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ORIGINAL PAPER

Evaluation of the Disintegration of Bioplastics and Rabbit Leather in Industrial Composting Plants and Laboratory Scale

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Abstract

Disintegration is one of the key parameters to be assessed when evaluating the compostability of materials, as it ensures their potential for organic recovery through composting. While standardised methodologies exist for laboratory and pilot-scale disintegration tests, no equivalent standardised methodology is available for industrial scale testing under real composting plant conditions. Consequently, existing studies at this scale often employ diverse methodologies, leading to inconsistent and sometimes irreproducible results. This study proposes a reproducible and easy-to-implement methodology for full-scale disintegration testing in composting plants, preserving key composting parameters such as temperature, moisture, aeration, turning, screening, and process duration. The methodology employs custom mesh bags as reactors to confine test materials and prevent contamination of the composting mass, enabling accurate assessment of disintegration under industrial conditions. The methodology was applied to bioplastics and leather materials developed to be sustainable by being biodegradable or compostable, allowing organic recovery at their end-of-life. Parallel laboratory tests were conducted following ISO 20200:2023 to compare results and validate the industrial scale approach. The findings demonstrated that laboratory and industrial scale results were consistent, supporting the methodology's applicability across various composting facilities and technologies. This approach provides a robust framework for evaluating the disintegration of sustainable materials, ensuring their compatibility with composting processes while maintaining compost quality.

Keywords Circular economy · Sustainable materials · Disintegration tests · EN 13432 · ISO 20200

Introduction

Individual or collective activities, such as producing and consuming goods, inevitably generate waste of different categories. Consumers' waste disposal behaviour is an essential element in the success of the efficiency of waste management systems [1].

Labels are allies of the consumer which allows them to understand the nature of the product and how to dispose of it properly. The label must be clear, and when it presents the material as biodegradable, it must specify in which environment [2] since certain materials may be biodegradable under composting conditions but not in soil or water. The absence of or poorly catalogued labels generate confusion on the part of consumers, mainly due to the similarity between compostable and non-compostable products, such as packaging and bioplastic materials that are easily confused with conventional plastic materials [3]. Consequently, there is a risk of contamination, as bioplastics are easily found in other waste fractions such as multi-material (e.g. paper and cardboard, plastic, glass, metal) or biowaste [4].

To fit into the circular economy and to increase the rates of organic recovery and close the material cycle, producers must think about the end-of-life disposal of materials from the beginning of the product design in the scope of waste management. Awareness of developing and producing more sustainable products has been growing, mainly due to incentives such as policy tools like the *European Green Deal and Circular Economy Action Plans* [5]. As a result, some fossil-based plastic has been replaced by other

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biodegradable or compostable materials such as bioplastics, just as the original or traditional constitution of other materials has been exchanged for biodegradable bases or constituents to reduce the negative impact on the environment and resources. However, this replacement must be conscious and acute, because despite advantages such as sustainably reducing the volume of bioplastic waste through industrial composting [6], the reduction of dependence on fossil fuels, the return of carbon to the soil in the form of compost, or the possible mitigation of plastic pollution; there are also disadvantages, such as low material performance, high production costs, uncertainties regarding real biodegradability, and contributing to landfill greenhouse gas emissions [7].

Bioplastics are the most emergent sustainable alternative materials, although their production remains limited, accounting for less than 1% of the annual global plastic production [8]. They are designed to replace some traditional plastics, particularly used in agriculture [3], light bags and single-use products such as food packaging, foodware, or straws, especially in Europe, where the use of traditional plastic for single-use products has been banned, pursuant to Directive (EU) 2019/904 of European Parliament [9]. Bioplastics can be either biodegradable or non-biodegradable and may be derived from biological or fossil-based sources. Biodegradability implies that these materials can decompose through the action of microorganisms under specific conditions. However, further research is needed to verify their complete degradation across diverse environments to prevent bioplastics from becoming an environmental problem comparable to traditional plastics, which are recognised as an environmental pollutant [10].

Another industry that has reinvented itself with sustainable alternatives is the leather industry, which has been developing alternative treatments for replacing the traditional chromium leather treatment because chromium is one of the main environmental concerns related to hide and skin tanning and is subsequently found in wastewater and solid waste [11]. Thus, the most common options for end-of-life leather are incineration or landfill, which result in significant economic and environmental losses [11, 12]. However, with significant growth, sustainable alternatives have been emerging on the market with the principles of a circular economy, such as biodegradable leather lines where animal welfare is considered and animal hides and skins are sourced from the food industry as waste from the milk and meat industry [13, 14]. In this way, composting chromium-free leather with food waste can be an environmentally friendly alternative to landfill and incineration [11].

Proper end-of-life treatment is needed to close the loop on new sustainable materials designed to be biodegradable or compostable. According to a study by [6], the organic recovery of biodegradable materials by composting can be considered more sustainable and economically viable than recycling or incineration. However, many current composting plants are not prepared to receive and treat bioplastic [15] or other biodegradable material, which may lead to non-agreement to accept and value these materials together with biowaste. This is due to the particularities and operating conditions of each plant [16, 17] or due to the uncertainty of the actual biodegradability of these materials in the composting plants [3] even if they are certified as compostable or biodegradable. As a result, many of these materials end up in landfill or incineration.

