                          |               | PBAT (film) – 34 % after 130 days <sup>a</sup>   | Sun et al. (2018)                                  |
|                          |               | PLA (film) – <0.1 % after 105 days <sup>a</sup>  | Lv et al. (2017)                                   |
|                          | ASTM D5988    | PHB (powder) – 96.4 % after 170 days             | Šerá et al. (2020)                                 |
|                          |               | Cellulose (powder) – 90 % after 170 days         | Šerá et al. (2020)                                 |
|                          |               | PBS (dumbbell specimens) – 65.2 % after 180 days | Phua et al. (2012)                                 |
|                          |               | PCL (sheet) – 51 % after 90 days                 | Mariani et al. (2007)                              |
|                          |               | PBAT (film) – 21 % after 180 days                | Palsikowski et al. (2018)                          |
|                          |               | PLA (film) – 16 % after 180 days                 | Palsikowski et al. (2018)                          |
|                          |               | PHB (film) – 10.5 % after 25 days                | Kim et al. (2000)                                  |
|                          |               | Cellulose (powder) – 88 % after 208 days         | Mistriotis et al. (2019)                           |

Table 4: (continued)

| Environmental conditions | Test standard | Biodegradability of biodegradable plastic      | Reference                               |
|--------------------------|---------------|--|---|
| Marine                   | ISO 19679     | PBS (monofilament) – 27.3 % after 180 days     | Kim et al. (2023)                       |
|                          |               | PCL (powder) – 3 % after 28 days               | Hino et al. (2023)                      |
|                          |               | PBAT (film) – 1.5 % after 82 days <sup>a</sup> | Delacuvellerie et al. (2021)            |
|                          |               | PLA (powder) – 54 % after 28 days              | Hino et al. (2023)                      |
|                          |               | PHB (film) – 57 % after 180 days               | Briassoulis et al. (2020)               |
|                          | ASTM D6691    | Cellulose (filter) – 86 % after 180 days       | Kim et al. (2023)                       |
|                          |               | PBS (film) – 1.3 % after 28 days               | Nakayama et al. (2019)                  |
|                          |               | PCL (film) – 14.5 % after 28 days              | Nakayama et al. (2019)                  |
|                          |               | PBAT (powder) – 1 % after 28 days              | Nakayama et al. (2019)                  |
|                          |               | PLA (film) – 0.3 % after 28 days               | Nakayama et al. (2019)                  |
| Aquatics                 | ISO 14852     | PHB (pellet) – 86 % after 28 days              | Tanadchangsang and Pattanasupong (2022) |
|                          |               | Cellulose (filter) – 100 % after 28 days       | Tanadchangsang and Pattanasupong (2022) |
|                          |               | PBS (film) – 0.38 % after 7 days               | Guo et al. (2010)                       |
|                          |               | PCL (powder) – 77.6 % after 117 days           | García-Depraect et al. (2022)           |
|                          |               | PBAT (film) – 10 % after 60 days               | Someya et al. (2007)                    |
|                          | ISO 14853     | PLA (film) – 0.03 % after 7 days               | Guo et al. (2010)                       |
|                          |               | PHB (powder) – 83 % after 117 days             | García-Depraect et al. (2022)           |
|                          |               | Cellulose (powder) – 86.8 % after 68 days      | García-Depraect et al. (2022)           |
|                          |               | PBS (powder) – 3.1 % after 77 days             | García-Depraect et al. (2022)           |
|                          |               | PCL (powder) – 4.5 % after 77 days             | García-Depraect et al. (2022)           |
|                          |               | PBAT (powder) – <2 % after 77 days             | García-Depraect et al. (2022)           |
|                          |               | PLA (powder) – 4.6 % after 77 days             | García-Depraect et al. (2022)           |
|                          |               | PHB (powder) – 83.9 % after 77 days            | García-Depraect et al. (2022)           |
|                          |               | Cellulose (powder) – 86.7 % after 56 days      | García-Depraect et al. (2022)           |

<sup>a</sup>Weight loss.

criteria of 60 % or more biodegradation extent for the reference material after 180 days, with CO<sub>2</sub> emission not exceeding 20 %. ASTM D6691 measures the extent and rate of anaerobic biodegradation via well-known anaerobic marine microorganisms or plastic materials exposed to natural seawater. The test duration was 180 days, the temperature was maintained at 30 ± 2 °C, and the CO<sub>2</sub> concentration was measured. The reference test materials used were cellulose powder or paper. The validity criterion was 70 % or more biodegradation after 6 months for the reference material.

