Journal of Polymers and the Environment
https://doi.org/10.1007/s10924-024-03311-8

REVIEW

Critical Factors in Lab-Scale Compostability Testing

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Accepted: 30 April 2024
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Abstract
Composting offers a solid waste management alternative to landfilling resulting in soil amendment products with fertilization and moisture retention benefits and collateral methane reduction by diverting organic wastes from anaerobic landfill environments. An increasing array of materials and products are marketed as compostable, though only a limited range of these are covered by certification schemes. Greater accessibility and deeper understanding of compostability testing is needed to promote meaningful evaluation of the viability and optimal conditions for composting wider ranges of materials. This article describes various critical aspects of laboratory-scale methodology that can be optimized for more consistent, accurate, and efficient testing. While most of the reviewed studies are based on standardized international test methods, modifications to vessel design, medium, control systems, and evaluation show promise. Learnings are also drawn from biodegradation tests using soil and aqueous media. Particular consideration is given to evaluating compostability of textiles, including nonwovens, which today are primarily disposed of in landfills yet have the potential to supplement organic carbon in compost mixtures with nitrogen rich food waste. Furthermore, biodegradation properties of both natural and man-made textile fibers have received growing attention in recent years. Fiber fragments found in oceans and the surge of disposable face coverings used during the COVID-19 pandemic have been widely covered in the mainstream media, highlighting the importance of understanding biodegradation properties for textiles. This review consolidates and organizes diverse and essential procedural details reported in various standards and studies with the goal of encouraging and guiding successful implementation of compostability testing more broadly in laboratories. Certain gaps in test methodologies are identified to help focus future research. Reliable, accessible testing is crucial to expand the beneficial impacts of composting in waste management.

Keywords Compost · Biodegradability · Test method · Textiles

Introduction

Composting

Composting is a potentially attractive alternative to landfill disposal for biodegradable materials. It can reduce both land use and global warming impacts; the resulting compost is also a valuable soil amendment product for agriculture and infrastructure, as shown in Fig. 1 [1, 2]. Composting, an aerobic process, is characterized by microbial breakdown of materials in the presence of oxygen (O_2) to produce primarily carbon dioxide (CO_2), water, and energy. In contrast, anaerobic degradation is the dominant process in landfill environments, producing methane (CH_4), with 27–30 times the global warming potential of CO_2 over 100 years [3]. Aerobic microbial respiration converts carbon and other nutrients stored in organic matter to CO_2, water, and energy. An example of glucose conversion is shown in Eq. 1. Biodegradation is often quantified by measuring O_2 consumed or CO_2 produced. These values can be used to calculate the amount of carbon consumed. Based on a balanced version of Eq. 1 for the test material, or other carbon analysis of the undegraded sample, researchers can determine the percent biodegraded to CO_2.

$$C_6H_{12}O_6(Glucose) + O_2 \rightarrow 6CO_2 + 6H_2O + ATP\,(Energy) \quad (1)$$

There are several categories of compost piles for both industrial and home use. Open static piles require the least intervention. Turned or aerated windrows and in-vessel piles use various techniques to improve consistency of

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Published online: 14 September 2024

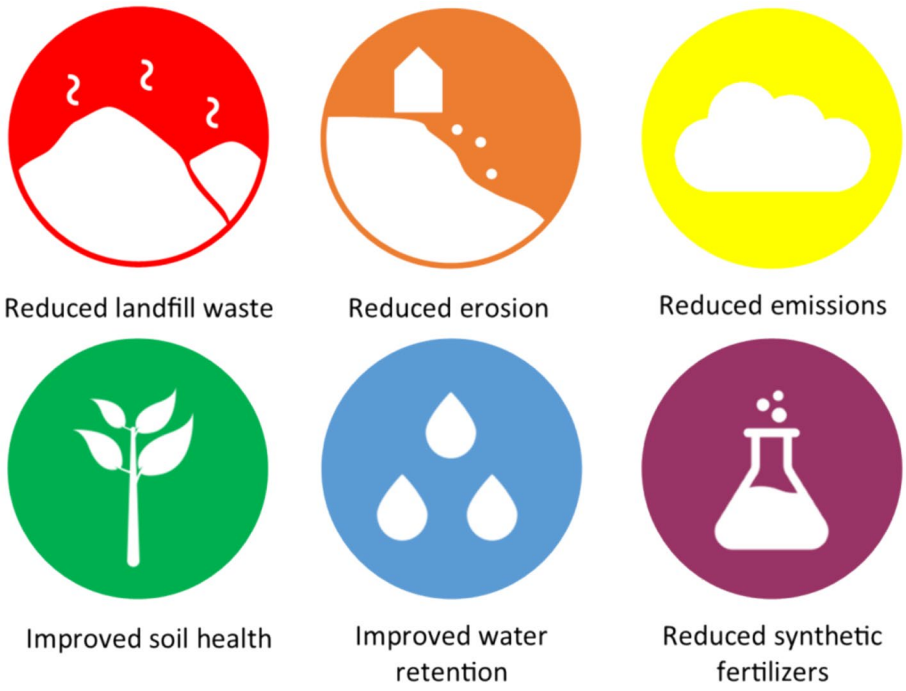

Fig. 1 Benefits of composting

temperature and aerobic conditions [2]. Laboratory systems are relatively small in-vessel systems intended to replicate the results of larger systems. While full-scale compost piles are heated internally by the degradation process itself, lab-scale systems typically require an external heat source to reach the thermophilic temperatures associated with optimal biodegradation. Air flow must also be provided in place of the wind and diffusion found in outdoor conditions.

One of the biggest challenges to industrial composting is contamination of the incoming waste stream with non-compostable materials [4]. Developing fully compostable products and identifying the proper composting conditions requires efficient, cost-effective testing.

Scope

While previous reviews have considered biodegradation from a broad perspective, the present review focuses on methodology for laboratory-scale compostability testing to promote utilization and further method development. Ruggero, et al. reviewed 20 years of research including aerobic composting and anaerobic digestion, from benchtop schemes through pilot-scale operations [5]. Other authors have surveyed textile fiber biodegradation studies in compost, soil, sludge, sea water, and other environments [6, 7].

While the critical test parameters and experimental guidance consolidated herein are applicable to diverse composting feedstocks, special attention is given to assessment of textile compostability based on ultimate biodegradability or mineralization measurements at lab scale that could support alternative waste management practices for waste textiles beyond landfilling and incineration. Discussion of relevant research using lab-scale composting apparatus is presented for a thorough understanding of testing best practices and limitations. Studies of textile biodegradation in other environments beyond composting are also mentioned where they provide useful context. Aside from controlled composting in managed facilities, there is interest in ready biodegradation of textiles and textile fibers that are accidentally or intentionally deposited in the environment. The following discussions on critical factors impacting the use of lab-scale composting test methods will help product developers, consumers, waste management professionals and regulators make informed decisions towards sustainable waste management practices in general and provoke attention on using these practices for textile materials in particular.

Test Methods for Aerobic Biodegradation

Aerobic Degradation in Compost

There are multiple standard, modified, and novel methods for evaluating aerobic degradation in compost. International standards specifically address aerobic biodegradation in "controlled composting conditions." Most of these methods are intended to evaluate compostability of plastics and are maintained by committees dedicated to plastic materials. They are used as part of larger certification schemes—also

limited to plastics—that enable products to be labeled as compostable.

The same polymers used in plastic films and cast items can be extruded as fibers for textile applications. The positive control material for compostability testing is typically cellulose, a natural polymer also used in textiles. Cellulosic textiles include cotton and linen as well as regenerated fibers such as viscose rayon. The principles of laboratory-scale composting are the same for textiles as for other plastics, although modifications may be required to accommodate the specific biodegradation profiles of individual materials.

The most frequently referenced standard in the aerobic biodegradation category is ISO 14855. Originally a single document, it is now published as two parts. ISO 14855-1 is titled Determination of the ultimate aerobic biodegradability of plastic materials under controlled composting conditions—Method by analysis of evolved carbon dioxide—Part 1: General method. As of this writing, the current edition is ISO 14855-1:2012. The scope states, "The material is exposed to an inoculum which is derived from compost. The composting takes place in an environment wherein temperature, aeration and humidity are closely monitored and controlled" [8]. The specifics of monitoring and control are of particular interest and the approaches taken by various researchers are detailed in the sections below. CO_2-free air is supplied to a composting vessel containing the test specimen. The CO_2 exhausted from the vessel is measured using titration of hydroxide traps, infrared (IR) analysis, or gas chromatography (GC). Percent biodegradation is calculated from the initial carbon content of the specimen and the CO_2 evolved. Replicates, blanks, and controls provide verification of results [8].

ASTM D5338 states that it is equivalent to ISO 14855; however, it is comparable only to Part 1 of the ISO method and does not include an option to replace mature compost with vermiculite as the inoculum [9]. The ASTM and ISO standards are technically very similar yet provide complementary descriptions and figures. Taken together, they offer a more thorough explanation of the principles and intended testing procedures. ASTM D5338-15(2021) is titled Determining Aerobic Biodegradation of Plastic Materials Under Controlled Composting Conditions, Incorporating Thermophilic Temperatures. Originally published in 1992, the reference to thermophilic temperatures was added to the title in 2011.

Part 2 of the ISO method (ISO 14855-2) is Gravimetric measurement of carbon dioxide evolved in a laboratory-scale test. According to the introduction, "Compared with ISO 14855-1, the amounts of compost inoculum and test sample used in this document are one-tenth the size." This method allows for adjustment of humidity, aeration, and temperature to determine an optimum rate of biodegradation [10]. Aside from the smaller scale, Part 2 of the test uses sea sand, along with mature compost and the test specimen, in the composting vessel to retain moisture. As in Part 1, CO_2-free air is supplied to the vessel. Instead of direct measurement by IR or GC, or absorption and titration, evolved CO_2 is measured by trapping and determining change in mass of the trap.

ISO 21701 Textiles—Test method for accelerated hydrolysis of textile materials and biodegradation under controlled composting conditions of the resulting hydrolysate is based on a method developed by the FITI Testing and Research Institute in the Republic of Korea. This method was created specifically for synthetic textiles and calls for exposure to high heat and humidity prior to the procedure described in ISO 14855-1. The initial exposure allows for accelerated hydrolysis, reducing molecular weight to make the polymers more susceptible to biodegradation [11].

Among the aerobic composting procedures referenced in the Ruggero review, ISO 14855-1, ISO 14855-2 and ASTM D5338 were used to determine the ultimate biodegradability of tested materials. ISO 20200 (lab-scale), ISO 16929 (pilot-scale), EN 14806 (lab-scale, packaging), and EN 14045 (pilot-scale, packaging) were also mentioned for determining the degree of disintegration. The main differences among standards are listed as "temperature, the duration and the scale of the simulation, and the composition of the matrix to which the test material is exposed." The review also noted that some authors "do not comply with any specific protocol" [5]. In the research reviewed for this paper, even those authors who cited standard methods used an array of variations both within and beyond the boundaries of standard procedures.

Most of the existing research used standard composting test methods, or slight variations, simply as tools to characterize biodegradation behavior of materials [12–18]. In a few cases, emphasis was on improving the methods themselves. Pickens developed an automated composting system within the scope of ASTM D5338-98(2003) as a thesis project. The system included data acquisition and logging as well as air flow and temperature control [19]. Other researchers sought to overcome the challenges of long test duration and costly equipment. They used a set of Bartha respirometers, also known as biometer flasks, as the combined composting vessels and CO_2 traps [20, 21].

Aerobic Biodegradation in Other Media

Studies and standards related to biodegradation in soil and aqueous media are useful resources for optimizing testing in compost. The chemical conversion process is the same and the same measurement techniques can be used although results vary based on environmental conditions.

ASTM D5988 is a standard test method for Determining Aerobic Biodegradation of Plastic Materials in Soil. Like the standard compost tests, ASTM D5988 also uses

$Ba(OH)_2$ or KOH to trap and quantify CO_2 as an indicator of biodegradation [22]. Several studies specific to biodegradation of textiles were performed by Cornell University researchers using ASTM D5988 to evaluate various cotton fabrics. Li, et al. compared results for cotton and polyester fabrics tested according to ASTM D5988-03 to those for the same samples buried in full-scale composting windrows. The ASTM D5988 medium was "natural soil" [23]. More recently, the researchers used ASTM D5988-12 to study cotton fabrics with various finishes over 154 days in a soil/compost mixture [24]. The first study found that all samples were more degraded in windrows than in laboratory testing based on weight loss. Results are discussed further in the Cotton section.

Aqueous biodegradation tests using activated sludge inoculum are based on the "Sturm test," first described by R.N. Sturm in 1973 [25] and standardized as OECD (Organisation for Economic Co-operation and Development) 301B [26] and ISO 7827 [27]. The air supply and CO_2 trapping mechanisms are directly transferable to testing in compost medium.

ASTM D5864 describes Determining Aerobic Aquatic Biodegradation of Lubricants or Their Components. The method includes a detailed description of a CO_2 scrubbing apparatus that is only vaguely referenced in the composting standard [28]. There are two related ISO tests: ISO 14851 Determination of the ultimate aerobic biodegradability of plastic materials in an aqueous medium—Method by measuring the oxygen demand in a closed respirometer [29] and ISO 14852 Determination of the ultimate aerobic biodegradability of plastic materials in an aqueous medium—Method by analysis of evolved carbon dioxide [30].

Massardier-Nageotte, et al. used ISO 14851 to evaluate aerobic biodegradation of polymer films. In this test, evolved CO_2 is absorbed by a NaOH solution and O_2 consumption is measured via the pressure or volume change in the respirometric flask [31]. Weytjens, et al. described the procedure for producing CO_2-free air for the OECD 301 test. The paper also describes the collection of evolved CO_2, including rotation of bottles. The authors explained, "These extra details are given since they are probably important in the efficient recovery of CO_2 in the absorber bottles" [32]. Pettigrew, et al. used the OECD 301B test as well as the ASTM D5338 compost test to study degradation of polycaprolactone. The authors observed that the Sturm test is "ideal" for evaluating biodegradability of low molecular weight polymers but is often extended to high molecular weight polymers for lack of better alternatives. One aim of the group's research was to investigate ways to improve material preparation and test reproducibility for higher molecular weight polymers [15].