Compostability certificates are awarded to materials when they fulfil specific requirements present in standards such as the European standard EN 13432:2000 [18] or its equivalent, such as the international standard ISO 17088:2021 [19] or the American ASTM D6400:2023 [20]. In addition to the characterisation of the materials to be certified, biodegradability, disintegration, compost quality and phytotoxicity tests are required. Regarding biodegradability tests, the EN 13432:2000 [18] specifies the use of the methodology of international standards as ISO 14851:1999 [21], ISO 14852:1999 [22] or ISO 14855:1999 [23]. However, EN 13432:2000 [18] only introduces the concept and some characteristics of the test methodology for classifying the degree of disintegration of the material but does not recommend a specific test methodology.

The assessment of biodegradability and the degree of disintegration has different objectives, characteristics and test methods, although some authors such as [24] assess the biodegradability of leather materials by following a disintegration test methodology in the laboratory. Disintegration is a physical process that results in the fragmentation of a material into smaller fragments, while biodegradation is the decomposition of organic compounds by microorganisms into carbon dioxide, water, mineral salts, and new biomass in the presence of oxygen. In the absence of oxygen, methane is also produced [18, 25].

Disintegration tests complement, but do not replace, biodegradability tests. However on a larger scale, the challenges of conducting tests in composting plants often result in disintegration and biodegradability studies being carried out primarily in laboratory settings [10]. For disintegration tests under composting conditions, the ISO 20200:2023 [25] and ISO 16929:2021 [26] standards are among the most commonly used that follow the general characteristics outlined in the EN 13432:2000 standard [10, 15].The ISO 20200:2023 [25] presents a simple and easy-to-reproduce laboratory methodology. The reactors, which can vary between 5 L and 20 L, are placed with mixtures of test material samples and synthetic waste as the solid matrix. For disintegration to occur, the reactors with the mixture are subjected to temperatures and moisture similar to composting.

The ISO 16929:2021 [26] is more complex and is carried out on a pilot scale with at least 35 L reactors. The moisture can be controlled and the temperature is monitored, but it is not forced, as it depends on the solid matrix, which can be only green waste or a mixture with biowaste. This standard becomes more challenging to comply with because of the test validation criteria, which depend on specific temperature ranges to be reached by the solid matrix. However, it has the advantage of being carried out on a larger scale than the laboratory standard test.

In both standards, the test material samples and the solid matrix must have specific characteristics, and the tests have an approximate duration of 12 weeks with regular and controlled maintenance. The degree of disintegration in both is calculated according to the weight loss between the amount of material sample placed initially and the amount recovered at the end, after screening at 2 mm.

The existing literature on tests that assess biodegradability and the degree of disintegration under composting conditions is predominantly focused on bioplastics, as the existing standards are designed for plastics and plastic packaging. Although these standards have also been applied to materials other than plastics, as [24] uses the methodology and composting conditions of the ISO 20200:2004 [27] standard to assess the biodegradability of different bovine leathers, and [28] evaluates the disintegration of linen fabrics based on the EN 14806:2005 [29] standard, which assesses the disintegration of packaging materials under composting conditions at a laboratory scale.

Certification by EN 13432:2000 [18], with proposals for laboratory scale tests, may not guarantee the compostability of materials under the specific conditions of composting plants whose process characteristics may differ between different plants. As a result, a particular material can be compostable in one composting plant and not in another. The gap in this standard is that the required biodegradability and disintegration tests are at laboratory or pilot scales. Although laboratory simulations of composting can provide better control and its results can be estimated, they cannot fully replicate the extreme conditions found in the natural environment of composting plants [15].

Despite the incentives to develop and produce biodegradable materials for composting, on the other hand, industrial composting plants may have the option of refusing to receive these materials and recover them along with their organic waste. The European Union has promoted compostable packaging and biowaste collection. Italy with the backing of national legislation, has been a strong advocate for this effort. On the other hand, countries like Belgium, Netherlands, France, Spain, Sweden, and Germany have adopted a different approach, by not allowing compostable materials in the biowaste stream and only allowing certified compostable bags for disposing of biowaste. Nonetheless, these materials are sorted during the pre-treatment phase at composting plants [1].

To close the gap in the composting standard, testing at a similar scale to that of the industrial plants is needed, in addition to laboratory testing or pilot scaling [10]. In the laboratory, disintegration tests are more straightforward to reproduce than biodegradability tests, and the same is true at larger scales such as composting plants. The difficulties of testing in composting plants lead to studies of disintegration and biodegradability essentially in the laboratory [10]. Still, authors such as [30] and [31] reinforce the need also to have studies on scales higher than in the laboratory.