#### 4.4 Aquatic conditions

Biodegradation under freshwater conditions was tested via ISO 14852 for aerobic biodegradation and ISO 14853 for anaerobic biodegradation. ISO 14852 measures the extent and rate of aerobic biodegradation of plastic materials through CO<sub>2</sub> production. The test duration was a maximum of 6 months, and a constant temperature of 20–25 °C was maintained with a tolerance of ± 1 °C. The reference test material for ISO 14852 can be microcrystalline cellulose, unbleached cellulose filter, or poly-β-hydroxybutyrate, with the validity criterion of a biodegradation extent of 60 % or more for the reference material at the end of the test. ISO

14853 measures the extent and rate of anaerobic biodegradation via the use of poly-β-hydroxybutyrate, cellulose, or poly (ethylene glycol) 400 as reference materials. The test duration was 60 days at 35 ± 2 °C. The validity criterion was a 70 % or greater biodegradation extent for the reference material after 180 days.

## 5 Common laboratory evaluation methods

The evaluation of degradable plastics can be categorized into two approaches: evaluating the physicochemical properties of the plastic and evaluating its degradability. An ideal degradable plastic should be both durable and degradable. Both aspects are equally important, and the overall certification process for biodegradable plastics is described in Figure 2. The analytical methods for analyzing biodegradable plastics that can be performed in the laboratory are presented in Figure 3 below. This does not directly demonstrate the biodegradability of biodegradable plastics. However, it provides supporting evidence by analyzing the changes in the properties of biodegradable plastics throughout the biodegradation process.

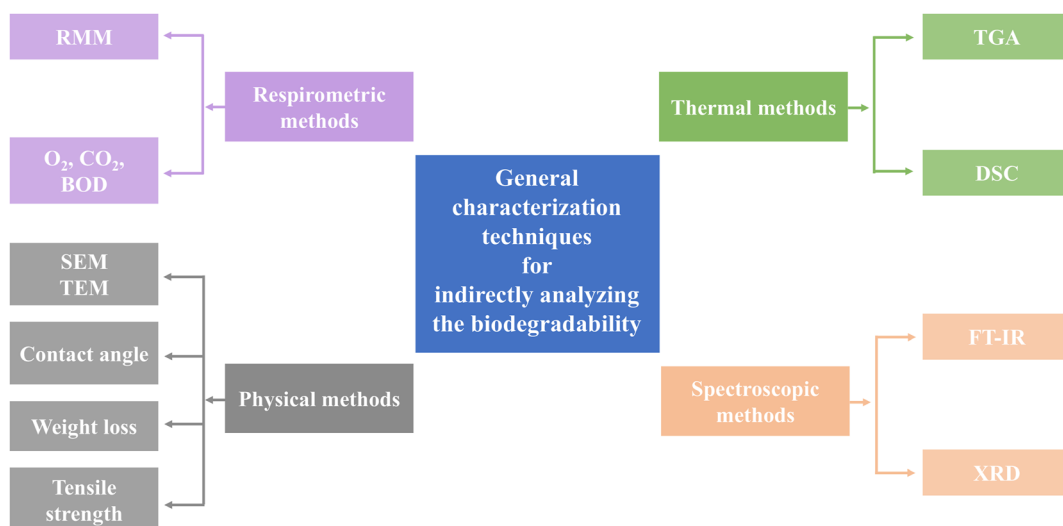


Figure 3: General characterization techniques for indirectly analyzing the biodegradability.