Composting Vessels

ASTM D5338 calls for at least 12 composting vessels, each with a volume of 2 to 5 L. No specific description is provided, though it is clear that some mechanism must allow for air flow in and out of the vessels. A schematic drawing in the standard suggests the vessels resemble round-bottom flasks with air inlet at the bottom and air outlet at the top. The 12 vessels are intended for one test material, blank, positive control, and negative control, each in triplicate [9]. The exact volume of test medium and sample depends on density, but the test method specifies the mixture should fill no more than three-quarters of the vessel. This allows for thorough aeration by manual shaking. The test is performed in dark or diffuse light [9].

As in the ASTM method, ISO 14855-1 calls for composting vessels with a minimum volume of 2 L, though indicates that smaller volumes may be suitable for screening purposes. The ISO standard includes a more detailed description of the composting vessels than the ASTM standard. Vessels are defined as "glass flasks or bottles that allow an even gas purge in an upward direction" [8].

ASTM D5988 does not require the continuous air flow specified in ASTM D5338. Instead, each specimen system is contained in a separate air-tight incubator. The standard suggests an internal volume of 2 to 4 L, "such as 150-mm desiccators." The incubators are opened for 15–60 min every few days or weeks to refresh the air inside [22]. Smith, et al. used desiccators as specified, with compost on the bottom and the CO_2 trap and humidifying water on a perforated plate above the compost. Each desiccator was sealed except to take daily or weekly titrations [24]. Fig. 2 illustrates some vessels described in literature for compostability testing with no air flow.

Several papers cited standard compostability test methods with no additional details [14, 15, 17]. One that referenced ISO 14855 (1999), noted only that air was blown into composting vessels at the bottom and that vessels were manually shaken to ensure aeration [18]. Most of the studies that cited standard compostability test methods and reported apparatus details used glass vessels smaller than the 2-L minimum specified in ASTM D5338, ISO 14855-1, and ASTM D5988. ISO 14855-2 was specifically developed to use 90% less compost and sample and suggests 500 mL as a suitable vessel volume [10]. Vessels of 0.5- and 1-L were prevalent in literature. Table 1 summarizes lab-scale composting studies in order of the increasing vessel size used. The vessels described in standard methods are listed in bold. Most studies cited these methods but performed them with various modifications, including vessel volume. Some studies did not indicate a method in their work or used novel procedures.

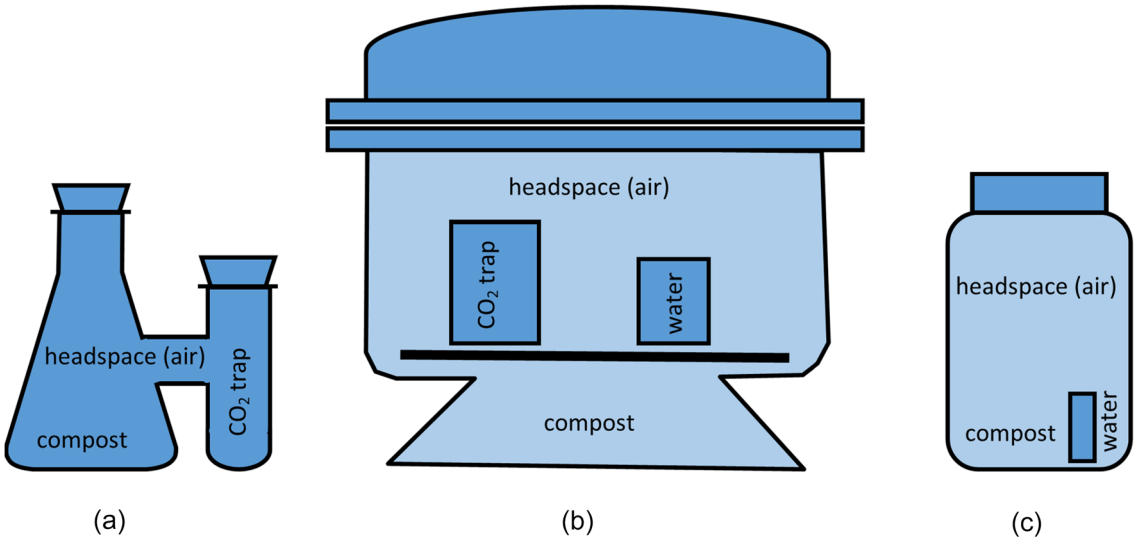

Fig. 2 Various degradation vessels for static testing (no airflow): **a** biometer flask, **b** desiccator, and **c** canning jar

Table 1 Composting Vessels for Lab-scale Testing

Standard Method Cited	Vessel Volume (L)	Reference
ASTM D5338	0.250	[20]
ASTM D5988	0.473	[12]
ISO 14855-2:201	**0.500**	[10]
N/A	0.500	[15]
ASTM D5338-98 (2003)	0.500	[19]
N/A	0.700	[35]
ASTM D5338	1	[36]
ASTM D5338	1	[16]
N/A	1	[37]
ASTM D5338	1.9	[12]
ISO 14855-1:2012	**≥ 2 (smaller for screening)**	[8]
ASTM D5338-15(2021)	**2–5 (smaller for screening)**	[9]
ASTM D5988-18	**2–4**	[22]
ASTM D5338	6	[13]
ASTM D5338	9	[38]
N/A	10–200	[39]
ASTM D5338	not specified	[14]
ASTM D5338	not specified	[15]
ASTM D5338	not specified	[17]
ISO 14855 (1999)	not specified	[18]
ISO/DIS 14855-1	not specified	[34]
ISO/DIS 14855-2	not specified	[34]
ISO 14855-2	not specified	[40]
ASTM D5988	not specified	[23]
ASTM D5988	not specified	[24]

Standard test method specifications are shown in **bold**

da Silva, et al. noted that "long duration and costs related to equipment and operations are the main disadvantages" of standard composting methods like ASTM D5338 and ISO 14855. The authors used biometer flasks to address the challenge of costly apparatus. As in ASTM D5988, the CO_2 trap was contained in the same vessel as the compost/sample mixture and there was no active air flow during the test [20]. The flask volume was not specified, but biometer flasks are readily available with 250 mL capacity.

Kunioka, et al. also commented on the practical challenges of large testing volumes, going so far as to state, "No company and organization can completely follow this ISO [15855-1] method in Japan." The need for a smaller, simpler method led to development of the Microbial Oxidative Degradation Analyzer (MODA), which was the basis for ISO 14855-2. The authors explain that a committee considered the optimal measurement conditions and reproducibility [34].

One study used 473-mL canning jars for a down-scaled version of ASTM D5988. The jars were fitted with gray butyl stoppers for headspace sampling. IR analysis was used to measure CO_2 evolution, so no chemical trap was required. A small scintillation vial of water inside the jar provided moisture [12]. For ASTM D5338, the same researchers used 1.9-L composting jars, each equipped with an air inlet tube near the bottom and an outlet tube in the lid, both sealed in place with epoxy [12].

In addition to an ASTM D5338 test with unspecified vessels, Pettigrew, et al. performed an additional study using 500-mL three-neck, round bottom flasks. Each flask had a stirring shaft, a condenser, and an air sparger. Flasks were connected to a CO_2-free air inlet and CO_2 trapping bottles

[15]. It is not clear if the same vessels were used for the ASTM D5338 test.

Pickens envisioned his automated system leading to expanded research in biodegradable materials. Specifically, he suggested, "developing and testing new biodegradable plastics for a variety of applications with better lifecycle characteristics that are robust during their intended use but degrade rapidly after the intended use of the product" and "developing techniques to enhance the biodegradation process to increase the biodegradation rate in which biodegradable plastics converts to biomass." The system uses 12 500-mL Erlenmeyer flasks as composting vessels. The air inlet is a stainless-steel tube that passes through a rubber stopper and below the compost [19].

Among the studies using 1-L glass composting vessels, designs varied. In one case, the vessels are described only as 1-L jars for ASTM D5338 [16]. Another specified the vessels were "air tight" with the CO_2 trap placed directly on the compost/sample mixture [36]. This suggests that there is no air flow in or out of the vessel, similar to the apparatus for ASTM D5988. A third study did not claim to follow ASTM D5338 but set up a "micro-composting experiment" using 1-L Quickfit spherical wide-neck flasks. Twelve vessels were split into two groups of 6, one group left open, and one fitted with multi-socket flange lids. Air was provided to the bottom of the flasks [37]. Round flasks were used in at least two studies, and possibly others [15, 37]. Shape is not specified in the standard methods, but ASTM D5338 includes an illustration of round composting vessels [9]. Organic Waste Systems (OWS) in Ghent, Belgium also uses round vessels for commercial compostability testing according to several standards. Fig. 3 Illustrates several of the composting vessels mentioned in this section. All vessels have at least one air inlet and one exhaust path though most studies did not provide detailed schematics of connections, tubing, or air path.

A couple studies described performing ASTM D5338 with vessels larger than specified in the standard. One reported using a set of 6-L glass bioreactors for testing in thermophilic composting conditions. Other portions of the study use 1.2-L bioreactors for mesophilic testing. A set of 16 bioreactors included two sets of blanks in duplicate and two samples, positive control, and negative control, each in triplicate. Design and connection of the vessels is not described. Vessels were disconnected for weighing and water was manually added and mixed before reconnecting to the air inlet and outlet [13]. Another system was unique in its use of stainless-steel composting vessels in place of glass. Seven 9-L reactors with a surface area to volume ratio of 26.5:1 were used, with one left empty for background headspace readings. Mesh screens were placed below and, optionally, above the compost. The air inlet was below the bottom screen and outlet was in the headspace above the compost. The paper explains that the apparatus "has the goal of providing the user a robust and valuable research tool to explore a wide range of research questions, from operational

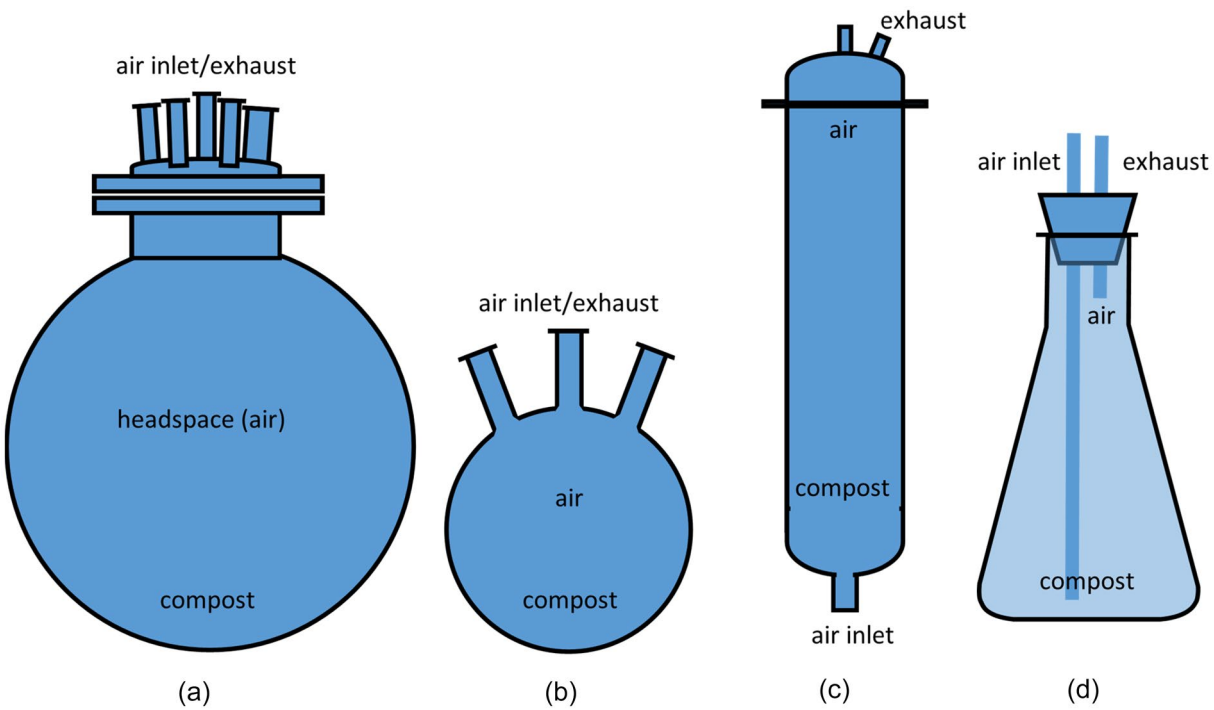

Fig. 3 Various degradation vessels for aerobic testing (with controlled air inlet and exhaust): **a** spherical flask with multisocket flange lid, **b** three-neck round bottom flask, **c** reaction column, and **d** Erlenmeyer flask

to molecular levels, related to the science and engineering of composting. The system was developed to enable high flexibility for simulating heating and aeration conditions as core controlling elements of composting processes" [38].

The largest vessels were for research focused on optimizing the composting vessel for a laboratory-scale system. The system did not resemble those in standard methods. Modeled volumes ranged from 10 to 200 L. The large volume was in part to allow biological self-heating from compost to maintain the desired temperature without external heating elements. A relatively low surface area-to-volume ratio was also selected to minimize convective heat loss though the vessel wall. The authors reported: "A volume of 50 L seemed suitable for a small-scale composting reactor that treats corn silage and cow manure and is insulated by 10 cm of polyurethane." Fiberglass, wood, and polyvinyl chloride (PVC) reactor walls were considered preferable to aluminum and stainless steel. "Materials with lower densities and smaller conductive coefficients should be used for building small reactor systems." The vessel was cylindrical with a screen and air inlet at the bottom [39]. While self-heating is not a critical property for lab-scale tests, the authors' observations regarding heat loss are relevant to any size system. The only other non-glass vessel was a "plastic cuboid container" with a total volume of 0.7 L [35].