In recent years, some authors have conducted experiments on the disintegration process at composting plants or similar facilities. While [32] tested mobile phone covers made of biocomposites inside mesh bags in compost piles [16], tested single-use bioplastics placed whole between two layers of nets in compost piles, and [17] examined various foodware and packaging materials inside 25 L net bags in four different types of composting conditions. These authors usually follow their own methodology which can be challenging to replicate, although most are based on established laboratory standards and the assessment of the degree of disintegration is not always quantified in weight and percentage. Such is the case with studies using visual analysis. For example [33], who analysed various bags labelled as degradable or compostable attached to mesh frames in compost piles; [34] who tested whole bottles of PLA in compost piles inside boxes with net bottoms; or [35] who tested PLA in rigid film and thermoformed trays inside cages in compost piles and through an intensive composting system that used closed containers.

This study focuses on industrial disintegration tests that preserve key composting parameters, such as moisture, temperature, turning, screening, and process duration as they occur in composting plants. The objective is to propose and analyse a methodology for conducting full-scale disintegration tests tailored for composting plant operations. The methodology is designed to be transversal, reproducible, and easy to implement, regardless of the specific process conditions of the composting plant or the material being tested, while ensuring that compost quality is not compromised. To validate the methodology and results, the study evaluates disintegration based on key composting and disintegration standards, comparing laboratory and industrial-scale disintegration outcomes for seven bioplastic and leather materials.

Table 1 Detailed description of the test materials

Test material	Description
Mater-Bi Bag	Mater-Bi Bag with length and width of 450 mm and 340 mm respectively, and 10.5 µm of thickness. Composition: starch, cellulose and vegetable oils. TUV Austria Certification: Ok Compost, Home Compost.
Kraft Cup	Kraft Cup with 240 ml. Composition: 90% of kraft and 10% of PLA (polylactic acid). DIN CERTCO Certification: Compostable.
PLA Cup	Transparent PLA cup with 300 ml. TUV Austria Certification: Ok Compost, Industrial.
Burger Box	Box with length, width and height of 152 mm, 148 mm and 79 mm, respectively. Composition: crystallised PLA, bagasse, recycled paperboard, virgin cardboard, and film NatureFlex. VINÇOTTE Certification: Ok Compost.
Fish Box	Compostable plastic box in foam, with a length, width and height of 400 mm, 300 mm, and 105 mm, respectively, and a thickness of 20 mm. Composition: polymer based on renewable raw materials - ecovio®, made by PBAT (polybutylene adipate terephthalate) and PLA. VINÇOTTE Certification: Ok Compost.
Rabbit Leather 1	Wet-white Rabbit Leather.
Rabbit Leather 2	Wet-white Rabbit Leather, with washable properties.

Materials and Methods

Test Materials

A total of seven test materials were analysed, consisting of five bioplastics that have been certified as compostable according to the standard EN 13432:2000 [18], as well as two leathers made from rabbit skin, developed to be biodegradable and chromium-free in the tanning process. Table 1 provides a detailed description of each test material, and Fig. 1 provides the photographs.

Disintegration Test in the Laboratory

The degree of disintegration of materials under laboratory scale composting conditions was assessed using the ISO 20200:2023 [25] procedure, and the interpretation of the results was in accordance with the EN 13432:2000 [18] standard, where the material recovered from the sieving procedure is considered to be non-disintegrated material, and not more than 10% of th original dry weight of test material shall fail to pass through a fraction greater than 2 mm.

The solid matrix in this test was a synthetic waste prepared in the laboratory and was made up of specific combination of components and their corresponding quantities, outlined in the ISO 20200:2023 [25] standard. The test materials were carefully measured and cut into 25 × 25 mm squares, except for the Fish Box, which was cut into 15 × 15 mm squares because it was more than 5 mm in thickness. The solid matrix and the test material samples were placed in simple reactors, such as polypropylene boxes, and incubated, to compost in an oven for a thermophilic period of 90 days, with air circulation and controlled temperature of 58 ± 2 °C (Fig. 2). The ratio of the solid matrix and the test material samples was the 50:1 (wet/wet), where only the Mater-Bi Bag and Fish Box samples were 200:1 (wet/wet) due to the volume of the samples. The test was carried out in triplicate, and in addition to the standard procedure, reactors with only synthetic waste were added as a control.

To maintain the suitable composting conditions in the reactors, the mixture was periodically turned according to the composting procedure in the standard [25]. Likewise, the moisture is adjusted by adding water in quantities and at intervals defined by following the same composting procedure. At the end of the test, the degree of disintegration was determined following Eq. 1.