Physicochemical analysis of degradable plastics can be classified as either physical analysis or chemical analysis. Physical analysis refers to changes in the bulk structure, such as cracking, embrittlement, peeling, and flaking. These physical changes are closely related to the material properties, durability, and structural stability of plastics. On the other hand, chemical characterization refers to changes at the molecular level, such as bond breaking or oxidation of long polymer chains to create new molecules, which are usually accompanied by a significant reduction in chain length. A reduction in polymer chain length is a crucial indicator of plastic biodegradability. Shorter polymer chains can undergo faster biodegradation by microorganisms and environmental factors, making them fundamental characteristics of biodegradable plastics (Chamas et al. 2020; Ghosh and Jones 2021; MacLeod et al. 2021).

## 5.1 Weight loss

The weight loss of tested samples, such as plastics and films, is widely used in degradation tests, although no direct proof of biodegradation has been obtained (Bano et al. 2017; Chinaglia et al. 2018; Shah et al. 2008). The degradability of compatible thermal–plastic starch/polyethylene was investigated in five different environments on the basis of mass loss percentage (Shujun et al. 2006). The results of the evaluation of the weight loss of poly (lactic acid) (PLA), poly ( $\epsilon$ -caprolactone) (PCL), and commercially available material (PHB) indicated that PLA degraded the most rapidly during the first 4 weeks of enzymatic degradation (Johari et al. 2022; Li et al. 2017; Richert and Dąbrowska 2021; Żenkiewicz

et al. 2013). In addition, it has also been reported that the weight loss rate of a poly (L-lactic)-grafted dextran film rapidly increases with increasing sugar unit content because of the introduction of hydrophilic Dex segments and low crystallinity (Ouchi et al. 2006). Furthermore, a relationship between crosslink density and mass loss has been established for degradable gelatin films (Chiou et al. 2008; Dalev et al. 2000; Martucci and Ruseckaite 2015). However, in experiments testing the degradation rates of degradable, nondegradable, and biodegradable plastics, the dry weights of some samples increased over time, which seems unlikely (Lott et al. 2020; Mudhoo et al. 2011; Wellfair 2008). Problems can arise due to excessive cleaning of the sample or incomplete collection. The screening and analysis of the matrix surrounding a sample can be used to measure the disintegration characteristics quantitatively. For finely distributed polymer samples (e.g., powders), appropriate extraction and separation techniques can be used to determine the amount of residual polymer. By combining structural analysis of residual materials with low-molecular-weight intermediates, detailed information about the degradation process can be obtained, especially if a defined synthetic test medium is used (Bano et al. 2017; Chinaglia et al. 2018; Shah et al. 2008; Witt et al. 2001).

## 5.2 Tensile strength

The tensile properties of films or plastics are the most important properties to study, especially for those used for commercial purposes. Plastic materials should have adequate strength to resist mechanical stress and maintain

integrity during transport, handling, and storage. According to previous reports, the degradation process causes a decrease in mechanical properties, which further promotes microbial action (Arrieta et al. 2014a,b; Bonilla et al. 2021; Meereboer et al. 2020). Increasing the content of chitosan in brown rice starch-based films may also improve the cohesive properties of polymer blends, potentially leading to high interpolymer resistance (Fonseca-García et al. 2021; Hasan et al. 2020; Hernández-García et al. 2021), which may be related to the infiltration of chitosan into the membrane matrix, resulting in enhanced van der Waals forces and the formation of hydrogen bonds in the polymer molecular chain (Divya and Jisha 2018). The incorporation of a cross linker and plasticizer caused a tremendous change in the mechanical properties of another chitosan film incorporated with citric acid and glycerol (Nilsen-Nygaard et al. 2021; Priyadarshi et al. 2018; Priyadarshi and Rhim 2020). A study of the biodegradation properties of mulching films made from natural fibers and biodegradable polymers revealed that the tensile strength and elongation of the better-performing fiber/polymer films were lower than those of commonly used PE films, but the high-tensile fiber/polymer films were strong enough to be used as mulching films in the test (Tan et al. 2016). An investigation of the relationship between apricot kernel skin (AKS) and tensile strength indicated that hydrophobic interactions and hydrogen bonds between soy protein isolate and polyphenols could improve the tensile strength of the film (Li et al. 2019).

