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TESI DI LAUREA

DISINTEGRATION OF DIFFERENT MATERIALS UNDER SIMULATED COMPOSTING CONDITIONS IN A LABORATORY-SCALE TEST

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ABSTRACT

The market of bioplastic has largely developed in the recent years due to an increasing awareness of the negative effects that the production and use of conventional plastic has on the environment. Consequently, compostable packaging production will continue to grow in the future, but many treatment plants are still not ready to accept this kind of waste. The compostability of these products is certified according to international Standards that don’t consider the real conditions of composting facilities. This can lead to a misassessment of the actual composting behaviour of the tested products: materials can be declared compostable even if under industrial plants conditions they don’t behave as such.

A laboratory scale test was done on compostable bags, PLA cups, kraft cardboard and conventional PET (negative control) to compare the composting process of these materials in different solid matrixes. To do so, Standard ISO 20200 was taken as the reference procedure followed in the lab to assess the disintegration degree of the chosen materials. Three different inoculums were used: Synthetic Waste, Fresh Waste and Mature Compost. The first one is the inoculum pointed out by the Standard, while the other two were selected because more representative of the real environment of industrial composting plants.

At the end of the test, the disintegration degree of the materials in the different inoculums was calculated. Even though not all the results passed the validity check, they highlighted the presence of discrepancies between lab simulations in optimal conditions, and lab tests that take into consideration aspects mirroring a more realistic situation of a specific treatment plant, including particular operational conditions and the type of waste involved.

Keywords: Disintegration; compostable packaging; ISO 20200; lab-scale test.

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CHAPTER 1: INTRODUCTION

1.1 OBJECTIVE OF THE THESIS

The focus of the thesis will be on the implementation of ISO 20200:2015 “Plastics — Determination of the degree of disintegration of plastic materials under simulated composting conditions in a laboratory-scale test”. This is an important standard because passing a disintegration test is one of the requirements to declare if packaging is compostable or not, according to standard EN 13432. Other than the conventional procedure that involves the use of a synthetic waste inoculum, two other types of inoculums will be tested: “fresh food” (after about 1 month in the composting tunnel) and “mature compost”. The quality of both is not actually known since mixed waste is present too. 4 materials will be studied to assess their degree of disintegration: compostable bags, compostable PLA cups, kraft cardboard and PET (as a negative control material).

The objective of the thesis is to demonstrate that the use of a synthetic waste inoculum suggested in ISO 20200 and the composting conditions maintained in the lab are not representative of the real situation of industrial composting plants. This can cause some problems, for example materials can be declared compostable even if in industrial plant conditions they are not. To better mimic the reality, both fresh compost and mature compost are used, and their results will be compared with the ones obtained with synthetic waste.

As times go on more sustainable materials will be used to substitute conventional ones, as in the case of compostable plastics. The quantity of compostable packaging produced will increase more and more, and they will need to be disposed of after their use. If the compostability of these materials is not tested in the best way possible, their treatment can cause issues to the plants and to the quality of the final product, which in this case is compost.

This research is related to another study conducted by NOVA University, that is part of a bigger project managed by SPV (Sociedade Ponto Verde), a private not-for-profit organization founded by a group of companies to promote selective collection and recycling in Portugal, with a focus on packaging waste.

1.2 OUTLINE OF THE THESIS

The thesis is organized into five chapters.

The first chapter include a brief overview of the objectives and contents of the thesis.

The second chapter collects all the important information on which the experimental research finds its theoretical foundation. More precisely, it is a comprehensive literature review on bioplastics, industrial composting, compostable packaging, legislation and standards, and finally examples of studies on disintegration tests with different materials and at different scale levels.

The third chapter is focused on the materials and methodologies used in the laboratory experience, from the characterization of the samples and inoculums, to the preparation of the actual lab test, including all the variations that were adopted making the test differ from the actual standard procedure ISO 20200.