$$D\,(\%) = \frac{m_i - m_r}{m_i} \times 100 \qquad (1)$$

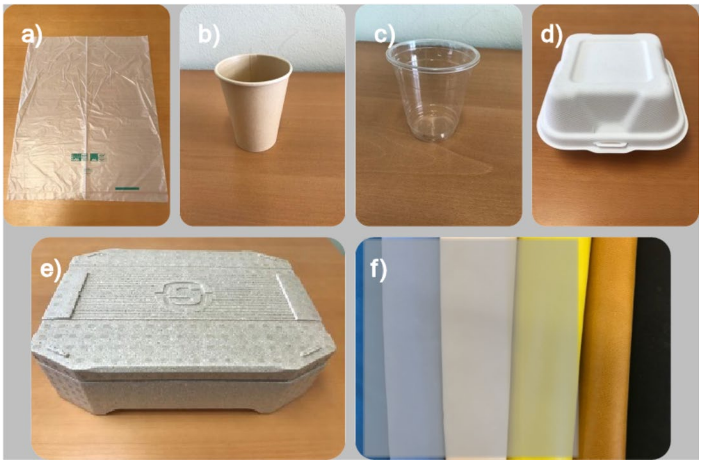

Fig. 1 Test material: (**a**) Mater-Bi Bag; (**b**) Kraft Cup; (**c**) PLA Cup; (**d**) Burger Box; (**e**) Fish Box; (**f**) Rabbit Leathers (the last two on the right)

Fig. 2 The reactors in the laboratory scale test, with the mixture of solid matrix and the test material samples to compost in an oven

Where m_i is the initial dry mass of the test material sample; $m_{r.}$ is the dry mass of the recovered material sample, after being screened, at the end of the test; and D is the degree of disintegration as a percentage.

The validation of this test implies the fulfilment of conditions, such as the reduction of volatile solids (R) shall be greater than 30%, and the difference in the degree of disintegration between replicates shall be less than 10%.

The reduction of volatile solids was verified by calculating the R as a percentage, shown in Eq. 2.

$$R\,(\%) = \frac{[m_i \times (DM)_i \times (VS)_i] - [m_f \times (DM)_f \times (VS)_f]}{[m_i \times (DM)_i \times (VS)_i]} \times 100 \quad (2)$$

Referring to the solid matrix, m_i and m_f are, respectively, the initial and final wet mass of the synthetic waste introduced to the reactor at the beginning of the test; $(DM)_i$ and $(DM)_f$ are the initial and final dry mass of the synthetic waste expressed as a percentage and divided by 100; and $(VS)_i$ and $(VS)_f$ are the initial and final volatile solids content of the synthetic waste divided by 100.

Disintegration Test at Industrial Scale

The degree of disintegration of materials under industrial scale composting conditions was assessed by adapting the ISO 20200:2023 [25] standard procedure to this scale. The results are also interpreted as in the laboratory scale test, in accordance with the EN 13432:2000 [18] standard.

The industrial scale test was conducted in a composting plant located in northern Portugal, with a processing capacity of 60,000 tonnes per year of biowaste from selective collection. The composting process took place in tunnels under controlled conditions, the process naturally generates heat, reaching 55–75 °C. The system used forced air recirculation and water irrigation. The process included two composting phases, each lasting approximately 15 days, in which the material was turned between phases. Screening was performed using a mesh size of 150 mm before the first phase and 60 mm before the second phase. After the second composting phase, a final screening with a 10 mm mesh was carried out. The maturation phase followed, where the material was placed in static aerated piles for 4 to 6 weeks. The total composting trial period for the test material samples was two months.

The solid matrix used in the test consisted of biowaste from the composting plant. The test material samples were exposed to industrial composting conditions, including the plant's specific parameters such as moisture, temperature, turning, screening, and the duration of the composting process. The leather materials were carefully measured and cut into 25 × 25 mm squares samples, and the bioplastic materials were either left intact or roughly broken down.

For this scale test, the reactors consisted of custom-made net bags constructed from non-biodegradable material, with dimensions of 120 × 60 cm and a 1 mm mesh. These bags were specifically designed to meet the requirements of disintegration assays under industrial composting conditions. The mesh bags played a critical role in confining the test material samples, preventing their loss within the compost mass, while allowing the necessary exchange of water and air throughout the process. Their porosity ensured that the samples were exposed to the natural temperature dynamics of the composting mass, replicating the plant composting conditions. Additionally, the design of these reactors facilitated the collection and study of material fractions below 2 mm, enabling further analysis of any microstructures from the test materials that might have persisted without disintegrating.

The solid matrix and the test material samples being tested were suitably mixed with the ratio of 50:1 (wet/wet), the moisture of the mixture was adjusted and the reactors were filled, weighed, tied with ropes connected to buoys for easy identification, and directly buried in the tunnels (Fig. 3). This test includes control reactors only with solid matrix, and five replicates for each for the control and for the test materials.