A previous review article described work with laboratory-scale biodegradation vessels ranging from 100 mL to a few liters. The reference cited for the 100-mL test was a study conducted in aqueous media. There was no further description of vessels and neither volume nor design were among the parameters identified as main differences [5]. Pilot-scale and full-scale studies have not been included in the present review, although a few are mentioned in sections where they provide relevant insight for lab-scale testing.

Discussion

Compostability certification schemes require standardized testing in 2–5 L vessels, but the literature suggests that smaller vessels can provide adequate results. Most of the surveyed literature used vessels in the range of 0.5–1.0 L. The ISO 14855-2 test also acknowledges the preference for vessels in this size range.

Down-scaling further poses challenges. Small vessels still require adequate headspace for gas exchange and to allow mixing of contents. Very small inoculum volumes dry quickly and may not have sufficient microbial populations to support degradation over several weeks or months. Standard lab tests already use a much higher sample-to-inoculum ratio than that used in industrial composting environments. Reducing inoculum volume means further increasing sample concentration or producing smaller CO_2 quantities that may be difficult to measure accurately. Additional research is needed to validate small-scale compostability testing.

Regardless of size and shape, most biodegradation vessels include the same elements. Air inlet is usually near the bottom, with exhaust from the headspace above the compost. Round vessels allow easier mixing by simple shaking.

Test Temperature

Unlike the other studies included in this review, Wang, et al. depended on biological self-heating to maintain the required temperature. No specific temperature is mentioned, but considerable attention is given to factors impacting the temperature. Microbial activity can create *too* much heat, inhibiting further activity. Aeration and evaporation can lower temperatures. Heat is also lost through vessel walls. This is especially true for small-scale systems with a high surface area-to-volume ratio. Thermal models were developed to determine an optimal balance of biological heat production from degradation, sensible heating of reactor system, sensible heat of input and output gas, conductive and convective losses through reactor wall, and latent heat of water evaporation. Simulations found that heat was lost through reactor walls and through evaporation. Larger volumes (> 50 L) were recommended for sustained self-heating. Smaller systems should incorporate vessel wall materials with lower densities and conductive coefficients [39].

Standard composting test methods are based on thermophilic temperatures. ASTM D5338, ISO 14855-1, and ISO 14855-2 all call for 58 °C ± 2 °C for the duration of the test. Most researchers reported adhering closely to this specification [12, 13, 16, 18, 20, 34, 36, 40]. Those who did not address temperature are assumed to have followed the standard as written [14]. A few studies used slightly lower temperatures, still within the thermophilic range [15, 17, 19, 35]. Table 2 summarizes composting studies according to the temperature used, in order of decreasing temperature. Studies that used the ASTM D5988 standard were conducted at mesophilic temperatures (20–28 °C) [12, 13, 23, 24]. Only one paper referenced an intermediate temperature of 34 °C (see Table 2) [37].

Some studies looked at the impact of various temperatures or temperature control on biodegradation. da Silva, et al. performed tests at both 58 °C ± 2 °C and 28 °C ± 2 °C. After about eight days, the higher temperature had a statistically significant advantage in CO_2 production. The authors attributed this to the higher activity of thermophilic microorganisms. They noted that previous studies with biometer flasks for biodegradation testing have used 20 °C or 28 °C temperatures "without a detailed explanation of such choice." The cited works are based on degradation in soil, so it is likely these temperatures were consistent with the applicable standards. The authors suggested that 58 °C is also a suitable temperature for use with the biometer flasks and particularly preferable for polymers with low degradation rates. The higher temperature supported faster

Table 2 Composting Temperature for Lab-scale Testing

Standard Method Cited	Temperature (°C)	Temp Control	Reference
ASTM D5338-15(2021)	58 ± 2	**water bath (or other means)**	[9]
ISO 14855-1:2012	58 ± 2	**not specified**	[8]
ISO 14855-2:2018	58 ± 2	**not specified**	[10]
ASTM D5338	58 ± 1	incubator	[12]
ASTM D5338	58	not specified	[20]
ASTM D5338	58	not specified	[13]
ASTM D5338	58 ± 2	thermostatic oven	[36]
ASTM D5338	58	incubator	[16]
ISO 14855 (1999)	58	incubator	[18]
ISO 14855-1	58	not specified	[34]
ISO/DIS 14855-2	58 & 70	ribbon heater	[34]
ISO 14855-2	58	ribbon heater	[40]
ASTM D5338	35-58C	not specified	[15]
ASTM D5338-98 (2003)	56.5	water bath	[19]
N/A	45 & 55	not specified	[15]
ASTM D5338	55 ± 3	oil bath	[17]
N/A	50	incubator	[35]
N/A	34	water bath	[37]
N/A	various	water bath	[38]
ASTM D5338	not specified	not specified	[14]
ASTM D5988-18	**20–28 ± 2**	**chamber or cabinet**	[22]
N/A	28	not specified	[20]
ASTM D5988	27	incubator	[12]
not specified	25	not specified	[13]
ASTM D5988	not specified	not specified	[23]
ASTM D5988	not specified	not specified	[24]

Standard test method specifications are shown in **bold**

biodegradation. There was no evidence that loss of microbes more suited to mesophilic temperatures had a negative impact on overall biodegradation [20].

Kunioka, et al. used ISO 14855-2 at 58 °C and 70 °C to biodegrade polylactic acid (PLA). Degradation started more quickly at the higher temperature but did not reach the same degree of biodegradation achieved at 58 °C. Other trials within the experiment were performed at the standard 58 °C. DiMauro, et al. also evaluated biodegradation at two temperatures; however, results at the two temperatures cannot be compared directly because of numerous other differences between the methods, including volume, air flow, and duration. The mesophilic test (25 °C) resembled the ASTM D5988 test with a closed system. The thermophilic test (58 °C) followed ASTM D5338 [13].

The system designed by Oazana, et al. included three different heating modes. Mode #1 utilized pre-set heating rate, maximum temperature, and duration. An increase of 5 °C per day up to 60 °C was given as an example. This mode allowed a natural progression from mesophilic to thermophilic phase as well as "snapshots" at various temperatures. The system was programmed to move to the next step only at the prescribed interval *and* when both water bath and compost reached the set point. The bath reached the set point more quickly than compost. [38] Mode #2 allowed for self-heating and used the compost temperature to control bath temperature. The bath was maintained at a set difference below the compost temperature. As an example, a difference of 0.8 °C or 0.5 °C allowed the compost to reach a maximum temperature of 72 °C. The authors observed that this mode could control for heat loss through vessel walls [38]. As others showed, a high-surface-to-volume ratio can lead to large losses [39]. Mode #3 used carbon dioxide levels to determine temperature [38].

Pettigrew, et al. did not provide details of the ASTM D5338 test except in a figure caption that described a temperature profile intended to simulate an "intensive aerobic composting process." The incubation temperature was held at 35 °C for day 0–1, 58 °C for days 1–5, 50 °C for days 5–28, and 35 °C for days 28–45. Confirmation tests intended to simulate a real composting environment were performed at 45 °C and 55 °C. Isothermal conditions were selected to simplify kinetic analysis. The authors concluded that "higher temperatures that do not kill microorganisms or denature enzymes will result in higher metabolic activities."

This confirmed a generally accepted rule but is extended to suggest that biodegradation of certain materials may be modeled by the Arrhenius equation relating temperature and reaction rate (Eq. 2). Studies done at high temperatures to accelerate the test could be used to predict biodegradation rates at more realistic temperatures [15].

$$k = Ae^{-Ea/RT} \tag{2}$$

where k = rate constant (1/s), A = Arrhenius frequency factor (1/s), E_a = molar activation energy (J/mol), R = universal gas constant (J/(mol*K)), and T = absolute temperature (K).

An incubator or oven was the most cited means of controlling temperature [12, 16, 18, 35, 36]. Water baths were also used [19, 37, 38] as suggested in ASTM D5338 [9]. One paper observed that using a water heater instead of a direct water temperature controller reduced accuracy [19]. One study used an oil bath [17], another group used a ribbon heater, [34] and several did not indicate the mechanism for temperature control (see Table 2). The Annexes of ISO 14855-2 provide two examples of temperature control. Annex A describes a thermostatic incubator containing humidifying water and the composting vessel. Annex B explains a system of electrically heating the composting vessel. This arrangement is designed for extended tests without the maintenance of a water bath. It also allows for easier handling since the vessels are not contained in an incubator [10].

Discussion

Temperature is the area of least variation among standards and studies. It is well established that key organisms thrive at thermophilic temperatures, with 58 °C generally accepted as the representative temperature. While full-scale composting goes through various temperature phases, several studies have shown that a steady thermophilic test produces comparable results.

Incubators are most often used for temperature control, perhaps for simplicity. For chemical trap systems, the entire system can be maintained at a set temperature with minimal heat loss and no concerns about evaporation. Systems with direct measurement of CO_2 require more elaborate arrangements because analytic devices are typically located outside the incubator and may require a condensation step to remove water from the air prior to analysis.

Test Duration

One of the most variable parameters among degradation tests in the literature is the length of the test. Table 3 summarizes composting studies in order of increasing test duration. ASTM D5338 [9] and both parts of ISO 14855 [8, 10] indicate a standard test of 45 days but allow for longer. Labeling standards ASTM D6400 [41] and ISO 17088 require 90% conversion of organic carbon to carbon dioxide within 180 days to be considered "compostable in aerobic municipal and industrial composting facilities" [42]. The slower ASTM D5988 soil test does not specify duration but notes that a test must be regarded as invalid if the positive control exhibits less than 70% biodegradation after 6 months. The precision study for this method lasted 4 months (120 days) [22].

Of the research reviewed, three studies used the standard 45-day test [15, 18, 19]. The shortest tests focused on optimizing the process rather than final degradation of test materials. One set of investigators used 15-day tests to investigate various aspects of an automated system, then conducted a full 45-day test [19]. Another ran a 480-h (20 day) simulation of compost biodegradation [39]. da Silva set out to create an accelerated test and reported results after 28 days. This paper indicated the test time was reduced from 45 to 28 days with yeast as a biostimulant. Further time reduction was predicted with improved control of experimental variability [20].

Many tests, including those performed on textile substrates, lasted longer than 45 days [12, 13, 16, 18, 23, 24, 34, 36–38]. A previous review found a similar duration range for tests of biodegradation in aerobic composting, from 28 to 130 days [5]. Longer test times are inconvenient for efficient data collection in the laboratory. More importantly, materials that require extended composting time or special treatment may not be suitable for processing by current industrial composting systems.

The proposal for a new ISO method to evaluate degradation rate of textile materials in compost includes a statement that "test duration for biodegradation [of synthetic fibers] shall be longer than plastics and biology-based or natural fibers." This is explained as due to "high molecular weight, degree of crystallinity and orientation occurred during the spinning." No further data or references are cited [43]. Project leader Hyun Jin Koo of FITI Testing & Research Institute clarified that the test should not be run longer than one year because the compost will become exhausted.

Correlation between lab and field tests is important for practical implementation of composting practices and biodegradation time has been a major area of concern and confusion. A recent report from Biocycle and BPI says, "Disintegration is the test to focus on for real world composting and the time-frame conversation, not Biodegradation." The report argues that disintegration is a property that can be visibly evaluated in the field while biodegradation is an invisible process [4]. However, disintegration could be achieved by milling a material to a very small size, and disintegration alone does not guarantee that degradation has occurred.

Table 3 Composting Time for Lab-scale Testing

Standard Method Cited	Duration (days)	Test Material	Reference
ASTM D5338-15(2021)	**45 (may be extended)**	**plastic**	[9]
ISO 14855-1:2012	**45**	**plastic**	[8]
ISO 14855-2:2018	**45 (may be extended up to 6 months)**	**plastic**	[10]
ASTM D5988	**not specified**	**plastic**	[22]
ASTM D5338-98 (2003)	15, 45	biopolymer film	[19]
N/A	20	compost (simulation)	[39]
N/A	22	leaves and branch cuttings	[35]
ASTM D5338	28	polymers (PHB, PLA, PVOH, PET)	[20]
ASTM D5338	30	biopolymer films (PLA, PHB)	[17]
ISO 14855 (1999)	39, 45, 47, 90	cyclodextrin powder	[18]
ASTM D5338	45	polycaprolactone (PCL) powder	[15]
ISO/DIS 14855-2	45–90	PLA powder	[34]
ISO 14855-2	56	PLA powder	[40]
N/A	56	winery waste activated sludge and grape stalks	[37]
ISO 14855-1	60	PLA powder	[34]
ASTM D5338	90	biodegradable plastic mulch films	[12]
ASTM D5988	90	cotton fabric	[23]
ASTM D5338	98	eumelanin and synthetic organic electronic materials	[13]
ASTM D5338	100	flax and PHBV composite sheets	[36]
ASTM D5338	112	mulch film in nylon mesh bag	[16]
N/A	120	N/A.	[38]
ASTM D5988	154	cotton fabric	[24]
ASTM D5988	365	biodegradable plastic mulch films	[12]
ASTM D5338	not specified	polymer films (HDPE, PLA, MAH)	[14]

Standard test method specifications are shown in **bold**

Discussion

Certification schemes for compostable products are based on their ability to break down within the traditional time frame for commercial composting. Longer tests can be performed in the laboratory but may have limited relevance for real-world applications. As a biological process, it is difficult to shorten the natural composting timeframe. Various enzymes as well as yeast have been used to accelerate biodegradation processes in laboratory settings. These are discussed in more detail in the next section.

Test Medium

As ISO 14855-1 states, "mature compost is a very heterogeneous and complex material" [8]. Thus, the specific composition of compost media or inoculum are inherently variable, but some general characteristics are controllable. To standardize testing, ASTM D5338 and ISO 14855 define numerous parameters for the compost inoculum, as summarized in Table 4 [8–10].

The specifications for all three standards are nearly identical, with slightly larger or smaller ranges for some parameters. For example, ASTM D5338 and ISO 14855-1 call for a carbon-to-nitrogen ratio between 10 and 40, while ISO 14855-2 recommends a more precise 15. There are also minor differences in which parameters are required, recommended, or reported [8–10].