The fourth chapter shows all the results of the analysis done during the experimental work, discussing them, and providing a comparison on the behaviours of the different tested materials in the different inoculums.

The fifth chapter summarises the problems and results of the research and provides also further suggestions for future studies dealing with the topic.

Finally Annex I collects all the tables showing the weights of the reactors during the test periods, measured in the same days as the scheduled operations.

CHAPETR 2: LITERATURE REVIEW

2.1 BIOPLASTICS

2.1.1 Introduction on the matter of bioplastics

Plastic is a valuable resource, and it is widely used in various applications because of its unique properties. It is a material with many advantages: lighter than other materials, versatile, good safety and good health properties for food packaging, high durability and resistance, and many more. At the same time, it is also known that plastic production and use has its own considerable drawbacks. Most of the plastic produced is made from fossil fuels with a process that has a negative impact in term of greenhouse gas emissions and pollution. The recycling rate of plastic is still relatively low. It is a material that can easily be dispersed into the environment through littering or improper waste management, and persists for years. Plastic particles can also enter the food chain, having possible effects on human health.

Nowadays there is an alternative to almost all conventional plastic materials: bioplastics. They can have similar or even identical properties to conventional plastics, with some additional advantages (e.g. they can be more sustainable if properly managed during their life cycle). Currently bioplastics represent only 1% of the 390 million tonnes of plastic annually produced, but their production rate has been increasing in the last years and is expected to continuously increase in the future, reaching 6.3 million tonnes in 2027, with more than half of it being biodegradable (European Bioplastic, 2022). Even though bioplastics are mainly used in packaging production, they are also present in different market sectors, such as food-services, agriculture, consumer electronics, transport, etc. Bioplastic for packaging can be rigid or more flexible. Also used for food protection, its characteristics are comparable to the ones of conventional plastic food films, and the application of bioplastic materials in this field will be even more efficient improving properties like antimicrobial barrier. Bioplastic can be used to produce single-use applications as well as reusable ones (European Bioplastic, 2021).

People often have difficulties when it comes to distinguish the different kinds of bioplastics because there is not enough informational initiatives on the topic. Bioplastics can either be bio-based, biodegradable or both; biodegradable bioplastics can be compostable and not-compostable. A simple division in categories could be:

- Bio-based plastics, that are made (entirely or partially) from biological raw materials; they can either be biodegradable or not.
- Biodegradable plastics, that have the ability to biodegrade under specific conditions and in a specific environment; they can be bio-based or made from fossil fuels.
- Industrially compostable plastics, biodegradable under composting conditions characteristic of and industrial composting plant or anaerobic digestion plant followed by a composting step.
- Home compostable plastics, biodegradable under composting conditions in a home composter that differs from industrial plant in terms of volumes and operative conditions (e.g. lower temperature than industrial plants).

(EEA, 2023)

2.1.2 Most used bioplastics and their properties

PLA (Polylactic Acid), is a bio-based and biodegradable polymer, produced mainly from acid fermentation processes instead of chemical synthesis. The most common raw materials used in this process are corn starch, cassava roots, sugarcane and potatoes. Products made with this material have a good transparency, very high rigidity, ability to act as a barrier and to withstand different processing conditions. On the other hand, they are not resistant to high temperatures and have a slow biodegradation rate.

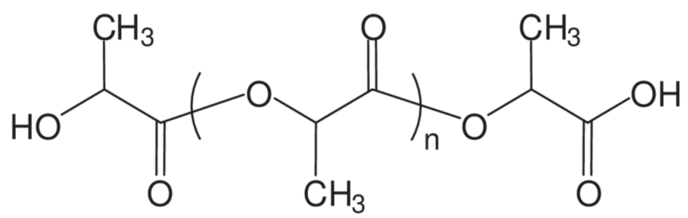

Figure 2.1: PLA chemical structure (source BioplasticsNews.com)

PHAs (Polyhydroxy Alkanoates) are a group of biodegradable polymers. They are synthesized by bacteria through anaerobic digestion of lipids or sugars. Food and agricultural wastes such as mango peel, potato peel, wheat bran, rice husk, straw and similar are also utilized in the production of PHAs. They are completely biodegradable in water, soil and compost, can withstand temperatures up to 180°C and they are non-toxic. However, they have issues in some applications because of their brittleness and sensitivity to thermal degradation.