### 5.3 Contact angle

The contact angle of a water droplet on the surface of a film or plastic is usually used to describe hydrophobicity, and changes in droplet volume as a function of time could be used to estimate wettability or hydration properties; this parameter is also a good indicator of how the wettability of a material changes during biodegradation with the molecular activity of the test material (Atef et al. 2014; Kalita et al. 2019; Kalita et al. 2020a,b). As the interknot distance can influence contact angle calculations, this parameter should be maintained at a reasonable and constant value within a study (Stalder et al. 2006). A decrease in the hydrophilicity of the agar-based nanocomposite films with increasing nanocrystalline cellulose (NCC) content was revealed by an increase in the contact angle, which could be related to the highly crystalline properties of NCC (Atef et al. 2014). The addition of tea extract tended to increase the hydrophobicity of the starchy film (Atef et al. 2014). Research on the influence of compost temperature on the degradability of PLA-based biocomposite films revealed that the stability of

the solvent droplets on the sample surface and the presence of holes and cracks on the sample surface caused by degradation were the factors leading to deviations in the test results (Kalita et al. 2019; Kalita et al. 2020a,b). Furthermore, droplet diffusion on the surface of a soluble membrane is faster than that on the surface of an insoluble membrane or swelling membrane when the droplet is in contact with liquid water (Basiak et al. 2017; Dong et al. 2009; Jimenez et al. 2012).

### 5.4 FT-IR

Fourier transform infrared (FT-IR) spectroscopy reveals the orientation and shape of the polymer chains at the molecular level. The idea is to determine the frequency of the bonds in different samples and whether there is a change in the frequency of certain bonds. A small plastic sample is placed in the device, through which IR radiation is absorbed at a certain frequency according to the chemical bonds contained in the sample, resulting in a spectrum containing different peaks. These peaks are likened to fingerprints of the bonds and functional groups contained within them (Lott et al. 2020; Mudhoo et al. 2011; Wellfair 2008). The position, shape, and intensity of the peaks in this spectrum reveal details about the molecular structure of the samples, so FT-IR can be used to identify unknown materials and determine material quality or consistency (Julienne et al. 2019; Kalođerakis et al. 2017; Wang et al. 2020). Generally, in an FT-IR spectrum, an absorption peak at approximately  $3,300\text{ cm}^{-1}$  is associated with the stretching vibration of C–O groups (El Knidri et al. 2018; Pereira et al. 2015; Prietto et al. 2017), and peaks at approximately  $2,900\text{ cm}^{-1}$  are associated with C–H stretching vibrations that are also characteristic of polymer matrix materials (Jaramillo et al. 2016; Qin et al. 2019; Thakur et al. 2019). This technique was successfully employed to determine the formation of anhydride and ketone groups in a degradable multilayer film during thermal crosslinking at  $110\text{ °C}$  for 16 h, which led to almost no bacterial adhesion in 24 h (Wang et al. 2013; Zhou et al. 2021; Zhu and Loh 2015). The biodegradation rate of gelatin films is related to the type of crosslinking agent and the crosslinking density. The water absorption of a crosslinked gelatin sample depends on the crosslinking density. The higher the crosslinking density is, the lower the adsorption capacity. Microorganisms and their enzymes cannot penetrate without water adsorption or the expansion of the crosslinked gelatin network (Chiou et al. 2008; Dalev et al. 2000; Martucci and Ruseckaite 2015). Moreover, peak shifting occurred during FTIR analysis for each test sample and was due to the formation of hydroxyl and carboxyl groups on the film samples (Kalita et al. 2019;



Kalita et al. 2020a,b). FT-IR analysis was carried out to investigate the interaction between agar and cellulose nanoparticles in the range of 400–4,000  $\text{cm}^{-1}$  (Atef et al. 2014). In addition, a study of starch-derived degradable plastics via FT-IR analysis suggested that starchy plastics could be used as substitutes for synthetic plastics such as polyethylene and polystyrene in several applications (Arikan and Bilgen 2019; Ashok et al. 2018; Rao et al. 2022).