Compost for ISO 14855-2 is sieved to a smaller particle size than for the other standard test methods because the entire test is on a smaller scale [10]. All three standards include instructions to create a homogeneous texture by removing "large inert objects" such as glass, stones, or metal, then sieving the compost. In contrast, a later section advises preventing clumping and clogging with the addition of inert material or small wood particles [8–10]. ISO 15855-1 explains that mature compost should feel sticky and release water when pressed by hand [8]. One paper cites this description almost verbatim and references personal experience in judging that a particularly wet compost batch is in an acceptable range [18].

A research group in Japan addressed the challenge of compost variability with "controlled compost" that can be

Table 4 Standard Compost Inoculum

Parameter	ASTM D5338	ISO 14855-1	ISO 14855-2
Source	Organic fraction of municipal solid waste	Aerobic composting plant	Aerobic composting plant OR lab-made
Age	2–4 months	*2–4 months*	*2–4 months*
Particle size	< 10 mm	0.5–1 cm	< 3 mm
Inert materials	Removed manually	Removed manually	Removed manually; sea sand added
C/N ratio	10–40	10–40	*15*
CO_2 production	*50–150 mg/g volatile solids (day 1–10)*[a]	50–150 mg/g volatile solids (day 1–10)	*50–150 mg/g volatile solids (day 1–10)*
Ash content	*< 70%*	N/A	N/A
pH	*7.0–8.2*	7.0–9.0	*7.0–9.0*
Dry solids	50–55%	50–55%	*35–55%*
Porosity	*Maximally aerobic*	*Maximally aerobic*	N/A
Volatile solids	Report	≤ 15% wet solids (≤ 30% dry solids)	*≤ 30% dry solids*
Total nitrogen	Report	*Report*	*Report*
Total organic carbon	Report	*Report*	*Report*
Fatty acids	Report for pH < 7	*Report*	*Report*

[a]Values in italics are recommended, but not required, by the cited standard

stored for five years or longer at room temperature and activated as needed. "A homogeneously controlled compost with almost the same biological activity can be obtained at any time and at any place" [34]. The authors also recommended this as an option for laboratories without convenient access to a composting facility. The controlled compost was activated with the addition of water to restore biological activity [40]. Sea sand was also added as prescribed in ISO 14855-2.

Mineral Medium

ISO 14855-1 includes an option for solid inorganic mineral (vermiculite) medium in place of compost, although a compost solution is still used to activate the vermiculite. The standard says vermiculite should be used whenever biodegradation is impacted by a "priming effect" of polymer-induced compost degradation and when retrieving residual test material for analysis [8]. Priming occurs when the test sample stimulates mineralization of organic material in the compost medium. Vermiculate is easier to standardize and work.

Vermiculite also supports microbial activity and has a water content capacity comparable to that of mature compost. "Concrete" type (apparent density 80 kg/m.^{3}) expanded vermiculite in flake form is used. This is much coarser than the vermiculite readily available as a soil amendment or substitute. Detailed instructions for activation of the vermiculite with compost extract and nutrients are included in the test method. According to the standard, final biodegradation percentage and degradation rate of vermiculite and mature compost are "identical, or very similar." No reference or data is provided to support this statement [8]. One study used a combination of compost and vermiculite as the medium for compostability testing [12].

For ISO 14855-2, mature compost is mixed with inert sea sand to hold humidity and microorganisms. Sand particle size is 35–20 mesh (approximately 0.8–0.5 mm sieve opening) [10]. Maintaining moisture is especially critical for this method because of the smaller volume (and higher surface area to volume ratio).

Biostimulant

Da Silva, et al. incorporated yeast extract into soil as a biostimulant. The object of this study was to develop an accelerated test. The yeast extract supplied proteins, vitamins, and minerals to stimulate microbial activity. The authors found that biostimulation with yeast extract was able to differentiate and rank the biodegradability of polymers with similar outcomes to tests without yeast, at an accelerated rate. Degradation was higher at all time points analyzed. Final values after 28 days were 37% degradation without yeast extract and 53% with extract [20]. The original Sturm test also used yeast extract in combination with raw sewage for evaluation of aqueous biodegradation [25].

Work by Biyada, et al. included inoculation of textile waste compost mixtures with three species of microorganisms in isolation and combination. *Streptomycetes cellulosae* was particularly effective at nitrogen biodegradation. *Achromobacter xylosoxidans* degraded lignocellulosic compounds, including those found in textile materials. *Serratia liquefaciens* was also identified as a ligninolytic strain, particularly

useful for breaking down azo dyes in textile waste. The species had a further synergistic effect when introduced as consortia. The authors attribute this to specialized complementary roles such as attacking the complex substrate and providing essential nutrients [44]. This also suggests that compost selection impacts test results and correlation to full-scale processes.

Kunioka, et al. tested PLA powder biodegradation using *Proteinase K* enzyme in buffer solution (without compost). The authors determined that degradation did not occur in the buffer without enzymes and that smaller particle sizes were more rapidly degraded by the enzyme solution. The paper also includes tests in compost medium but did not provide correlation between enzymes in solution and those existing in or added to compost [34].

The Salmon research team showed that cellulosic textiles could be degraded by cellulases to various glucose-containing "syrups" and "slurries" containing microcrystalline fiber fragments [45, 46]. These pourable and pumpable liquid mixtures had particle sizes less than 2 mm, which is the "disintegration" size specified in ASTM D6400, [41] and could therefore be mixed with or sprayed on compost. This indicates that cellulases can also act as a pretreatment or "biostimulant" to enhance material compostability.

Discussion

Compost inoculum is an important source of variability both in terms of microbial population and other physiochemical properties. A standard compost recipe has been suggested to minimize this variability but has not been widely adopted. As seen in Table 4, several properties are already defined for standardized testing. Published research contains surprisingly little detail or discussion of these properties so it is not clear if all studies adhere strictly to these specifications. Moisture content (or the inverse, dry solids) is widely cited as a critical criterion. Carbon/nitrogen ratio is less often reported or controlled.

The complex heterogenous composition of compost makes it difficult to retrieve partially degraded specimens for analysis and to identify chemical biproducts of the degradation process. Often, a toxicity test is conducted using seeds or earthworms to determine if the resulting compost is suitable for agricultural applications. The activated vermiculite option in ISO 14855-1 is one option to address these issues. Smaller vessels may also be beneficial in this area because there is less material to sift through and likely a high concentration of any residual components. General observations from the Chair of ASTM subcommittee D20.96 on Environmentally Degradable Plastics and Biobased Products Kelvin Okamoto are that the same materials biodegrade in all composting environments. The rate of degradation may vary with inoculum, temperature, or other conditions of both lab and commercial composting. Based on this, the importance of selecting or standardizing inoculum depends on the goals of each research project.

Compostability Metrics

Ruggero, et al. defined the three main types of biodegradation evaluation as (ultimate) biodegradability, disintegration, and compost quality. Biodegradability is further broken down by monitoring methodologies. Of the studies considered in that review, 19 used CO_2 measurements to evaluate biodegradation, 19 used mass loss, 14 used spectroscopy, and 19 used visual analysis. Of the CO_2 measurement group, 58% used cumulative respirometry measurements, 21% used direct respirometry measurements, 16% use gravimetric respirometry, and 5% (1 study) used an OxiTop® respirometry system [5].

Ultimate Biodegradability

The most objective measure of biodegradation, and the one used in ASTM D5338 and ISO 14855, is ultimate biodegradability. OECD 301 defines ultimate biodegradation as "The level of degradation achieved when the test compound is totally utilised by micro-organisms resulting in the production of carbon dioxide, water, mineral salts and new microbial cellular constituents (biomass)" [26]. The process is also called mineralization. It can be determined by CO_2 evolution, O_2 consumption, or direct measurement of organic carbon.

The standard composting tests measure CO_2 evolution [8–10]. The exact process and apparatus for this measurement is discussed in other sections. CO_2 evolution is compared to the theoretical yield if all carbon in the sample is converted to CO_2. Equation 3–9 is an example calculation of theoretical CO_2 evolution from a 100-g cellulose ($C_6H_{10}O_5$) sample. Carbon content of more complex materials may be determined by elemental analysis.

$$C_6H_{10}O_5 + 6\,O_2 \rightarrow 6\,CO_2 + 5\,H_2O \tag{3}$$

$$\begin{aligned} &\mathrm{C_6H_{10}O_5\ molecular\ mass} \\ &= (6 \times 12.011\ \mathrm{g/mol}) + (10 \times 1.008\ \mathrm{g/mol}) \\ &\quad + (5 \times 15.994\ \mathrm{g/mol}) = 162.116\ \mathrm{g/mol} \end{aligned} \tag{4}$$

$$\begin{aligned} \%\,\mathrm{C} &= (100\% \times (6 \times 12.011\ \mathrm{g/mol})) \\ &\quad /(162.116\ \mathrm{g/mol}) = 44.5\% \end{aligned} \tag{5}$$

$$\mathrm{C} = 100\ \mathrm{g} \times 44.5\% = 44.5\ \mathrm{g} \tag{6}$$

$$\begin{aligned} CO_2 \text{ molecular mass } &= 12.011 \text{ g/mol} \\ &\quad + (2 \times 15.994 \text{ g/mol}) \\ &= 43.999 \text{ g/mol} \end{aligned} \tag{7}$$

$$\%\,C = 100\% \times (12.011 \text{ g/mol})/(43.999 \text{ g/mol}) = 27.3\% \tag{8}$$

$$CO_2 = 44.5 \text{ g}/27.3\% = 163 \text{ g} \tag{9}$$

One study measured both CO_2 and O_2 while testing according to ISO 14855 [18] .Oazana, et al. also used both CO_2 and O_2 sensors to monitor their novel compostability test [38].

ASTM D5988 for biodegradation in soil measures CO_2 evolution and includes an alternate option for measuring O_2 consumption. [22] Surveyed research that cited this standard measured CO_2. [12, 23, 24]

Among the aqueous medium tests, ASTM D5864 measures CO_2 evolution [28]. ISO has separate tests—ISO 14851 for O_2 demand [29] and ISO 14852 for CO_2 evolution [30]. OECD 301 also includes several subtests with different assessment metrics. OECD 301A and 301E are assessed by measuring dissolved organic carbon (DOC); 301B uses CO_2 evolution; 301C and 301F use O_2 consumption; and 301D measures dissolved O_2 [26] .Eq. 10–11 illustrates the O_2 demand for an 100-g cellulose sample and the stoichiometry shown in Eq. 3.

$$\begin{aligned} O_2 \text{ molecular mass } &= 6 \times (2 \times 15.994 \text{ g/mol}) \\ &= 191.928 \text{ g/mol} \end{aligned} \tag{10}$$

$$\begin{aligned} O_2 = 100 \text{ g} \times (191.928 \text{ g/mol})/ \\ (162.116 \text{ g/mol}) = 118 \text{ g} \end{aligned} \tag{11}$$

Disintegration

Disintegration represents a physical rather than chemical breakdown of the specimen, though biodegradation may result in disintegration into smaller pieces. Biodegradation may also increase as pieces become smaller, with more accessible surfaces. Disintegration without degradation can result in microplastics or fiber fragments that are an increasing environmental concern [5].

The most common measure of disintegration is mass loss [23, 24, 47–49]. Theoretically an objective measure, challenges include collecting all pieces of the sample and excluding compost particles.

Visual analysis is a simple way to evaluate the degree of sample disintegration. [48, 50]. This is essentially a subjective evaluation. One paper described holes, missing fabric portions, thinning samples, and even color change. Photographs are also included [24].

According to Bruno De Wilde, Managing Director, OWS uses a digital camera and computer software to quantify the cover factor of samples mounted in slide frames. The frames are placed in compost medium and retrieved periodically for analysis.

Scanning electron microscopy (SEM) allows more detailed examination of surface and fiber morphology [23, 24, 49, 50].

In March 2022, ISO Technical Committee (TC) 38 on Textiles approved a preliminary work item for development of a test method to determine the degradation rate of textile materials under simulated composting conditions in a laboratory-scale test. The metrics are still being determined at the time of this writing. The proposed scope refers to physical degradation and potential measurements include thickness, mass, and tensile or bursting strength. The physical degradation is described as a preliminary step for exposing additional surface area of the test material for biodegradation by microbes and enzymes [43]. Only one of the papers surveyed mentioned sample thickness and tensile strength. The work addressed nonwoven PLA mulch in soil, samples intended to remain intact for extended periods before eventually disintegrating and/or biodegrading [50].

Compost Quality

Compost quality refers to the ability of plants to thrive in the compost resulting from sample degradation. Germination is a measure of the number of seedlings that emerge. Values may be reported in terms of days to germination, percentage of seeds germinating in test compost, or as a ratio between seeds germinated in test compost and those germinated in a control medium. Another way to evaluate compost quality is by determining the total biomass of plants grown from seed in test and control compost mixtures.

ASTM D6400 includes germination rate and plant biomass among the requirements for labeling as "compostable in aerobic municipal and industrial composting facilities." Specifically, germination and biomass in test sample compost must be at least 90% that of seeds in blank compost [41]. Other specifications call for a germination index above 50% [51].

Most lab studies of compostability surveyed did not include compost quality evaluation. One measured percent germination, time to germination, plant height, and root length of parsley seeds [37]. Another measured the number of seeds germinated as well as wet and dry biomass of shoots cut just above the soil line. All conditions of soil, water, light, and temperature were controlled during the 19-day phytotoxicity test [13].

Among textile composting studies, one found an acceptable germination index for corn seed in compost composed of 40%, 60%, and 80% textile waste, although germination was lower for the 80% blend than for others. The authors note that Quebec places compost from textile waste in category A, allowing its use without any risk [51]. Linen fabric also achieved mature compost after 90 days with a 73.88% germination index of cress seeds [47].