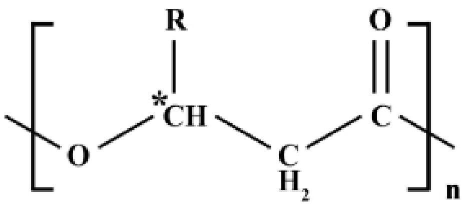

Figure 2.2: PHAs chemical structure (source < https://doi.org/10.3390/polym6030706 >)

PBS (Polybutylene succinate), a polymer synthetized by monomers produced from fossil-based sources or by fermentation through bacteria. Nowadays, products of 100% bio-PBS are available in the market. It’s a flexible material, with a good biodegradation rate, heat resistance and other mechanical properties (e.g. tensile strength, impact strength, rigidity).

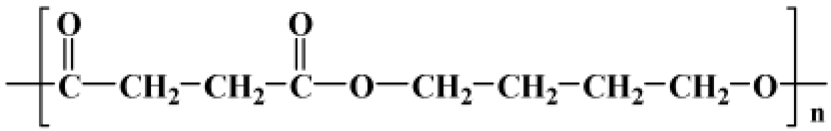

Figure 2.3: PBS chemical structure (source <https://doi.org/10.4236/ojpchem.2019.91001>)

Bio-PE (Bio-polyethylene) has similar characteristics to conventional PE, but it’s synthesized by microbial fermentation. It has good performances, and it is also cost effective, with the big disadvantage of being non-biodegradable. Attempts are being made to recycle Bio-PE to solve the problem of non-biodegradability.

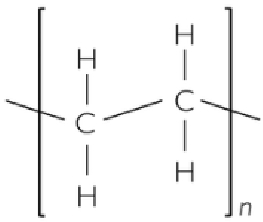

Figure 2.4: Bio-PE chemical structure (source <https://doi.org/10.1063/5.0023759>)

Bio-PET (Bio-polyethylene terephthalate) is a polymer whose monomer is obtained from sugarcane-derived ethylene after fermentation of glucose. Bio-PET packaging has similar properties to other conventional PET products. It is a non-biodegradable plastic, but can be degraded by bacteria Nocardia with its esterase enzyme (Saharan R. et al., 2022).

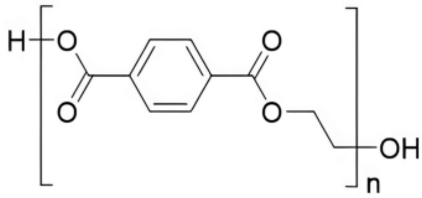

Figure 2.5: Bio-PET chemical structure (source <https://doi.org/10.1021/acsomega.2c01834>)

2.2 COMPOSTING

2.2.1 Phases of the process

Compost is an essential material that allows the improvement of soil quality, especially when used for agricultural purposes. It brings nutrients for the plants growth, it helps improving the structure and physiochemical properties of the soil, it allows more aeration and prevents the soil from drying out in case of lack of water. This substance is by definition "an organic matter (plant and/or animal residues), which has been degraded by the action of microorganism i.e., bacteria, fungi, actinomycetes etc. over a period of time". It is the result of composting, an aerobic process involving the activity of different microorganisms that, under optimal conditions, are able to transform the organic waste in a homogeneous and plant available material. These bacteria use N, C and O for their metabolic process, generating heat and a solid material, the compost. Composting can be divided in four phases:

1. Mesophilic phase (also called hot phase) is the initial stage when the temperature rises to 45°C during the first few days (or hours) due to the metabolic activity of microorganisms. During this stage, pH can drop to 4.0 because of the production of organic acids after the decomposition of sugars.
2. Curing phase (thermophilic and hygienization phase) starts when the temperature rises above 45°C and the mesophilic microorganisms are replaced by thermophiles, other aerobic bacteria working in higher temperature ranges. They are responsible for the degradation of more complex substances, such as cellulose and lignin, converting also N in ammonia, which results in a pH rise. Then, temperature will rise above 60°C, favouring the presence of actinobacteria, useful for the decomposition of other complex carbon substances. This phase is relevant also due to its hygenization ability, a function of the process that utilises high temperatures to eliminate pathogens from the compost.
3. Cooling phase (also called mesophilic phase II) is characterized by a temperature drop meaning that the metabolic processes of the microorganisms are coming to an end. When temperature goes below 45°C mesophiles reappear together with some fungi, and the pH usually remains slightly alkaline. This stage can last several weeks.
4. Maturation phase is the final stage of the composting process. Ambient temperature (20-25°C) is reached, and a series of secondary reactions takes place, leading to the formation of humic acids, important components of a good quality compost.

2.2.2 Factors affecting the process

As already said, composting is a biological process. It involves the activity of various types of microorganisms which are highly susceptible to environmental changes and need optimal conditions to be maintained in order to reach the end of the process with good results.

Parameters affecting composting are:

1. Aeration (Oxygen).
2. Moisture.
3. Temperature.
4. Carbon-nitrogen (C:N) ratio.
5. pH.
6. Particle size.

2.2.2.1 Aeration (Oxygen)

The microorganisms engaged in the process are aerobic, so by definition they need oxygen (optimal level 10%). Aeration also prevents soil compaction and water stagnation. Levels of O_2 below 5% result in excessive water content and consequently the creation of anaerobic areas. On the other hand, if the level of O_2 is above 15%, water evaporation is excessive, and consequently the composting process stops due to lack of water.

2.2.2.2 Moisture

Water is fundamental to the microbial activity since bacteria use it to transport nutrients and energy inside their cells to start their metabolic process. The optimal level of water content is about 55%. When the level drops below 40%, there is not enough water for the microorganisms to continue their activity; when the level rises above 60%, water in excess accumulates and creates anaerobic areas.

2.2.2.3 Temperature

The optimal temperature changes during the different phases of the composting process because bacteria working on them are different, and work better under different environmental conditions. Low temperatures can be caused by insufficient moisture, lack of nutrients or low C:N ratio, all conditions that prevents microbial activity to develop. Furthermore, temperature can also drop in case of insufficient amount of material or inadequate pile shape. On the contrary, temperatures can rise above 70°C when there is not enough aeration.

2.2.2.4 pH

As for the temperature, each microorganism has its own ideal pH. Generally, the ideal pH range during the process should be 5.8 – 7.2. A value of pH under this range is obtained when there is an excess of organic acids, mostly released by kitchen waste and fruit. If the pH is higher than the optimal value, it means that there is an excess of nitrogen in the source material.

2.2.2.5 Carbon-nitrogen (C:N) ratio

The C:N ratio depends on the components of the initial material used to produce compost. In case of a surplus of carbon, the process cools and slows down. When there is an overabundance of nitrogen, more heat is produced, and ammonia is released. The ideal ratio ranges between 35:1 and 15:1.

2.2.2.6 Particle size

Particle size and material density affect microbial activity because they determine the accessibility to the substrate. The ideal range is between 5 cm and 30 cm. If the particles diameter is too big, there is an excess of aeration; if the particles are too small, the soil is compacted, and low aeration combined with accumulation of water will result in anaerobiosis (Agriculture and Food Newsletter, 2021).