## 5.5 XRD

X-ray diffraction (XRD) peaks indicate a hydrated crystallite structure due to the integration of water molecules in the crystal lattice. Relatively high peaks with sharper reflections indicate improved crystallinity or dense packing in the main chain and a relatively well-organized long-range structure (Giannakas et al. 2014; Mujtaba et al. 2019; Thakhiew et al. 2015). A shift toward lower angles indicates the formation of an intercalated nanocomposite structure (Giannakas et al. 2016). The analysis of the brown rice starch-based biodegradable chitosan membrane revealed a scattering peak and a wide range of amorphous backgrounds, indicating that the membrane is a semicrystalline material. However, owing to the strong interaction between the two molecules during the process of film formation, the regularity of the molecular chain is reduced, resulting in the loss of the A-type crystal peak structure (Fonseca-García et al. 2021; Hasan et al. 2020; Hernández-García et al. 2021). Similar results showing the absence of A-type crystals were also presented in another study on corn starch-based polymers, which showed that the native corn starch structure was completely destroyed (V.M. Azevedo et al. 2017; Chen et al. 2022; Fang et al. 2020; Mendes et al. 2016; Ojogbo et al. 2020; Vieira et al. 2022). In addition, the V-type crystallinity peaks of the rice starch-based films disappear in the blends, which may be related to the decrease in the number of molecules formed by hydrogen bonds between the plasticizer and starch during the process (V.M. Azevedo et al. 2017; Mendes et al. 2016; Ojogbo et al. 2020; Suriyatem et al. 2018). Notably, the degree of crystallization of amylopectin is low at high water concentrations, which leads to the gradual attenuation and peak shift of pure starch (Todica et al. 2016). Furthermore, the high crystallinity of cellulose nanoparticles in an alginate bionanocomposite film indicated that the amorphous region of the cellulose chain was removed by acid hydrolysis, whereas the crystalline region remained intact (Abdollahi et al. 2013; Azeredo et al. 2017; Deepa et al. 2016). A comparison of the XRD patterns of chitosan films prepared from aqueous solution revealed that the presence of corn oil or olive oil had no obvious effect on the

interaction between chitosan and clay (Díez-Pascual and Díez-Vicente 2015; Giannakas et al. 2017; Kumari et al. 2015; Qu and Luo 2021; Wang et al. 2018).

## 5.6 Thermal property analysis

### 5.6.1 TGA

Thermogravimetric analysis (TGA) is used to characterize the degradation and thermal stability of plastics or films, and the continuous weight loss and temperature are recorded and analyzed during differential thermal analysis (Arikan and Bilgen 2019; Ashok et al. 2018; Rao et al. 2022; Suriyatem et al. 2018). The thermal stability of neat PLA and PLA-active nanocomposite films was studied with TGA under a nitrogen atmosphere; the first degradation step started at approximately 120 °C and was related to thymol degradation. The loss of PLA may be related to some degradation of these materials during treatment caused by the presence of metal nanoparticles, which increases the thermal conductivity of the nanocomposites, accelerating the degradation process of the polymer matrix (Fortunati et al. 2019; Munteanu et al. 2014; Ramos et al. 2014). The thermal properties of the neat chitosan films and crosslinked citric acid- and glycerol-plasticized modified films were compared via TGA, and the initial weight loss due to water loss was approximately 10–20 % for both films. However, the temperature at which the loss occurred was greater for the modified films because the added glycerol was bound to the water in the film, making it difficult for the water to escape, and more energy was needed to overcome this effect (Debandi et al. 2016; Nilsen-Nygaard et al. 2021; Okur et al. 2019; Priyadarshi et al. 2018; Priyadarshi and Rhim 2020). Similarly, TGA-DTA of the films was performed to identify the thermal degradation patterns of the chitosan films. The loss of surface mass was also associated with the decomposition of glycerin-rich phases observed in starch-glycerol-based films but more significantly with membranes containing yerba mate extracts (Jaramillo et al. 2016; Qin et al. 2019; Thakur et al. 2019).