A study of sewage and textile sludge with azo dyes looked at changes in phytotoxicity over the course of composting. Lettuce growth was inhibited by initial compost mixtures containing 60% or more textile sludge. After 7 days, toxicity increased but after 60 days, mature compost derived from up to 80% textile sludge showed no inhibition of plant growth [52].

Another study focusing on the environmental impact of degraded PLA fibers found that bioplastic debris does remain in mature compost. A concentration of 1% PLA increased earthworm mortality, although additional studies were recommended to confirm the effect. PLA fiber fragments did not impact plant growth or seed production [53].

Other Metrics

Fourier-transform infrared spectroscopy (FTIR) can be used to look at changes in chemical structure of samples during degradation. This technique was used for studies of cotton [23, 24], linen [47], and PLA [49] textile compostability.

Molecular weight (M_W) decreases and molecular weight distribution or polydispersity (M_w/M_n) increases during the biodegradation process [48–50].

Thermal analysis of thermoplastic polymers can elucidate the mechanisms of biodegradation. Several studies have measured decreased glass transition temperature (T_g) as samples become biodegraded [48, 50].

Various analytical techniques have been used to characterize compost medium as well as test samples. One research group studied the microbial population [51], physio-chemical properties [51, 54], and spectroscopic changes [54] of the compost mixture. They found species capable of producing cellulase and acid/alkaline phosphatases, enzymes that support the biodegradation of lignocellulosic fiber in the textile waste tested. In one experiment, the researchers monitored temperature, moisture, pH, and electrical conductivity of compost mixtures containing textile waste. They reported previous work indicating neutral pH is one of the most important indicators of the final quality of compost and the progress of composting [51]. The pH generally declines at the start of comporting and becomes weakly alkaline as ammonia is produced, then near neutral as the active phase of biodegradation ends [2]. Another experiment used UV–Visible spectroscopy, infrared spectroscopy, and X-ray diffraction to analyze changes in textile waste mixtures during composting. The authors suggest these analyses as tools for assessing compost maturity. There is no discussion of applying them to determine if or how a textile sample is compostable. The studies were carried out over a longer period than most lab tests but found no reason to doubt the eventual compostability of textile waste [54]. Tests were performed on waste from a textile plant in Fez, Morocco, described elsewhere as high in lignocellulosic fiber; more detailed composition was not reported [55].

A review of thermogravimetric analysis (TGA) in compost research described using the technique to identify phases of compost degradation alone or in combination with other analytical methods. Among the methods mentioned are FTIR and differential scanning calorimetry (DSC) [56].

Discussion

Various studies have criticized aspects of traditional compostability testing, particularly assumptions about the measured CO_2 as a complete and accurate indicator of biodegradation. The trapping efficiency has been estimated as less than 10% in some cases. [32] Calculated CO_2 values are also impacted by theoretical carbon content determined at the start of the test, incoming air composition, microbial consumption, loss or contamination throughout the system, and accurate measurement of air flow. The general concept of CO_2 capture as a measure of biodegradation has been established and accepted for more than 50 years and at least one recent study reported it remains an "acceptable" analytical method for compostability testing [13]. Disintegration and compost quality are often evaluated in conjunction with biodegradation, but are not equivalent alternatives.

Closed laboratory systems can result in lower biodegradation due to nutrient and product removal limitations not encountered in commercial composting [15, 23]. Photodegradation is minimized in the laboratory but may be an important factor in accelerating biodegradation. On the other hand, the crosslinking effect of UV exposure can reduce real-world biodegradation [12]. "Priming," or an initial spike in microbial activity with the introduction of test specimens, is also cited as a disconnect between lab and field studies and can result in calculations of more than 100% biodegradation in the lab [18].

Air Flow

As an aerobic process, composting requires a constant supply of oxygen to support microbial activity. Knowing the flow rate out of the compost vessel is also critical to calculating CO_2 generation if a periodic sampling technique is used. On the other hand, air flow through the compost vessel

Table 5 Air Flow for Lab-scale Biodegradation Testing

Standard Method Cited	CO_2 Scrub	Flow Rate (mL/min/vessel)	Vessel Volume (L)	Ref
Compost				
ISO 14855-1:2012	**synthetic air *or* NaOH solution *or* Normal air (subtraction)**	**not specified**	**≥ 2**	[8]
ASTM D5338-98 (2003)	synthetic air (O_2 only)	200	0.5	[19]
not specified	10 M NaOH solution	60	not specified	[57]
ASTM D5338 & ISO 14855	10 M NaOH solution	40	1.9	[58]
ISO 14855-2:2018	**NaOH column (soda lime)**	**10–30**	**0.5**	[10]
ISO/DIS 14855-2	NaOH column (soda lime)	10	0.5	[34]
ISO 14855-2	NaOH column (soda lime)	10	0.5	[40]
not specified	NaOH column (soda lime)	not specified	0.25	[21]
ISO 14855-2	NaOH column (soda lime)	10	0.5	[62]
not specified	NaOH column (soda lime)	40 ± 2	1.9	[65]
ASTM D5338	NaOH column (Drierite)	40	1.9	[12]
ASTM D5338-15(2021)	not specified	not specified	2–5	[9]
ISO 14855	not specified	50–110	2–3	[66]
not specified	not specified	333–1,167	10–200	[39]
not specified	not specified	0–5,000	9	[38]
Aqueous				
OECD 301 B (1992)	**synthetic air (80% N_2/2% O_2) *or* scrubbing apparatus**	**30–100**	**2–5**	[26]
ISO 14852:2021	**synthetic air *or* NaOH column (soda lime) *or* 10 M KOH solution**	**50–100**	**not specified**	[30]
ASTM D5864-18	**10 M NaOH solution**	**50–100**	**4**	[28]
not specified	5 M NaOH solution	50–100	5	[32]
ISO 14852	10 M KOH solution	~33	not specified	[60]
not specified	5 M KOH solution	not specified	3	[59]
not specified	NaOH column (Ascarite)	50–100	6	[25]

Standard test method specifications are shown in **bold**

increases drying and cooling. Aqueous biodegradation methods are included in this section because some of the same techniques and challenges are equally applicable to composting methods (see Table 5). Anaerobic methods generally do not require airflow. In the case of ASTM D5988, an air-tight desiccator eliminates airflow in and out of the system. [22] One study using this method indicated the sealed containers were opened once per week to purge accumulated CO_2 [12].

CO_2 Scrub

Standard test methods for biodegradability call for CO_2-free air flowing into the system to prevent interference with measuring the CO_2 generated by microbial degradation of compost and test samples. ASTM D5864 is the only method to include quantitative scrubbing criteria, indicating that a suitable system will produce air with less than 1 ppm CO_2. Several standards require aerating the system overnight or 24 h to purge it of CO_2 before starting the test [26, 28, 30]. Some of the studies reviewed include no mention of CO_2-free air and a few note it with no further description or discussion [15]. At least one used ambient air, with CO_2, but the evaluation was by volume reduction and plant growth rather than by percent mineralization [37].

Synthetic Air

ISO 14855-1 and ISO 14852 offer several options for achieving CO_2-free air [8, 30]. The simplest, but potentially costly choice, is to purchase synthetic air in canisters. Laboratories can also use various commercially available filtration or adsorber apparatus. OECD 301B mentions a mixture of

CO_2-free oxygen and nitrogen from gas cylinders as an alternative to scrubbing CO_2 from air [26]. One paper reported using gas cylinders ahead of scrubbing solution for a 28-day test [32]. Another used canisters of normal air for 90 days [47]. A shorter 15-day study used research-grade O_2 [19]. A 45-day test with air flow at 10 mL/min requires a minimum of 650 L of air per composting vessel.

Chemical Absorption

A second option is a CO_2 absorption system. An example system is described in ISO 15855-1 using sodium hydroxide (NaOH) solution to remove CO_2. An optional barium hydroxide $(BaOH)_2$ trap can be included after the NaOH scrub to indicate the absence of CO_2. Any residual CO_2 reacts with $(BaOH)_2$ to form a visible solid precipitate of barium carbonate ($BaCO_3$) [8]. ASTM D5864 includes a more detailed description of a similar CO_2 scrubbing apparatus. A series of five plastic bottles containing 10 M NaOH are followed by an empty 1-L Erlenmeyer flask, one with 0.0125 M $(BaOH)_2$, and a second empty flask. The empty flasks prevent liquid from carrying over to the next vessel. The method calls for 700 mL solution in each scrubbing bottle to supply a total of 15 4-L biodegradation vessels (3 samples, plus standard and blank, each in triplicate). One additional bottle of NaOH solution is used for each additional sample (three vessels) [28].

While none of the papers reviewed described a system exactly as specified in ASTM D5864, several used some form of NaOH solution to scrub CO_2 from incoming air. Two used two bottles of 10 *N* (equivalent to 10 M) NaOH and no $Ba(OH)_2$ verification [57, 58]. One of these included an empty jug to prevent carryover. This scrubber series reduced the CO_2 concentration from 400 ppm in ambient air to about 30 ppm, still well above the 1 ppm specified in ASTM D5864. The authors note that standard methods allow the use of normal air, but a lower starting concentration should improve the accuracy of measurements [58]. Another study bubbled air through a bottle of 5 M NaOH to remove CO_2. The solution volume is not specified but the paper does state that it was sufficient for the entire 28-day test period [32].

ISO 14852 describes a system of 10 M KOH and 0.0125 M $Ba(OH)_2$. An empty flask before the biodegradation vessel is optional [30]. Calmon, et al. used four vessels of 5 M KOH and Raschig rings to maintain CO_2 at a concentration of less than 0.3 ppm. The Raschig rings increase surface area for interaction between gas and liquid. Scrubbed air was pumped through the system for 24 h before testing to purge CO_2 [59]. A study of leather degradation in aqueous medium used 10 M KOH [60]. On a molar basis, KOH is more expensive than NaOH and about as effective at removing CO_2 [61]. $Ba(OH)_2$ is less expensive than either, particularly since each mol can react with two mols of CO_2. The drawback is that the solid precipitate can clog aeration tubes and create a film that limits accessibility of the remaining solution.

Scrubbing Column

A third option for removing CO_2 mentioned in ISO 14852 is dry soda lime [30]. ISO 14855-2, the gravimetric compostability method, includes two example systems with soda lime as the CO_2 scrubbing medium. In both, compressed air is shown entering at the bottom of a 1,000 mL column of soda lime and exiting to a flow controller at the top [10]. Several studies utilized The MODA system that includes a soda lime column [34, 40, 57, 62]. In one instance an indicator was included in the column [57]. In others, the soda lime flake was combined with soda talc [34, 40]. Both are forms of immobilized NaOH. The MODA was developed by the Biodegradable Plastics Society (BPS) in Japan and is the basis of ISO 14855-2.

Soda lime was also used to scrub air entering a biometer flask [21]. Anunciado modified ASTM D5338 to use three Drierite columns for CO_2 scrubbing [12]. Soda lime contains calcium oxide (CaO), NaOH, and a small amount of KOH. Drierite is granulated anhydrous calcium sulfate ($CaSO_4$). Drierite can produce small amounts of CO_2 so an initial flush and continuous flow is required for this approach. Drierite is most often used as a desiccant and must be monitored for water saturation which will increase CO_2 production. Soda lime becomes less effective as a scrubber when dried [63].

The original Sturm test method used Ascarite, a dry form of NaOH, for CO_2 removal. The CO_2 scrubbing column was followed by 0.05 *N* (0.025 M) $Ba(OH)_2$ solution to confirm removal of CO_2 and humidify the air. A single scrubbing column was used to prepare the air supply to 10 degradation vessels [25].

Subtraction

A third option for ISO 14855-1 is included in as a note in the procedure. Normal air can be used with direct measurement of CO_2 concentration at the inlet and outlet of every compost vessel. The inlet concentration is subtracted from the outlet concentration to determine the quantity evolved by the inoculum and sample [8]. ASTM D5338 also indicates that normal air may be used if CO_2 is measured directly [9]. The figure of a direct measurement system in ISO 14855-1 includes air input with no indication of CO_2 removal [8]. One of the reviewed studies performed at OWS used the same figure [18]. De Wilde confirmed current practices at this laboratory are to use ambient air and subtract the CO_2 exhausted by blank compost vessels from that exhausted by vessels with test and control specimens.

Degli-Innocenti, Tosin, and Bastioli explicitly studied the use of normal and decarbonated air. When CO_2 present in

the incoming air is measured and subtracted, the authors concluded that "normal air does not affect the CO_2 assessment" [64].

Humidification

Sufficient moisture is critical to composting both for microbial activity and for hydrolysis [57]. Standard test methods call for about 50% dry solids (see Table 4). Biodegradation is more efficient and consistent when this 1:1 ratio of water to solids is maintained throughout the test. Samples allowed to dry before adding water exhibit slow degradation with a rapid increase when water is available [65]. If composting vessels become too saturated, standard methods suggest moisture can be removed by injecting dry air or draining excess liquid. If vessels are too dry, water can be added [8, 9].

ASTM D5338 calls for water-saturated air and a figure shows individual humidifiers for each composting vessel, placed immediately after the flow control for each line [9]. ISO 14855-2 also specifies water-saturated air and shows the same sequence of CO_2-free air to flow controller, humidifier, then composting vessel [10]. Air flow for ISO 14855-1 may be dry or water-saturated as needed to maintain about 50% moisture content [8].

If an aqueous solution is used to scrub CO_2 from the incoming air, this can also be used to humidify the air [8]. For synthetic air or a dry column scrubber, the humidifier can be simply a bottle of water [10, 47, 57]. Air is introduced into the liquid and exhausted from the headspace above.

Anunciado, et al. referenced ASTM D5338 and described bubbling CO_2-free air through deionized water. In this case, the flow was controlled *after* the humidifiers [12]. Kijchavengkul, et al. divided the CO_2-free air into two lines, one dry and one bubbled through water. Precision flowmeters were used to adjust the mix and provide the desired humidity to multiple compost vessels [58]. Verstichel, et al. also used dry and wet aeration to control compost moisture content. In this study, moisture was checked weekly and water was also added directly to compost vessels if necessary [18].