2.2.3 Biodegradation and composting: what's the difference?

Biodegradation is a process in which organic materials are converted into CO_2, water and biomass thanks to the action of microorganisms. The process depends on environmental conditions and on the composition of the material itself, so the outcome can vary considerably. Under specific conditions, a biodegradable material can be compostable. These specific conditions are described in standards such as the EU standard on industrial composting for packaging (EN 13432) or more in general for plastic materials (EN 14995). If materials comply with these standards, they can be labelled and certified as compostable (European Bioplastics, 2016).

Composting is the controlled decomposition of organic material (e.g. yard trimmings, kitchen scraps, wood shavings, cardboard etc.). Its aim is to recover organic matter in the form of compost, that based on its quality can be used for soil improvement in agriculture or covering material in landfills. There are two types of composting processes: industrial composting and home composting.

Industrial composting is the controlled biological decomposition of organic waste under monitored aerobic conditions and, thanks to the production of a considerable amount of heat, it's able to reach thermophilic temperatures (50-60°C or more).

On the other hand, home composting does not deal with a large quantity of waste, so the heat produced is less and the maximum temperatures reached are 20-45°C (mesophilic range) (European Bioplastic, 2015).

Up to now, there is not a common international standard on the certification of home compostable materials. Home composting is not a professional waste management and, despite the presence of guidelines on how the process should work, it is difficult to monitor, and its conditions may vary situation-to-situation. Because of this, most of the compostable plastics present on the market are certified referring to industrial composting standards. However, some national certifications, such as "OK compost home" by TÜV Austria Belgium are present. (European Bioplastic, 2018).

2.2.4 Industrial composting

The process is divided into two phases: active composting and curing. Active composting (minimum 21 days) starts with the growth of microbial population in the organic waste mass, which is then responsible of the conversion of waste into CO_2, water and biomass. They release high amount of energy in the form of heat; this causes the temperature to rise, and consequently mesophilic microorganisms (working in ambient temperature) die and are replaced by thermophilic microbes. At this point the temperature in the composting heaps ranges between 50°C and 60°C (or more if there are pathogenic microorganisms to eliminate). After this, the curing phase starts and the decomposition rate and temperature decrease; the process becomes slow and steady, and compost matures producing humic substances. At the end of the stage, compost is analysed to verify if it meets the quality standards (European Bioplastics, 2009). Sometimes, the composting process is preceded by anaerobic digestion which leads to the formation of biogas as an energy source (Kędzia G. et al., 2022).

Industrial composting plants are large-scale facilities dealing with significant amounts of putrescible waste. The table below shows the typical feedstocks for industrial composting.

Table 2.1: feedstocks for industrial composting (European Bioplastic, 2009)

Description	Waste EU-code	Notes
Kitchen and canteen waste (foodwaste)	20 01 08	from households, resaturants, canteens, bars, coffee-shops, hospital and school canteens, etc.
Waste from public markets	20 03 02	only biodegradable materials equivalent to codes n° 200108 and n° 200201
Garden and park waste (yardwaste)	20 02 01	from private gardens and public parks and areas, etc.
Wood waste	20 01 38	not containing dangerous substances no furniture and bulky household-waste

SOURCE:
EU CODES ACCORDING TO COMMISSION DECISION N° 2001/118/EC

As already said, industrial composting differs from home composting because process parameters are controlled; these process parameters are:

- size of particles,
- moisture content,
- aeration,
- temperature,
- pH,
- C/N ratio.

Technologies which are usually applied in these types of plants are windrows, aerated static piles, tunnels or vessels. Different aspects influence the interaction between compostable plastics and technologies present in composting facilities. One of them is thickness: when a material is certified as compostable, the maximum thickness (the thickness of the tested sample) should be specified. Then, if the composting plant is equipped with an upstream screening system to remove contaminants (not all are removed), compostable plastics can be excluded from the composting process with other materials. This is avoidable if a second stage of manual sorting is introduced.