### 5.6.2 DSC

Differential scanning calorimetry (DSC) is usually employed to determine the melting temperature of plastics or films from the peak temperature of the melting endotherm (Jaramillo et al. 2016; Qin et al. 2019; Thakur et al. 2019). The latest results of DSC analysis of natural and synthetic biopolymers indicate that a reduction in the amount of mobile amorphous phases leads to a decrease in macromolecular chain length due to the increased mobility of shorter chains,

making crystallization possible (Bonilla et al. 2021). DSC was used to investigate the effects of the addition of thymol and Ag-NPs on the thermal properties of the PLA-based films, but no crystallization or melting phenomena were detected. The addition of low-molecular-weight compounds decreases the glass transition temperature of PLA and increases the mobility of the polymer, thus reducing the rigidity and brittleness of PLA (Brdlik et al. 2021; Fortunati et al. 2019; Luo et al. 2019; Munteanu et al. 2014; Ramos et al. 2014; Ramos et al. 2016; Yu et al. 2020). The literature shows that the absence of melting and cold crystallization may be related to the mixing of PLA with enzymes after heating during the biodegradation process (Kalita et al. 2019; Kalita et al. 2020a,b). A decrease in both the melting temperature and enthalpy of starch-*yerba mate* extract films may indicate that a less stable crystal structure requires less energy to melt, resulting in better quality packaging (Ashogbon and Akintayo 2014; Jaramillo et al. 2016; Kaur et al. 2004, 2006; Qin et al. 2019; Thakur et al. 2019).

## 5.7 RMM

The respiration measurement method (RMM), which includes cumulative measurement respiration (CMR), gravimetric measurement respiration (GMR), and direct measurement respiration (DMR), is a commonly used experimental approach for assessing biodegradability. This method involves measuring the cumulative amount of CO<sub>2</sub> generated as microorganisms decompose organic substances in test samples.

The experimental procedure utilized soil and suitable environmental samples containing organic materials in an environment conducive to microbial activity. It quantifies the concentration of CO<sub>2</sub> produced during the decomposition of organic materials in test samples, representing the kinetics of the biodegradation process and the biodegradability of the samples. The evaluation of biodegradability and degradation kinetics is based on CO<sub>2</sub> measurement data over time. Since the measurement results reflect the decomposition of organic materials by microorganisms, the biodegradability and degradation rate of the samples can be estimated. The respiration measurement method serves as a quantitative approach for assessing biodegradability, degradation potential, and degradation kinetics, making it a valuable tool in various studies related to biological or chemical processes, including microbial activity, biodegradation efficiency, organic matter decomposition, and the evaluation of biodegradability in environmental samples.

This information can be used in your paper for discussing the application of Respiration Measurement Method in

evaluating biodegradability and its significance in environmental and biological studies.

## 5.8 Electron microscopy

### 5.8.1 SEM

Scanning electron microscopy (SEM) allows monitoring of changes in the morphology of materials at the microscale (Adamcová and Vavrková 2014; Markowicz and Szymańska-Pulikowska 2019). This method was used to examine the surface structure of agar and agar-based nanocomposite films sputtered with gold under high vacuum and at an accelerating voltage (Atef et al. 2014). During the biodegradation process, the sample first exhibited holes of different shapes and sizes, the film on the sample surface became completely eroded, the smoothness disappeared, the film broke, and finally, a large area of the film disappeared (Adamcová and Vavrková 2014; Markowicz and Szymańska-Pulikowska 2019). SEM micrographs revealed that the corn starch–chitosan blend had a uniform surface, and the fractured samples did not show agglomeration of chitosan in the completely deconstructed starch matrix (V.M. Azevedo et al. 2017; Mendes et al. 2016; Ojogbo et al. 2020). The SEM micrographs also revealed that surface erosion of the modified PLA sample occurred prior to biodegradation, as hydrolytic degradation resulted in intermediate products being assimilated by microorganisms and converted to water. Cracks and pores on the sample surface confirmed biodegradation, reducing the amount of the amorphous part (Kalita et al. 2019; Kalita et al. 2020a,b). The surface of the original nanocomposite was obviously covered with chitosan, which made the surface smooth and uniform. However, after 6 days of incubation with activated sludge, the graphene oxide was completely exposed. In other words, some bacteria can efficiently utilize chitosan as a carbon source by producing chitosanase to degrade chitosan into glucosamine oligomers (Fan et al. 2017).