As mentioned above, there are intermediate processes such as sieving and turning in the composting plant. These processes are simulated in the test reactors when they are temporarily removed from the composting line, and the sieving and turning of the reactor contents are simulated manually. If the test material samples remained intact or were retained on the sieve during the manual turning phase,

Fig. 3 The reactors in the industrial scale test. (**a**) The reactors ready with the mixture of test material and solid matrix, before entering the composting tunnel. (**b**) The reactors in the composting tunnels before being covered with another layer of biowaste

Table 2 Parameters and method description of the analytical determinations

Parameter	Method description
TS	EN 13040:2007 [36]
VS	EN 13039:2011 [37]
pH	EN 13037:2011 [38]
Heavy metals	EN 13346:2000 [39]
Self-heating	EN 16087-2:2011 [40]
Production of CO_2	ISO 14855-1:2012 [41]

Table 3 Characterisation of the test materials and solid matrices

Test material	TS (%)	VS (%)	pH
Mater-Bi Bag	98.3	100	3.3
Kraft Cup	96.1	88.6	5.6
PLA Cup	99.2	99.9	4.5
Burger Box	95.6	99.8	5.2
Fish Box	99.5	99.5	4.7
Rabbit Leather 1	86.5	97.7	3.6
Rabbit Leather 2	90.6	97.8	2.8
Synthetic waste (laboratory scale)	44.7	94.1	7.0
Biowaste (industrial scale)	39.6	73.9	7.3

they have to be weighed and returned to the reactors, however this observation was recorded for future reference.

At the end of the test, the reactors were recovered and transported to the laboratory, where they were weighed, physicochemically characterised, screened, and the degree of disintegration of the test material samples measured. In cases where recovered material above the 2 mm sieve cannot be washed with water due to fragility or other factors that may damage the material samples, washing may be omitted. It should be noted that caution must be exercised in interpreting the results, even if the recovered material has been dry cleaned using brushes to remove particles adhered to its surface and its dry weight has been determined.

As well as on a laboratory scale test, the degree of disintegration can be determined by comparing the initial and final amount of test material sample recovered from the fraction greater than 2 mm in dry weight using the same equation (Eq. 1). The validation of the test with the reduction of volatile solids was verified with the calculation of the R through Eq. 2, and the difference in the degree of disintegration between replicates less than 10%.

Analytical Determinations

Analytical determinations, including total solids (TS), volatile solids (VS), pH, and heavy metals support both laboratory and industrial tests. These determinations provided insight to analyse the solid matrix and the test materials at the start of both laboratory and industrial tests. Table 2 outlines the parameters and standards utilised with all determinations calculated as an average of duplicate or triplicate.

For the analysis of the activity and stability of the solid matrices, two tests were carried out: the self-heating test in Dewar vessels according to the EN 16087-2:2011 [40] standard, and the measurement of carbon dioxide (CO_2) of the solid matrices in vessels according to the ISO 14855-1:2012 [41] standard. Both tests lasted 10 days each.

Statistical Analysis– Non-parametric Correlation

The statistical analysis to evaluate the relationship between the laboratory and industrial composting methods was performed using SPSS software (version 30, 2024). Due to the relatively small number of sample pairs (seven), which, although scientifically justifiable, ends up not meeting the assumptions of normality, so the non-parametric Spearman's rank correlation test was applied.

Results and Discussion

Characterisation of Test Materials and the Solid Matrices

Initially, the test materials and the two solid matrices were characterised physicochemically to understand their initial state and assist in the assembly of the tests (Table 3). The test materials have low initial moisture and high organic matter content, as indicated by their VS values above 88%. The pH of the samples falls within the acidic range, ranging from 3.3 to 5.6.

When it comes to composting, analysing heavy metals is crucial to ensuring that the final quality of the compost is not compromised. In this study, the evaluation of heavy metals was based on the limit values of the EN 13432:2000 standard, and values that are demanding, representing around 50% of the maximum permissible concentrations established for the EU Ecolabel for growing media and organic improvers [42]. Therefore, the heavy metal results of all the test materials under study are in conformity with acceptable values and all are below the limits of standard EN 13432:2000 [18], as demonstrated in Table 4.

The solid matrices used in this study both reached a maximum temperature of 65 °C and activities of 383 mg CO_2/gVS and 258 mg CO_2/gVS, respectively for the laboratory scale solid matrix and the industrial scale solid matrix. A mature compost usually reaches a maximum temperature less than 40 °C, and an activity less than 150 mg CO_2/gVS. Thus, the solid matrices used were active and suitable for the disintegration studies of the test material samples, and present conditions to simulate an intensive aerobic composting process, a requirement of the ISO 20200:2023 [25] standard.

Results of the Degree of Disintegration in the Laboratory and Industrial Scale

The laboratory scale test was conducted in triplicate; and at the industrial scale, the disintegration test was evaluated with five replicas for each test material sample. The results of the averages are presented in Fig. 4, with the corresponding standard deviations. It is imperative to acknowledge that conducting tests at an industrial scale may necessitate a greater number of replicates than those employed at a laboratory scale. This necessity arises due to potential heterogeneity between reactors, which can be attributed to factors such as the substantial volume of biowaste mass and the position of reactors within this mass. Additionally, there is a risk of reactors rupturing during the loading and unloading of the tunnels.

Apart from the Kraft Cup, all test materials exhibited disintegration in the laboratory scale, with an average of over 95% disintegration. The variation between results from the same test material, was less than 10%, which meets the validation criterion of the ISO 20200:2023 [25] standard, also visible by seeing the standard deviation in Fig. 4. These findings align with other bioplastic studies, testing PLA and Mater-Bi Bags where disintegration values typically exceed 90% [30, 43, 44].