Table 2.2: benefits and challenges of compostable packaging

BENEFITS	CHALLENGES
Higher participation in biowaste collection	Labels are not always enough clear for citizens to understand their meaning
Increase quality of source separation	Exclusion of compostable packaging from composting process due to intensive screening
Reduction of contamination by non-compostable plastics	Lack of uniformity of waste management system at national and local level
Higher quality of final compost	Need of development of appropriate industrial composting infrastructures
Alternative recovery option for products difficult to recycle because contaminated by organic waste	

The table above summarises benefits and challenges on the relationship between the use of compostable packaging and industrial composting facilities (European Bioplastics, 2009).

2.3 COMPOSTABLE PACKAGING

Compostable packaging and plastics are defined as packaging and plastics which, when introduced in a composting plant together with organic waste, are biodegraded under specific conditions and bring no negative effects to the process, the compost production, or the environment in general (European Bioplastics, 2009).

2.3.1 Is it a solution? Challenges and opportunities concerning the use of compostable packaging

Fossil-based plastics are recognized to be one of the causes of natural resources depletion. Their production and disposal lead to negative impacts on the environment. In fact, conventional plastics take up to 1000 years to decompose, and even though their recycling rate is increasing, less than 30% of collected plastic waste in Europe is recycled, with 31% being dumped in landfill and 39% incinerated. Another worrying reality is that over 80% of the marine waste contamination is caused by littering of plastics products, mostly single-use ones (Koenig-Lewis et al., 2022).

In this thesis the focus will be on compostable packaging, which appears to be a solution regarding plastic pollution. However, it's not acting at the source of the problem. As reported by the Ellen MacArthur Foundation, single-use products, including compostable ones, consume more energy and cause more GHG emissions than recycled or reused alternatives. Then, there is the problem of collecting, sorting correctly and processing the packaging in industrial composting plant, because they can only totally degrade if some specific conditions are present. Moreover, there are not enough certified industrial composting facilities to actually be able to deal with the total flux of compostable packaging/SUP waste and transporting these materials to the proper plant increases the carbon footprint. A remarkable amount of compostable products exit the recycling stream, and is either disposed of in landfill or dispersed in the environment, where the composting process is not completed causing the same problems as other types of conventional waste.

Composting is the biological equivalent of recycling, and it's known that these kind of recovery processes won't be enough to overcome the huge amount of waste the population produces. The attention should be on reducing waste production at the base, not on recycling it after it's generated. That being said, in some cases compostable alternatives may be the best solution in a circular economy perspective when they contribute to increase the nutrients present in the soil or when they avoid potential contamination of organic materials (e.g. compostable fruit stickers, compostable food packaging, etc). In this way good quality compost can be produced and sold to farmers, closing the system loop (Ellen McArthur Foundation, n.d.).

Some studies claim that composting is not true recycling. As stated also by the European Environment Agency, compostable packaging doesn't contain nutrients to enrich the soil or compost, and during the composting process it is converted into water and carbon dioxide: the original material is lost and cannot be used to generate new products. However, composting is a biological process where organic materials are reprocessed into compost, and this cannot be analysed in the same way as a mechanical recycling process. Compost can be considered a secondary raw material if it complies with the end-of-waste criteria, which are:

- The product can be used in agriculture/horticulture.
- The product is sold as a soil improver.
- The product must respect the EU requirements on compost quality.
- The product does not cause harm to human health or the environment.

Therefore, if all these conditions are met, composting can be regarded as a form of recycling. Compostable packaging can be compared to cellulose in terms of its role in the composting process: it is a source of energy and carbon fairly easy to biodegrade. Thus, even though it does not contain nutrients, it is an essential element for the correct functioning of compost production and the material is not "lost" when CO_2 is produced, but it is used as source of energy for the microorganisms activity (Francesco Degli Innocenti, 2021).