### 5.8.2 TEM

The transmission electron microscopy (TEM) technique can be valuable for evaluating biodegradable plastics because TEM uses an electron beam to penetrate a sample, providing high-resolution images and allowing for the observation of fine details and changes. This enables the observation and understanding of the decomposition and degradation processes of biodegradable plastics at the microscopic scale. Surface morphology analysis of the poly (3-hydroxybutyrate-co-3-hydroxyhexanoate) (PHBHHx)/PBAT and PHBHHx/PLA

blend sheets was conducted via TEM, confirming their biodegradability in marine environments. The microbial degradation behavior of marine polyester blends containing PHBHHx was assessed by monitoring the biodegradation process over time and periodically conducting TEM analysis, and the biodegradation of the biodegradable plastic samples was verified. This approach allows for the assessment of biodegradability results by considering factors such as the degradation rate, the form of decomposition products, and chemical composition changes.

## 6 Discussion

Currently, standards for evaluating the biodegradation of biodegradable plastics are being developed by international standardization organizations such as the ISO and ASTM. However, there is a lack of internationally recognized standard testing methods. Manufacturers and research institutions face challenges in ensuring consistency and comparability of results, as they adopt their own unique testing methods on the basis of existing standards. Additionally, biodegradability tests for biodegradable plastics are conducted mainly in laboratories, making it difficult to fully replicate the complex conditions and interactions of real-world environments. Consequently, test outcomes may not align with actual biodegradation rates, which can vary owing to factors such as material type, thickness, environmental conditions, and microbial communities, making predictions challenging.

To address these issues, encouraging active participation from manufacturers and research institutions in the development of standardized testing protocols is crucial. Standard tests should consider various conditions, including flexible testing periods on the basis of anticipated usage and degradation environments, as well as regional microbial diversity. Moreover, collecting data from real-world degradation environments can aid in the development of equipment and technologies that more accurately replicate these conditions, thereby complementing and validating laboratory test results.

Finally, some biodegradable plastic products are labeled with terms such as “biodegradable” or “eco-friendly,” but the lack of clear definitions and standardized criteria for these terms can cause consumer confusion. To resolve this, clear definitions for terms such as “biodegradable” must be established, and standardized regulations should be implemented to minimize consumer misunderstanding. Additionally, the credibility of biodegradability claims and certifications should be enhanced through independent third-party certification systems.

Continuous research and regulatory improvements are essential to ensure the environmentally friendly use of biodegradable plastics.

## 7 Conclusions

Research on biodegradable plastics has been ongoing for decades, and significant progress has been made. In response to these advancements, various countries and institutions have developed and announced standards for biodegradable plastics. The development of international standards for biodegradable plastics has two main advantages. First, it sets a criterion for biodegradability, which will guide future research and product development in biodegradable plastics. This provides a scientific standard for the somewhat abstract concept of “biodegradable plastics” and will serve as an objective indicator. Second, it ensures consistent product performance. This consistency builds consumer trust. These benefits indicate that standards for biodegradable plastics will greatly contribute to the development of the biodegradable plastics industry. However, a summary of the standards and research published to date has revealed several problems.

Currently, biodegradability assessments are performed on materials instead of on finished products. Testing small material samples instead of finished products affects the actual surface area of the product, which can impact microbial attachment during the biodegradation process. Additionally, when materials are transformed into finished products, additives are mixed to enhance their physical and thermal properties. These additives can affect the biodegradability of the final product. Furthermore, the environmental conditions under which standards are developed often differ from those under which biodegradable plastics actually decompose, leading to significant variances because of various factors. These factors can lead to discrepancies in the biodegradability of finished products, thereby reducing consumer trust and posing significant obstacles to industry development. Research on standards should proceed in a direction that addresses these issues.

The development of biodegradable plastics is essential for reducing CO<sub>2</sub> emissions and preventing environmental pollution. This represents a green and sustainable path and is a direction for future development. Therefore, it is essential that a clear standardization method for biodegradable plastics be established, as suggested in this paper.

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