Copinet, et al. placed the entire system in a UV exposure cabinet to enable study of photodegradation as well as biodegradation. They passed air through distilled water at the bottom of the cabinet. Relative humidity was maintained above 70% [48]. Wang, et al. also used 70% relative humidity inlet air to model laboratory-scale composting [39].

Oazana, et al. also placed the humidifier in the same temperature-controlled environment as the compost vessel. The system can be run with no humidifier or with nearly-saturated air. This allows control of drying as well as cooling of the compost. Dry air causes evaporative cooling. Some evaporative cooling also takes place with saturated air if the active compost is warmer than the ambient environment. Nearly-saturated air can increase compost water content because the mineralization process produces water while evaporation is minimized. This system uses a separate humidifier for each compost vessel [38].

A study by Castro-Aguirre, et al. explored the impact of moisture and reported that water-saturated air is not usually sufficient to maintain the desired moisture content in composting vessels. Additional water must be added periodically. An integrated sensor was used to monitor soil moisture [65]. In other studies, vessels were weighed and water added to return them to their initial mass [19, 40, 49, 67]. ISO 14855-1 indicates moisture level may be evaluated by visual observation or instrumentally [8]. A system of circulating condensed moisture back to the composting vessels is also possible. Pickens replaced approximated 20 mL distilled water per 200 g inoculum every four days [19].

Dehumidification

For some CO_2 measurement methods, the air from biodegradation vessels must be dehumidified. ASTM D5338 shows a large cooling unit and individual condensate collection vessels for the GC measurement option [9]. Verstichel, et al. used the same figure [18]. ISO 14855-1 also recommends removing water from the air only for direct measurements such as those taken by an IR analyzer or GC and mentions cooling as a possible technique [8]. There is no detailed description or explanation of dehumidification in any of these documents.

The automatic direct measurement system developed by Kijchavengkul utilizes an oil-bubbler over a water bath at 15 °C to remove condensed water from air before it reaches the IR gas analyzer [58].

Pickens used a Graham condenser at the top of each composting vessel to cause most of the water vapor to condense back into the vessel. This helps maintain moisture level in the vessel and minimize water in the air lines [19]. Oazona, et al. also used air-cooled Graham condensers for each composting vessel [38].

The gravimetric MODA system uses a series of silica gel columns to remove moisture from the air. The first column contains silica gel with an indicator; the second is a mixture of 80% calcium chloride and 20% silica gel [34, 57]. ISO 14855-2 describes slightly different arrangements in the Procedure section and in two Annex examples. All include a silica gel dehumidifying trap followed by anhydrous calcium chloride. The MODA system and the generic gravimetric system described in ISO 14855-2 also include an ammonia trap consisting of dilute sulfuric acid between the composting vessel and the dehumidifiers [10].

Flow Rate

Composting Tests

ASTM D5338 calls for controlling air flow based on the exhaust composition. At least 6% oxygen in exhaust air indicates aerobic conditions in the composting vessel. A minimum of 2% CO_2 v/v in the exhaust enables accurate measurement [9]. ISO 14855-1 and ISO 14855-2 also mention maintaining "truly aerobic conditions" and checking regularly for leaks [8]. The smaller-scale ISO 14855-2 specifies an equal flow rate of 10–30 mL/min through each 500-mL composting vessel. The system schematic shows a flow meter with flow rate controller between the scrubbing column and humidifier [10].

The MODA system employed in several studies was usually operated with a flow rate of 10 mL/min [34, 40, 62]. Three studies used a flow rate of 40 mL/min through 1.9-L vessels [12, 58, 65]. In one of these, researchers used three flow rates and four known quantities of CO_2 to determine the optimal flow. They found no difference in CO_2 evolution with flow rates of 20, 40, and 60 mL/min but settled on 40 mL/min for the remainder of the study [65]. Kale's system began with air at 2 psi. After passing through the NaOH solution scrub and deionized water humidifier, air was adjusted to 60 mL/min through individual flowmeters at each composting vessel [57].

An interlaboratory study during the development of ISO 14855 indicated that air flow was "carefully dosed or recorded" and the rate was dependent on the system used. One lab's rate was reported as 50–100 mL/min. No other rates were reported and vessel size is only described as a minimum of about 2–3 L [66]. Pickens used a 200 mL/min flow of pure O_2 through 0.5-L vessels. Initial testing and calibration trials were performed at 500 mL/min [19].

Wang modeled a wide variety of composting scenarios, all with higher air flow rates (and larger vessels) than those used in other laboratory-scale tests [39]. Oazana also used relatively large vessels and higher flow rates. In this case, flow varied from 0 to 5,000 mL/min in response to changes in temperature, CO_2 concentration, or O_2 concentration.

Aqueous Tests

The standard tests for aqueous degradation use a higher flow rate through each vessel and cite a range rather than a specific rate. Flow rate is reduced as air passes through liquid, especially if it is bubbled or sparged. Vessel volume is also generally larger to accommodate aqueous tests. ASTM D5864 and ISO 14852 call for air flow of 50–100 mL/min [28, 30]. OECD 301 indicates 30–100 mL/min [26]. The ISO tests further specifies that flow is to be held constant within 10%. Flow should not vary from 50 to 100 during a single test. If the flow rate is set to 75 mL/min, an acceptable range is 67.5–82.5 mL/min.

An automated system for aqueous degradation testing used 750 mL/min air input, but this appears to be before passing through scrubbing and humidifying solutions, and before being split to feed nine 3-L vessels [59]. Air flow rate entering the biodegradation vessel is likely in the range specified by standard methods. Other aqueous studies also used similar flow rates [25, 32]. Weytjens, et al. gives further explanation that 50–100 mL/min is equivalent to 1–2 bubbles per second for the described system [32]. Pantazi, et al. reported air flow was around 33 mL/min [60].

Direction

Despite wide variation in the lab-scale composting systems described in literature, most studies agree that aeration should be from the bottom of the composting vessel [12, 18, 19, 34, 37, 40, 58, 64, 68]. This facilitates delivery of moisture directly to the compost rather than the surface where it would quickly evaporate. A few papers emphasized the contribution of upward aeration to uniform air distribution and homogeneous biodegradation [38–40].

None of the standard test methods specifically call for aeration from below, but a figure in ISO 14855-1 shows the air inlet as low in the compost vessel. The air outlet comes from headspace above the compost [8].

The biometer flask system is an exception to the aeration from below. Air is injected from the top of the Erlenmeyer flask and exhausts to the side arm [20, 21, 69].

Aeration from below presents a challenge in keeping air lines clear of compost. Anunciado, et al. mentioned using a screen in the vessel to prevent mulch from sticking to the vessel walls as well [12]. Several researchers used mesh screens to support the compost mixture above the air inflow to prevent clogging [12, 38, 39, 58].

Kjchavengul, et al. described a particularly elaborate screen arrangement. Two layers of 18-mesh aluminum screens were rotated 45° from each other and supported by a metal grid. Compost and specimens were placed above the mesh and the air inlet was below it. A port in the vessel lid was used to exhaust air as well as injecting water or other substances.

Oazana, et al. also used a second screen, but this was placed above the compost mixture. The level of the top screen was adjustable to control volume and bulk density.

Measurement

There are multiple approaches to knowing and controlling air flow rate. A rotameter was a common choice among

surveyed literature [12, 32, 60, 64]. Various types of flow meters were also used. One study specified a mass flow meter [19], one specified a thermal flow meter [18], and others mentioned simply a flow meter [47, 57].

Considerations

It is interesting that cited air flow rates for aqueous biodegradation tests are quite consistent, yet Weytjens, et al. notes that the origin of this range is unknown and that no studies were found on the effects of higher flow rates. The authors suggest that removing all CO_2 as it is produced in the biodegradation vessel would require an impractically high air flow rate [32]. Castro-Aguirre, et al. reported that higher flow decreases CO_2 concentration in the exhaust air so air flow rate must be adjusted to suit the sensitivity of the detector system used. The researchers found no impact when varying flow rate between 20 and 60 mL/min but advise that too low a rate limits oxygen availability and slows biodegradation. Higher air flow rates lead to quicker drying and cooling, which can also retard biodegradation [65].

The need for adequate O_2 to enable optimal microbial activity and measurable CO_2 concentration is also reflected in ASTM D5338 [9]. Smaller biodegradation vessels may use lower flow rates, since O_2 needs and CO_2 production are both expected to be lower than for a larger vessel.

Air flow is mentioned as a source of "aeration" in ASTM D5338, but it is clear that air flow alone is not adequate to mix or separate compost. A separate instruction is included to shake composting vessels weekly to redistribute the contents [9]. Even aqueous methods include a mechanism for stirring or agitation in addition to aeration [28].

Discussion

Air flow is obviously a key component of aerobic biodegradation. Air must deliver oxygen to enable microbial breakdown of test samples. Air also carries moisture to the degradation vessel and delivers evolved CO_2 to traps or sensors. Consistent flow through each vessel is critical for valid test and control comparison. For methods measuring evolved CO_2 by GC (or IR), air flow to the instrument requires separate control [11]. Accurate measurement is more important than precise control of flow rate. Scrubbing CO_2 from incoming air is a best practice but one regularly ignored.

CO_2 Measurement

A paper by Castro-Aguirre, et al. provides a good summary of approaches to quantifying CO_2 production from biodegradation. In cumulative measurement respirometry (CMR), the evolved CO_2 is accumulated in a chemical solution trap and titrated. Gravimetric measurement respirometry (GMR) is also cumulative, but CO_2 is captured in dry absorption columns and quantified by the increase in mass. Direct measurement respirometry (DMR) is measured in real time using an IR sensor or GC [65]. Table 6 groups studies according to the CO_2 measurement method used.

Cumulative Measurement Respirometry

CMR is the default method for ASTM D5338, ASTM D5988, ASTM D5864, and OECD 301B; it is the alternate method for ISO 14855-1. Chemical absorption traps may be the same or similar to those used for scrubbing CO_2 from incoming air.

Barium Hydroxide Traps

ASTM D5338, ASTM D5988, ASTM D5864, and OECD 301B include a $Ba(OH)_2$ solution for CO_2 traps (see Table 6). The prepared solution is filtered, standardized, and sealed to prevent absorbing CO_2 from the air. ASTM D5864 further calls for storing under nitrogen [28]. OECD 301B notes that the concentration must be determined immediately before use [26].

Several of the standard test methods include instructions for preparing a suitable $Ba(OH)_2$ solution. ASTM D5338 directs dissolving 4.0 g $Ba(OH)_2$ per liter of distilled water [9]. ASTM D5988 uses the same 4.0 g per liter recipe, specifying *anhydrous* $Ba(OH)_2$ [22]. ASTM D5864 calls for 4.0 g $Ba(OH)_2 \cdot 8\ H_2O$ per liter [28]. Calculations show 4.0 g/L of the octahydrate produces the prescribed 0.0125 M concentration. ASTM D5338 and ASTM D5988 concentrations are specified by normality (0.024 *N* and 0.025 *N,* respectively). Because one mole of $Ba(OH)_2$ produces two hydroxide ions or two equivalents, molarity of this basic solution is typically considered to be half the normality. Coincidentally, 4.0 g/L of anhydrous $Ba(OH)_2$ has a molarity close to 0.024 M. If $Ba(OH)_2$ is assumed to have only one equivalent since each mole reacts with one mole of CO_2, the stated 0.024 *N* value is also correct. The reviewed studies all used higher concentrations, ranging from 0.024 to 0.12 M. No explanation is provided for this modification of the standard methods [12, 13, 17, 25, 40, 59].

Reviewed studies also used smaller volumes of $Ba(OH)_2$ solution than specified in ASTM D5338. This is aligned with smaller composting vessel volumes as previously discussed. It may also be that higher concentrations allow for lower volumes to achieve the same CO_2 absorption capacity.

Most of the standard and modified procedures use a series of $Ba(OH)_2$ traps. ASTM D5988 uses only one, but this system is contained in a desiccator with no airflow [22]. Systems utilizing KOH or NaOH typically use a single trap although OECD 301B does suggest a second NaOH trap

Table 6 CO_2 measurement in lab-scale biodegradation testing

Standard Method Cited	CO_2 Measurement	CO_2 Trap	Reference
ASTM D5988-18	**CMR**	**0.025 *N* $Ba(OH)_2$ *or* 0.5 *N* KOH**	[22]
ASTM D5864-18	**CMR**	**0.0125 M $Ba(OH)_2$**	[28]
OECD 301B (1992)	**CMR**	**0.0125 M $Ba(OH)_2$**	[26]
ASTM D5338-15(2021)	**CMR**	**0.024 *N* $Ba(OH)_2$**	[9]
ISO 14855-1:2012	**CMR**	**20 g/L NaOH**	[8]
ASTM D5338	CMR	0.024 *N* $Ba(OH)_2$	[12]
modified Sturm (CEN draft 1995)	CMR	0.05 M $Ba(OH)_2$	[59]
ASTM D5988-96	CMR	0.05 *N* KOH	[70]
ASTM D5338	CMR	0.40 M KOH	[20]
ASTM D5338	CMR	KOH solution (5 mL, 50% wt.)	[13]
ISO 14855-1 (JIS K6953)	CMR	not specified (alkaline solution)	[40]
ASTM D5988-03	CMR	0.5 *N* KOH	[23]
not specified	CMR	80 mmol/L $Ba(OH)_2$	[71]
ASTM D5338	CMR	not specified	[14]
ASTM D5338	CMR	0.5 *N* KOH	[36]
not specified	CMR	NaOH solution	[66]
ISO 14852:2005	CMR	0.05 mol/L NaOH	[60]
ASTM D5209-92	CMR	not specified	[72]
not specified	CMR	0.4 *N* KOH	[21]
ASTM D5338	CMR	0.5 *N* KOH	[16]
ASTM 5988–12	CMR	0.5 *N* KOH	[24]
OECD 301	CMR	$Ba(OH)_2$ solution	[33]
not specified	CMR	0.05 $Ba(OH)_2$	[25]
ASTM D5338	CMR	0.1 M $Ba(OH)_2$	[17]
OECD 301B	CMR	0.05 M NaOH	[32]
ISO 14851:2019	CMR	30% KOH	[73]
not specified	CMR (NH_3, not CO_2)	H_3BO_3	[68]
ISO 14855-2	CMR (frozen $BaCO_3$)	$Ba(OH)_2$ solution	[40]
ISO 14851 (1999)	CMR (OxiTop)	NaOH solution	[31]
ASTM D5338	CMR (scintillation)	$C_3H_8O_2$ and C_2H_7NO	[15]
ISO 14855-2:2018	**GMR**	**NaOH (soda lime and soda talc)**	[10]
ISO 14855-2	GMR	NaOH (soda lime and soda talc)	[62]
ISO/DIS 14855-2	GMR	NaOH (soda lime and soda talc)	[34]
ISO 14855-2	GRM	NaOH (soda lime)	[74]
ISO 14855-1:2012	**DMR**	**IR or GC**	[8]
ASTM D5338-15(2021)	**DMR**	**GC or other**	[9]
ISO 21701:2019	**DMR**	**GC**	[11]
modified Sturm (CEN draft 1995)	DMR	IR (0–1,000 ppm)	[59]
not specified	DMR	IR (0–20,000 ppm)	[38]
ASTM D5988	DMR	IR	[12]
ISO/DIS 14855:1997 *and* ASTM D5338-92	DMR	IR	[64]
not specified	DMR	IR	[66]
ASTM D5338 & ISO 14855	DMR	NDIR (0–3,000 ppm)	[58]
ASTM D5338 & ISO 14855	DMR	NDIR (0–20,000 ppm)	[75]
ISO 14855-1:2007	DMR	NDIR	[76]
ASTM D5338-98 (2003)	DMR	NDIR	[19]
ISO 14855 (1999)	DMR	GC	[18]