Despite consistent results across replicas, with disintegration results in the laboratory ranging from 37 to 45%, the Kraft Cup failed to meet the requirements to be classified as disintegrable. This Kraft Cup comprises two distinct materials, kraft and PLA, with PLA being solely a layer on the inner side of the cup. This research indicates that the sample containing only PLA (PLA Cup) satisfies the criteria for

Table 4 Heavy metal concentration data for the test materials

Test materials	Cd (mg/kgDM)	Cr (mg/kgDM)	Cu (mg/kgDM)	Hg (mg/kgDM)	Ni (mg/kgDM)	Pb (mg/kgDM)	Zn (mg/kgDM)
EN 13,432*	0.5	50	50	0.5	25	50	150
Mater-Bi Bag	< d.l.	0.9	< d.l.	< d.l.	< d.l.	< d.l.	8.1
Kraft Cup	< d.l.	< d.l.	< d.l.	< d.l.	< d.l.	< d.l.	2.9
PLA Cup	< d.l.	< d.l.	< d.l.	< d.l.	< d.l.	< d.l.	59
Burger Box	< d.l.	< d.l.	< d.l.	< d.l.	< d.l.	< d.l.	3.5
Fish Box	< d.l.	< d.l.	< d.l.	< d.l.	< d.l.	< d.l.	43
Rabbit Leather 1	< d.l.	3.3	< d.l.	< d.l.	< d.l.	< d.l.	5.6
Rabbit Leather 2	< d.l.	4.3	< d.l.	< d.l.	< d.l.	< d.l.	9.9

*Limit concentrations by EN 13432:2000.

d.l. detection limit (Cd = 0.7 µg/L; Cr = 2.2 µg/L; Cu = 2.3 µg/L; Ni = 1.6 µg/L; Pb = 11 µg/L; Zn = 0.07 µg/L).

Cd– Cadmium; Cr– Chromium; Cu– Copper; Ni– Nickel; Pb– Lead; Zn– Zinc.