Standard test method specifications are shown in **bold**

CMR Cumulative measurement respirometry; *GMR* Gravimetric measurement respirometry; *DMR* Direct measurement respirometry

to verify that all CO_2 is absorbed by the first. A third trap is used for the OECD 301B $Ba(OH)_2$ series because the solution is more quickly saturated and the traps have to be rotated and refreshed over the course of the test [26]. The trap closest to the compost vessel is removed for titration, others are shifted closer, and a new trap added to the end of the series [28].

Traps must be changed out before the solution is fully saturated. ASTM D5988 emphasizes this and includes a process to estimate the time interval for replacing $Ba(OH)_2$ solution. The time is dependent on the headspace volume as well as the inoculum medium and test sample. The interval increases as the carbon content of the vessel contents is exhausted. For methods with forced airflow, flow rate must also be considered. For the static ASTM D5988 test, it is estimated that $Ba(OH)_2$ traps will need to be replaced every 3–4 days for the first 2–3 weeks and then only once every 1–3 weeks. ASTM D5864 does not specify the replacement frequency, but recommends preparing 5 L $Ba(OH)_2$ solution at a time. For 45 traps (3 each for 15 composting vessels), this would only allow replacement of five 100-mL traps over the course of a 28-day test.

Potassium Hydroxide Traps

ASTM D5988 includes an option to use KOH solution in place of $Ba(OH)_2$. The standard notes that this avoids the problem of precipitate formation on the liquid surface. This is particularly relevant to this method because the only interaction between evolved CO_2 and the trap solution is at the surface. Other methods provide forced airflow from below the surface. All the literature citing ASTM D5988 for the procedure, as well as several studies citing ASTM D5338, used a KOH solution. Cheillini & Corti discussed the trade-offs between $Ba(OH)_2$ and KOH in detail. While KOH does not form a film during CO_2 absorption, it also does not produce as clear a color change upon titration [70].

$Ba(OH)_2$ concentrations in practice were higher than those specified in standard test methods; the opposite was true for KOH concentrations. ASTM D5988 calls for a 0.5 *N* solution [22]; nearly all reviewed studies used 0.05 *N* [16, 21, 23, 24, 36, 70].

Sodium Hydroxide Traps

NaOH traps are less frequently used. Two reviewed studies mention 0.05 M NaOH solution as CO_2 traps [32, 60]. ISO 14855-1 includes a 20 g/L solution of NaOH as a possible alternative to direct measurement by IR or GC [8]. OECD 301B provides an option to use either 0.05 M NaOH or the $Ba(OH)_2$ solution previously mentioned. The NaOH solution does not produce a visible or filterable precipitate on interaction with CO_2, but will contain small quantities of carbonates, both Na_2CO_3 and $NaHCO_3$. OECD 301B indicates that this will be corrected by subtracting the value of the blank specimen [26].

Titration

All titrations described in the literature use HCl with reported concentrations ranging from 0.05 *N* to 1 *N*. Standard test methods call for 0.05 *N* HCl for titration of 0.025 *N* $Ba(OH)_2$. ASTM D5988 notes that 0.25 *N* HCl should be used for the 0.5 *N* KOH option. Several reviewed papers use a higher concentration of HCl in line with higher than specified concentrations of $Ba(OH)_2$. Research using KOH as the CO_2 trap all used HCl concentrations of 0.1–0.5 *N*, with one exception. Work by Muniyasamy, et al. was performed with 1 *N* HCl for titration of 0.05 *N* KOH [36].

Titration measures the solution *not* reacted with CO_2. Equation 12 shows that the hydroxide reacts with CO_2 to form a carbonate and water. It is the remaining hydroxide that is titrated with HCl to determine concentration (Eq. 13). From the original concentration, the amount consumed can be calculated.

$$Ba(OH)_2 + CO_2 \rightarrow BaCO_3 + H_2O \tag{12}$$

$$Ba(OH)_2 + 2\,HCl \rightarrow BaCl_2 + 2\,H_2O \tag{13}$$

Titration procedures specifying an indicator all include phenolphthalein. One study with KOH traps and one with NaOH used a two-part titration with phenolphthalein to indicate neutralization of the excess base and carbonate conversion to bicarbonate. Methyl orange was used to indicate subsequent neutralization of the bicarbonate [20, 60].

Titration is performed on samples drawn from the traps throughout the course of the biodegradation test. ASTM D5864 provides additional guidance for final measurement at the end of the test. Concentrated HCl is added to the composting vessel to release any trapped CO_2 and the test is continued for an additional 24 h to trap the released CO_2. In addition to removing and titrating the trap closest to the biodegradation vessel, contents of the remaining bottles are also titrated. As the designation implies, a cumulative total of the CO_2 absorbed over the course of the test is calculated [28].

Variations

A few studies used a CRM approach without manual titration. OxiTop bottles measure O_2 consumption based on pressure change as CO_2 is absorbed by NaOH or other alkaline solution [31]. Another study used the standard titration measurement but used H_3BO_3 solution to collect ammonia (NH_3) instead of CO_2 [68].

The most unusual technique was employed by Kunioka, et al. Rather than titrating the unreacted $Ba(OH)_2$ solution, the group collected the precipitated $BaCO_3$, freeze dried it, and analyzed the carbon content using accelerator mass spectrometry (AMS). Researchers determined the $^{14}C/^{12}C$ ratio for CO_2 captured as $BaCO_3$ powder. The reported advantages were a high level of accuracy and elimination of the need for a blank vessel; however, the authors concluded that the method was not widely used due to the high cost and sophisticated instrumentation [40]. ASTM D6866 Determining the Biobased Content of Solid, Liquid, and Gaseous Samples Using Radiocarbon Analysis and the equivalent ISO 16620-2 differentiates "modern" biogenic carbon from that of petroleum sources [77].

Pettigrew, et al. used a trap solution of ethylene glycol monomethyl ether ($C_3H_8O_2$) and ethanolamine (C_2H_7NO) with scintillation counting to determine the radioactivity of radiolabeled PCL samples [15].

Challenges

The most obvious drawback of CMR is the time-consuming process of changing and titrating traps. One author reported that a 45-day test required 100 h of titration work [59]. ASTM D5988 and ASTM D5864 include provisions for use of an automatic titrator [22, 28]. The process is still time-consuming, but is more accurate and less subjective than manual titration.

Absorption of CO_2 from the atmosphere is another frequently cited source of error. Hydroxide trap solutions readily react with the air during preparation and titration steps. The additional filtration required for $Ba(OH)_2$ traps compounds this challenge. ASTM D5864 includes several reminders to minimize exposure to air [28]. Other literature also mentions the importance of sealed vessels and immediate measurement [32, 40]. Blank specimens are intended to offset overestimation of evolved CO_2 due to air exposure but Srinvansan noted that this approach of calculating the difference between two "relatively large titration values" lacks precision as trapped CO_2 is not measured directly [33]. ASTM D5864 notes that uncertainty increases with more titrations [28]. Calmon also reports that errors accumulate from daily titrations [59].

Underestimation of evolved CO_2 is also an issue. This can be due to incomplete trapping [59, 66]. Weytjens, et al. provide several additional explanations. Even in a perfectly designed test, some of the CO_2 produced by microbial activity is utilized for growth. In addition, the authors express doubt about the efficiency of chemical traps and report "it is expected that flow rates which would remove CO_2 at its production rate would be impractically high." ASTM D5864 and some aqueous studies use HCl or other acid to release CO_2 that may be trapped in the biodegradation medium at the completion of the test [25, 28]. There is no parallel procedure for tests using solid compost or soil media and there is no data reported for the relative quantity of CO_2 released in this step in relation to the cumulative total.

Gravimetric Measurement Respirometry

ISO 14855-2 uses gravimetric analysis to determine the amount of CO_2 evolved by biodegradation vessels. The standard test method calls for a column filled with a mixture of soda lime and soda talc to absorb CO_2. These materials are discussed in detail in the Scrubbing Column section. A separate column of anhydrous calcium chloride ($CaCl_2$) absorbs water. The change in mass of both columns combined is measured to the nearest 0.01 g at regular intervals. The system also requires an ammonia trap and dehumidifying traps before the absorbent columns. The method notes that 80 g of equal parts soda lime and talc can absorb 15 g of CO_2 but columns should be refilled with fresh absorbent when they reach about 80% of their capacity [10].

Diagrams in work by Kunioka, et al. show the absorption columns as described in the ISO method. Researchers refreshed columns when they reached 40% of their theoretical capacity. Mass was measured daily [34, 40, 62]. For the commercial MODA apparatus, users are instructed to place soda lime (without talc) in the first absorption column and Ca_2Cl_2 in the second [78].

Kunioka explains that the increase of both columns is equal to the CO_2 produced based on Eq. 14.

$$CO_2 + 2NaOH \rightarrow Na_2CO_3 + H_2O \tag{14}$$

For this to be true, all moisture must be removed from the air exiting the compost vessel before it reacts with the absorbent. Ammonia is also removed "to obtain an accurate carbon balance" [40].

Despite being adopted as a standard ISO test method in 2007, there is limited literature on use of the gravimetric approach beyond the research group that developed the method and the MODA apparatus.

Challenges

As with CMR (and DMR), GMR measures evolved CO_2. One paper notes that even biodegradation of the positive control cellulose is only measured at 60–80% because the remaining 20–40% of carbon atoms are otherwise used, including for the "body of microorganisms" [62].

The absorption trap must still be removed, measured, and replaced regularly, but weighing a trap is simpler and more repeatable than titration.

Direct Measurement Respirometry

DMR is specified in ISO 21701, as the primary method in ISO 14855–1, and as a secondary option in ASTM D5338. These standard methods call for direct measurement by GC although ISO 14855-1 also mentions IR and ASTM D5338 allows for "other" techniques [8, 9, 11]. Of the studies reviewed, DMR was used in about half as many as CMR (see Table 6).

Infrared Spectroscopy

The ISO working group on textile biodegradability (ISO/TC38/WG30) is currently discussing revision of ISO 21701 to include an IR option, specifically using a non-dispersive infrared (NDIR) detector. The delegation proposing the revision reported that the dual-wavelength NDIR technique allows for measurement under pressure or with interference from moisture and dust while single-wavelength IR techniques do not. NDIR also enables continuous real-time measurements [79].

Several authors specified NDIR sensors in their work [19, 58, 75]. Others did not specify ND but used the same Li-COR 820 model referenced by those who did. One study utilized an automated respirometer with several sensor technologies, including NDIR for CO_2 [76]. Prasad Duminda, Senior Executive—Analytical for Bureau Veritas Consumer Products Services Lanka (Pvt) Ltd. said that lab uses an IR detector to conduct tests according to ASTM D5338.

Among the papers indicating measurement ranges for IR sensors, there was significant variation. Two studies used sensors with relatively low ranges of 0–1,000 ppm and 0–3,000 ppm [58, 59]. Oazana, et al. used a sensor with much wider range of 0–20,000 ppm [38]. Castro-Aguirre also specified an NDIR gas analyzer measuring 0–20,000 ppm for his automated DMR system [75].

Calmon, et al. explained that IR was selected because it is simpler than either titration or GC. An experimental comparison with $Ba(OH)_2$ trapping and titration showed the automated IR system was "less labor-intensive, more compact and cost-effective" [59].

Challenges

According to Calmon, et al., the CO_2 produced in biodegradation of small specimens is too low in concentration to be properly measured by conventional IR probes. The researchers developed a method to concentrate the CO_2 in the headspace before it was swept into the detector [59].

DMR provides CO_2 measurements at discrete points in time rather than a cumulative value over the course of the test. To extrapolate the total CO_2 evolved, a known flow rate is critical. Researchers employed various mass flow controllers and rotameters to ensure an accurate and consistent flow rate through the analyzer [18, 64, 75].

Additional steps must also be taken to protect the sensitive measuring equipment. Castro Aguirre noted that water condensing after exiting the biodegradation vessel can damage the IR analyzer, so a water trap was installed. A mass flow controller was also installed to ensure an accurate and consistent flow rate through the analyzer [75]. Degli-Innocenti, et al. described a similar system for trapping vapor and measuring airflow using a rotameter before measuring CO_2 concentration by IR detection [64].

Gas Chromatography

Only one of the papers reviewed mentioned gas chromatography for measuring evolved CO_2. A cooling unit and trap removed condensation. Air flow was measured using a thermal mass flow meter [18].