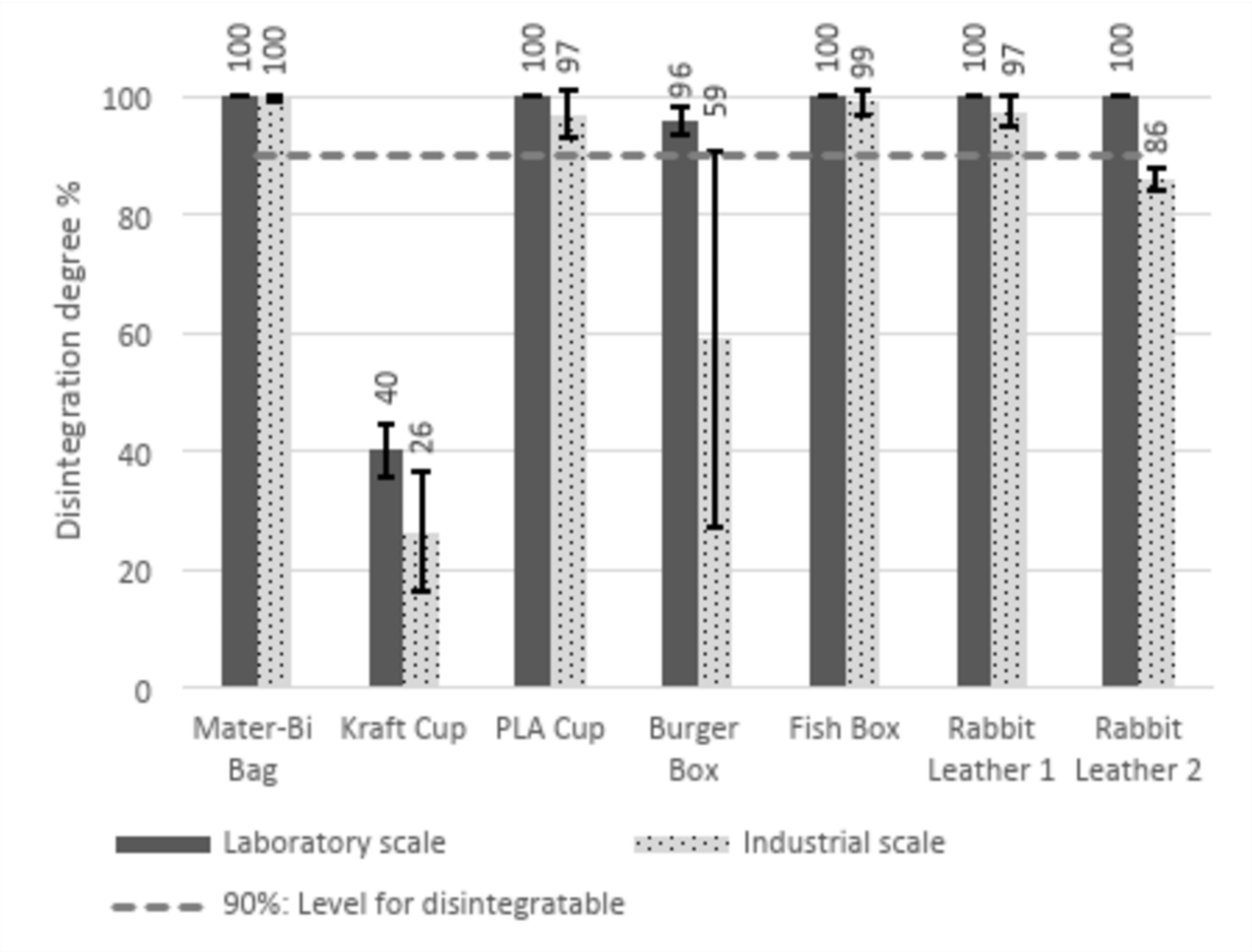

Fig. 4 The mean values for all materials were determined through the calculation of three replicates on a laboratory scale, and five replicates on an industrial scale, with the exception of the Kraft Cup, Burger Box and Rabbit Leather 1, for which three, four and two replicates were used respectively

disintegration and is therefore considered disintegrable, and according to [45] the low degree of disintegration observed in the kraft component could be attributed to the lignin content in the paper sample.

At industrial scale, all the samples, except the Kraft Cup, Burger Box and Rabbit Leather 2, are considered disintegrable, as shown by the result of the degree of disintegration in Fig. 4. The Kraft Cup and the Burger Box had replicates with negative values at industrial scale, which suggests that the samples collected at the end of the test had some solid matrix attached despite being cleaned thoroughly. This usually happens with reactors that have low or no disintegration of the test materials. Therefore, the values of these reactors were not considered while calculating the average of the degree of disintegration. The average degree of disintegration for the Rabbit Leather 1 sample was determined based on two out of five replicates. The calculation was not possible for the remaining three reactors, as two were ruptured, and the result for one reactor was 21%, which may be an outlier which was influenced by the position effect and potential drying of the composting mass, ultimately affecting the ideal moisture and composting process.

The high variability in Kraft Cup and Burger Box sample replicate results on an industrial scale, can be attributed to the positioning in the composting plant within the composting mass. This is a consequence of the plant's unique operating process, which may result in some regions of the mass becoming dry. These observations emphasise the importance of conducting replicates, reinforcing their significance.

According to the results obtained on both test scales, the industrial scale disintegration tests tend to reflect the laboratory results (Fig. 4), including the Kraft Cup and Burger Box samples, because although there are significant differences between the mean results of the methods, on the industrial scale these two materials show individual replicate results that are close to the laboratory scale values.

For test material samples of cellulose materials with PLA film as Kraft Cup sample, [16] showed, for a sample with similar characteristics, values close to 80% in tests on an industrial scale through composting in a pile with a volume of 11 m^3 placed in a box with a cover and between two layers of 2 × 2 mm diameter mesh. In the present study, although the Kraft Cup sample did not achieve average results that would be considered disintegratable by either method, the results on both test scales are consistent, with this material having the lowest disintegration values. The relationship between the methods may be positive, although it is not clear when comparing the values of the averages, unlike the individual values of the replicates on an industrial scale (−19%, -2%, 16%, 29% ad 35%) here there are two values of replicates that are close to the average result in the laboratory.

The average degree of disintegration for the Burger Box sample in the laboratory was 96%. On an industrial scale the average was 59% but with great heterogeneity between replicates (−21%, 21%, 45%, 83% and 88%), possibly related to the factors of the industrial composting plant process in question, where the different positions of the reactors in the composting tunnel might have led to a temporary delay of composting inside the reactor. Therefore, with two of the five reactors showed values higher than 80%, which may suggest that the Burger Box sample might eventually disintegrate at industrial scale with additional composting time than it did when tested at this scale in this composting plant.

The Rabbit Leather 2 sample, despite the consistency and reduced variability between replicates in both methods, on an industrial scale with results close to 90% (84%, 85%, 85%, 87% and 89%), and the sample of this material not only seemed to approximate the results of both methods, but also demonstrated the ability to disintegrate under these composting conditions and with additional time. As for the Rabbit Leather 1, the results for the degree of disintegration fulfil the requirement to be considered disintegrable on both scales, and with the results of the averages being close. Regardless of the nature of the leather or the composting method used, there are no studies in the literature relating to the disintegration of this material in tunnels on an industrial scale. Tests to assess the biodegradability of leather in liquid medium are more common [46–48], although some authors have evaluated the disintegration of chromium-tanned leather and chromium-free alternatives in the laboratory, in accordance with the standard ISO 20200:2004 [27] for plastics [14, 49].

The results of PLA and Mater-Bi Bag samples are consistent with other studies at industrial scale [16, 17], with values close to 100% of th degree of disintegration. The characteristics of these materials make it easier to disintegrate and have better biodegradability than other materials [10], as expected for the Fish Box sample, which is a material made by PLA and PBAT, those being from renewable sources.

As for validation of the tests, according to ISO 20200:2023 [25], the two scale tests are valid as they successfully fulfilled the R requirement of more than 30%, hich indicates that the composting process had taken place according to the decrease in volatile solids index. For the laboratory scale, the added control reactors have an average of 49% for the solid matrix, and the test material samples reactors showed an average of 54%, with values ranging from 46 to 63%. For the industrial scale the values ranged from 32 to 51% in average for each group of test material samples, and the control had an average value of 45% for eight replicates.

The R value holds significance as an indicator of composting prevalence, making it a crucial parameter to consider. This is particularly true on an industrial scale, where there might be limitations in the composting process, such as limitations of water, oxygen, turning or even the composition of the biowaste which may change. These limitations are more easily controlled in the laboratory due to the small scale and the synthetic waste used as a solid matrix always having the same composition.

Regarding the state of the bioplastic samples at the start of the test, which were roughly broken, did not affect their composting process. This can be attributed to composting plants typically handling much larger volumes than laboratory tests, resulting in more rigorous physicochemical and biological conditions, including a wider diversity of microorganisms.

At industrial scale, there are still few studies in the literature assessing the degree of disintegration, where the most studied materials are starch-based bioplastics and PLA [10]. Other materials, mainly for foodware of plant origin such as bagasse, sugar cane and cellulose cardboard with PLA, have also been studied [16, 17].

The methodology used and the results of this study are not comparable with others in the literature, although there are some concordant results. Other authors essentially assess the biological component of the composting process, whether with qualitative or quantitative results, and adapt their methodology to the type of composting plant conditions under study. In this way, they make the methodology unviable for other types of composting conditions, except for [17], who applies the same methodology to different types of composting conditions, although he also focuses only on the biological process.

The method presented in this study is independent of the type of composting conditions used, by respecting not only the biological processes but also other intermediate operations in the composting plants, such as screening or turning. However, the behaviour and results of the degree of disintegration of the materials under test will always depend on the composting conditions in the composting plant, such as the nature of the incoming waste, the temperature reached by the composting mass, aeration, moisture or composting period.

This approach can complement the EN 13432:2000 [18] standard regarding the requirements for recovering materials through composting and biodegradation and responds to the concerns of some composting plants about the behaviour of materials certified as compostable in their facilities. This methodology also has other advantages such as preserving the composting mass from degradation of the materials being tested and allows the fraction below 2 mm to be analysed for microstructure studies, if necessary.

Statistical Analysis Results

The Spearman's rho correlation coefficient applied to the data set revealed a strong relationship between the two methods studied (laboratory scale and industrial composting scale), with a rho = 0.809, which is statistically significant at the 5% level (p-value = 0.028). This result indicates a high level of agreement between the two methods, supporting the proposed methodology for industrial composting scale. The findings confirm that materials exhibiting a high degree of disintegration at the laboratory scale also achieve similar results at the industrial composting scale.

Conclusions

This study analysed the disintegration of emerging compostable materials, including bioplastics and leather, under industrial and laboratory conditions, proposing a methodology for industrial scale disintegration tests that replicates key composting parameters observed in composting plants.

The comparison between the methods revealed a strong and statistically significant correlation between the results obtained at both scales (Spearman's correlation coefficient $R = 0.809$, p-value = 0.028). However, the methods are not interchangeable. Industrial scale validation is crucial for composting facilities to understand the behaviour of the emergent compostable materials they may receive, not only during biological processes, such as composting itself, but also during mechanical processes, including screening and turning.

The results confirmed that most tested materials, except for the Kraft Cup, achieved a high degree of disintegration at both scales, supporting the applicability of the proposed methodology. Although the Burger Box did not meet the threshold for disintegration at the industrial scale, it displayed heterogeneity in the results of individual replicates. Some replicates showed disintegration levels near the threshold for being considered disintegrable and close to the average results observed in the laboratory tests. This finding suggests that the Burger Box could perform better under optimised conditions or with process adjustments, warranting further investigation.

Certain limitations of this study must be acknowledged. Industrial scale tests are subject to the real conditions and processes of composting plants, which limits the degree of control compared to laboratory tests. Additionally, the number of replicates (five per material) may not fully capture potential heterogeneity among samples.

Future studies should also evaluate other aspects of compostability, such as biodegradation and compost quality, including phytotoxicity tests, to ensure that materials truly biodegrade under composting conditions and that the resulting compost does not negatively impact the environment.

The proposed approach provides a practical, safe, and reproducible solution for composting plants handling sustainable materials such as bioplastics and leather, ensuring these materials can be organically recovered without compromising compost quality.

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Author Contributions L.B. was responsible for the study's conception and design, methodology development, data collection, analysis, interpretation of results, and writing the main manuscript text.M.J.C. contributed to the methodology, data collection, analysis, and interpretation of results.A.S. supported the study's conception and design, methodology, analysis and interpretation of results, project administration, resource management, and supervision.All authors reviewed and approved the final manuscript.

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

Declarations

Competing Interests The authors declare no competing interests.

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