Sampling is particularly important for GC because it is unlikely that each biodegradation vessel will be measured continuously for the duration of the test. Verstichel, et al. used a multiport valve and analyzed evolved gas from individual biodegradation vessels every 6 h [18].

Although a complete survey was not performed, commercial labs OWS and Bureau Veritas Consumer Products Services Lanka use GC for measurement of evolved CO_2. OWS autosamples vessels on a rotating basis. Exhaust from one vessel at a time is analyzed by GC. The others are exhausted to the room.

Discussion

Researchers have developed means of automating various controls and measurements but there is no evidence of widespread adoption of any such technology. It is likely that cost still outweighs convenience for most small laboratories. For large labs with an expectation of high volume, profitable testing, the investment is more palatable. It is likely that instrumental measurement of CO_2 evolution will become increasingly common as prices drop and accuracy improves. Instrumental options are already included in standard test methods ASTM D5338 and ISO 14855–1. While GC and IR techniques save considerable time and materials over trapping and titration, they necessitate other accommodations including dehumidification and enhanced air flow measurement. DMR has the potential advantage of showing a more complete profile of biodegradation over the course of the test but requires more interpretation, and even interpolation, to calculate the percent biodegradation of a sample.

Compostability of Textiles

Optimizing a laboratory composting system requires some understanding of the expected degradation rate. Findings are presented here, not as a comprehensive review, but to provide benchmarks for planning future studies There are limited studies specifically addressing composting of textile materials, perhaps because infrastructure for doing so on an industrial scale does not yet exist. A review by Egan and Salmon concluded that "comparison of textile biodegradation results is hindered by variabilities in test methods, conditions, physical form of samples, and duration of testing" [6]. Patti, et al. lists several biodegradable products currently on the market with potential application in textiles (see Table 7). Both reviews focus on polymers with textile applications, but the surveyed literature is not limited to textile substrates.Reviewers emphasized that environment plays a significant role in biodegradation. Polymers do not degrade equally in compost, soil, and marine environments [6, 7]. A 2004 study of textile biodegradation refers only to soil burial and explains that biodegradability "is often used as a standard measurement for the environmental friendliness of textile products" [72]. Biodegradable fibers in uncontrolled environmental conditions can lead to waste accumulation and increased greenhouse gas emissions.

PLA

One area that does have current applications is biodegradation of agricultural mulch film. Mulches are used to prevent weeds, moisture loss, and soil erosion. Traditional polyethylene films must be removed and disposed of at the end of their service life; biodegradable nonwoven mulch is intended to be naturally incorporated into the soil at end of life. One study found that addition of poly(3-hydroxybutyrate-co-4-hydroxybutyrate) (PHA) to biobased PLA enhanced the biodegradability of meltblown mulches, but also provided lower tensile strength and molecular weight before burial [50]. PLA and PHA are both categorized as "renewable" polymers from natural sources [7]. Spunbond PLA mulches showed less degradation after 30 weeks of soil burial. The authors suggest these materials may be useful for long-term applications [50]. Another study showed that PLA nonwovens can be completely biodegraded in 16 weeks under composting conditions. This study found that crystallinity, varying from 8.9% to 31.2%, had no impact on the process [49].

Egan's review found that PLA was biodegradable in compost and semi-biodegradable in anaerobic digestion and seawater environments; it was nonbiodegradable in soil burial tests because the initial mechanism of degradation is by temperature-dependent hydrolysis of ester bonds [6]. Patti's review reported that PLA was 60%-70% biodegradable within 30 days. While PLA is the most widely used synthetic biopolymer in textile applications, the microorganisms required to degrade it are not naturally widespread in soil [7]. Another paper confirms that PLA degrades very slowly in soil, but can be hydrolyzed in a compost environment after 45–50 days at 50–60 °C [74]. An earlier study also emphasized the importance of medium but reached somewhat different conclusions. In that study, biodegradation of PLA was at least 90% in liquid medium, 80–83% in inert solid (vermiculite) medium, and about 64% in compost. Trials were conducted at a steady 58 °C and with temperatures ranging from 35 to 58 °C and back down over the 45-day test. The temperature profile had no significant impact in any medium. All biodegradability tests were performed after ultraviolet exposure, which was determined to be "very important to stimulate the biodegradation" [48].

Research on the potential ecotoxicity of compost containing PLA fiber showed no influence on plant growth or seed production over the 2-month study. PLA fiber fragments were ingested and transported by earthworms, but the impact on mortality was unclear. The authors suggested further studies with longer duration and more replicates [53].

Linen

Linen fabric was tested under composting conditions using EN 14806:2005. The researchers concluded that more degradation occurred during the final mesophilic phase of the test than during the initial thermophilic phase. The linen sample was 55% disintegrated after 90 days, weight loss of the compost/sample mixture was 61%, and the germination index

Table 7 Biodegradability of Polyester Polymers in Compost Environment

Polymer	Egan, et al. [6]		Patti, et al. [7]	
	(days)	(%)	(days)	(%)
Poly(lactic acid) (PLA)	45	>70	30	60–70
Poly(hydroxyalkanoate) (PHA)	_	_	180	94
Poly(3-hydroxybutyrate-co-3-hydroxyvalerate) (PHBV)	_	_	28	80
Poly(caprolactone) (PCL)	45	>70	91	100
Poly(butylene adipate terephthalate (PBAT)	45	moderate	45	33–67
Poly(butylene succinate) (PBS)	_	_	160	90

was 73.89%. The study authors reported that these results qualify the linen fabric as biodegradable under composting conditions and the resulting compost as mature [47]. These values do not meet the 90% carbon conversion and 90% relative germination required by ASTM D6400 for labeling as compostable [41].

In a separate study, linen ranked third of four fabrics evaluated (rayon, cotton, linen, and acetate) in terms of CO_2 evolution from a sludge test and total organic carbon from enzymatic hydrolysis. The authors attributed the relatively slow biodegradability to linen's highly crystalline structure [72]. In contrast, they found linen had the highest degree of degradation in a soil burial test evaluated by loss of tensile strength. The linen sample also developed the most fungi and exhibited the greatest shape deformation. The extensive physical degradation is explained by the proportion of non-cellulose components, including lignin. These areas provide access for larger organisms such as earthworms [72].

Cotton

Based on the research surveyed by Egan, cotton is > 70% biodegradable in soil and anaerobic digestion, but only semi-biodegradable in compost due to the short study times employed [6]. In a study of cellulose fabric biodegradation, three separate tests were performed with similar results. Biodegradability was ranked rayon > cotton > > acetate in soil, activated sludge, and enzyme hydrolysis; linen had the highest biodegradability in soil but was less biodegradable than cotton in sludge. Biodegradability is influenced by crystallinity, orientation, polymerization, and hydrophilicity. Crystallinity, orientation, and molecular weight are negatively correlated with biodegradability, favoring the more amorphous structures of rayon and acetate. Hydrophilicity is positively correlated, giving rayon an added advantage. Acetate is less hydrophilic than other cellulose fibers because of acetyl substitution for some hydroxyl groups [72].

Several studies evaluated biodegradation of cotton fabrics with various finishes. In a 2010 paper, cotton fabric with softener degraded fastest and cotton with resin was slowest in both lab and field tests. All cotton fabrics in this study were considered compostable based on weight loss (50–77%) after 90 days buried in windrows. Lab tests in soil (not compost) measured the percent conversion of carbon to CO_2 after 90 days. The cotton fabric with softener had the highest rate of conversion at about 28%. Cotton with resin was less than 17% converted [23]. Another version of this study was performed with additional finishes and extended lab test duration. Results confirmed that crosslinked fabrics produced less CO_2 after 154 days in soil. The authors concluded that CO_2 production alone is insufficient for evaluating cotton biodegradation. The polyfunctional blocked isocyanate crosslinker (PBI) was around the middle of 9 finish combinations in terms of CO_2 production but was the only sample to have significantly higher mass loss than the control fabric. The PBI fabric was visibly missing large portions after 154 days. This was not true for PBI in combination with other finishes. It is believed that the brittleness of PBI fabric led to physical disintegration. Breakdown of the coating also makes the fabric more hydrophilic, leading to fiber swelling and cracks that provide greater surface area for microbial activity. The cotton fabric with flame retardant finish had the highest initial mass loss and among the lowest at the end of the experiment. Several fabrics showed slow loss in the first 75 days followed by more rapid loss from day 75 to day 154 [24].

Tests of aquatic biodegradation of cotton fabrics had similar results. Crosslinking in durable press and water repellent finishes had the greatest impact on the process. Surface hydrophobicity from the water repellent finish also appeared to slow the initial adsorption of enzymes. The authors report that even with finishes that decrease the rate of biodegradation, there was no significant difference in the final extent of degradation. They also note that the active compounds in dyes and finishes are generally not biodegradable and are released into the environment as the fiber substrate is degraded [73].

Other Fibers

In addition to the polymers listed in Table 7, Patti, et al. include poly(glycolic acid) (PGA), polysaccharides, and proteins as available biodegradable products. Polysaccharides and proteins are derived from biomass. Polysaccharides include cellulosic, lignocellulosic and chitosan fibers; proteins include silk, collagen, and soy [7].

The review by Egan [6] showed poly(3-hydroxybutyrate-co-3-hydroxyhexanoate (PHBH) to be generally capable of "fast and complete" biodegradation and wool biodegradation is "slow and eventually complete," but neither was specifically studied in a compost environment.

Polyethylene terephthalate (PET) and nylon are nonbiodegradable [6]. In a test including cotton and PET fabrics with various finishes, PET remained mostly intact [23].

Cellulose

Microcrystalline cellulose for thin-layer chromatography is often used as a positive control in compostability tests, meaning it is expected to fully mineralize in the course of the test. This may give some indication of cellulosic textile material behavior in composting although textiles are not as purified, nor as finely milled. Plant fibers used in textiles are typically 75%-90% cellulose [80].

Degli-Innocenti, et al. performed several trials with cellulose alone to determine the accuracy of standard methods

(ASTM D5338-92, ISO/DIS 14855:1997, and the CEN counterpart). The authors were particularly interested to identify any "priming effect." The authors cite previous studies indicating that glucose polymers, including cellulose, can have this effect. Their own study showed that while calculated cellulose biodegradation was higher than 100% in some cases, this may be measurement error rather than evidence of priming. The average of 11 replicates over 47 days was 96.8% ± 6.7%. The authors conclude that the variability is acceptable for a biological system and report, "the coco [controlled composting] test is a reliable system also for starch and cellulose and, consequently, for starch-based and cellulose-based materials" [64].

Cellulose and hemicellulose account for most of the CO_2 and heat produced during composting. Lignin retards biodegradation of lignocellulosic materials. A paper focused on paper products rather than textiles offered a concluding remark relevant to either. "Whether or not composting is the best or most appropriate usage of a given lignocellulosic resource will need to be considered on a case-by-case basis" [2]. It is worth noting that even materials from natural plant fibers are not necessarily compostable in a convenient time period, even without the added complication of dyes and finishes.

Discussion

Textiles and textile fibers are obvious candidates for compostability and other biodegradation testing. Most studies show that these materials take longer than the standard 45 days to degrade, but there is no systematic approach to evaluating textile compostability. Polymer length, yarn twist, weave pattern, chemical finish and more can all influence biodegradation rate. Clearer guidance is needed, particularly to address the numerous variables and additives in textile construction. If textiles do not degrade under commercial composting conditions, including the standard timeframe, new waste streams, infrastructure, and labelling would need to be developed, with testing adjusted to represent real-world scenarios.

Conclusion

Laboratory tests are simplified and standardized models of real-world phenomena. There must be some reasonable and understood correlation between the two to derive value from lab-scale results. Despite necessary simplification for laboratory testing, a 1996 study of lab-, pilot-, and full-scale biodegradation tests concluded that "without exception, the degradation results obtained in a higher-level test equaled or exceeded those obtained in a lower-level test" [81]. Positive results in the lab suggest even better biodegradation performance in a commercial composting environment.

Compostability testing standards have been in place for more than 30 years. Many components are derived from aqueous biodegradation testing developed at least 50 years ago. In that time, there have been numerous refinements and variations, but there is always room for further optimization.

Biodegradation vessels as small as 0.5 L are in regular use. Even smaller vessels could further reduce testing costs and space requirements if challenges related to specimen concentration can be overcome. Accelerated testing with enzymes or other biostimulants shows promise, though probably for studying specific interactions rather than as a standard test.

Variations in temperature, compost inoculum composition, and air flow have all been studied. A steady temperature of 58 °C is well established and most researchers use compost inoculum from a local commercial facility. Air flow rate varies among studies but does not appear to be a determining factor in degradation if sufficient oxygen is supplied.

Automation and instrumental measurement of CO_2 is becoming increasingly widespread and represents a key area for optimization. For reliable, repeatable testing, more detailed description or specification of appropriate instrumentation should be defined and incorporated in standard methods. IR sensors vary widely in range and accuracy. Clear instructions for conversion of DMR data (typically in CO_2 ppm) to percent biodegradation can also be added to ensure consistent reporting.

To enable commercial composting of textiles, work is needed to optimize both full-scale operations and corresponding laboratory-scale testing. Several studies have looked at compostability of textile fibers and a few have included the impact of functional finishes on degradation rate. There is little published research on the degradation percentage or products of dyes and finishes applied to textiles. A thorough analysis of existing research on laboratory-scale compostability testing provided insight into best practices and areas for further improvement. Taken collectively, the literature also provides clarification and details of apparatus and procedures not fully explained in any single document. This review compiles and organizes the insights and procedural details to equip current researchers to efficiently explore compostability of more materials, including textiles.

Author contributions D.W. wrote the main manuscript text and prepared figures. All authors reviewed the manuscript.

Funding This work was supported in part by a research seed fund of the Wilsone College of Textiles at NC State University.

Data availability No datasets were generated or analysed during the current study.

Declarations

Conflict of interests The authors have no relevant financial or non-financial interests to disclose.

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