Another possible critique aspect is that bio-based compostable plastics are made using raw materials coming from cultivations in crops dedicated only for that aim, and this may cause competition with food production, generating an ethical problem. From an economic point of view, in some cases compostable plastics are more expensive than conventional ones, so the latter are preferred. Furthermore, most compostable packaging still has limited properties in respect to conventional packaging, thus research on how to improve their characteristics should be done. In addition, the extended producer responsibility of the compostable packaging manufacturer should also cover the end-of-life phase of their final products, including collection and processing (Kędzia G. et al., 2022).

Many consumers struggle to understand environmental claims and labels; this is a barrier in the management of compostable packaging because it is not always disposed of as it should be. To avoid this, informational and awareness-raising campaigns must be adopted with the aim to communicate in a transparent way with the citizens, clearing all their doubts on the matter. All these improvements will be possible only if governments commit to the arrangements of funds destined for further research, innovations and investment into the compostable plastics sector (EEA, 2023).

On the positive side, a study showed that the public already implicitly and explicitly perceive compostable packaging more positively from an environmental perspective, but also more healthy than conventional plastic packaging (Koenig-Lewis et al., 2022).

2.3.2 Legal framework

There is currently no comprehensive EU law on biobased, biodegradable and compostable plastics. However, some other directives refer to them directly or indirectly.

- <u>Directive 94/62/EC on packaging and packaging waste.</u>
  Its aim is to harmonise national rules concerning the management of packaging and packaging waste in the EU, by preventing and reducing its environmental impact. The last version of this directive is Directive (EU) 2018/852, with a focus on prevention of packaging waste production, reuse, recycling and recovery instead of disposal in landfills, to reach specific targets set by law. It covers all types of packaging and packaging waste, regardless of their materials and origin. This document also refers to compostable and biodegradable packaging waste, specifying the cases in which a recovery (composting process) is possible, adding that oxo-degradable plastic packaging (i.e. plastic with additives that helps it to break down into microscopic particles, with possible dispersion of microplastic into the environment) are not considered biodegradable plastics. Other topics contained in the directive are the extended producer responsibility and the reporting system between member states (EUR-Lex - L21207 - EN - EUR-Lex, n.d.). In November 2022 the European Commission proposed a revision on this Directive, changing it into a Regulation. They propose to expand the list of

applications for which the use of compostable packaging should be mandatory (EUROPEN, 2022).

- Directive (EU) 2019/904 on reducing the impact of certain plastic products on the environment.

  The scope of this directive is to prevent and reduce the impact of SUP products on the environment, ensuring that more sustainable alternatives are placed on the market, moving towards a circular economy. Different measures are adopted, such as bans on specific single-use or oxo-degradable products and labelling to avoid littering. Also, some biodegradable or compostable plastic should apply this labelling system. The directive doesn't refer in different ways to conventional, bio-based, biodegradable or compostable plastics, they are all considered together as plastic materials, with no exceptions. Anyway, in 2027 a review of the Directive will take into consideration new researches and discoveries concerning the real environmental impact of bioplastics and the law will may undergo some changes (European Bioplastics, n.d.).
- Directive (EU) 2015/720 on plastic bags, amendment to the Packaging and Packaging Waste Directive 99/62/EC

  It was introduced to reduce the problems caused by an intensive and unsustainable use of lightweight plastic carrier bags. The need to reduce the use of these kind of bags, strengthens the importance of biodegradable and compostable bags as a more sustainable alternative (European Bioplastics, 2015).

Other strategies were taken to regulate the use of plastic materials, including bioplastics, such as the European Green Deal (2020) and the Plastics Strategy (2018).

2.3.3 Standards, certifications and labelling

Based on the environment in which the tests are done, different European standards are involved in the assessment of compostability of plastics materials. Some comprehensive standards, for example in case of assessing biodegradability in water, are yet to be implemented. If materials are tested and comply with the requisites of these standards, they may be then certified and labelled by a third party.

All is resumed in the table below.

Table 2.3. European standards for compostability and biodegradability of plastics in different environments and selected logos and certification schemes (EEA, 2020)

Environment	European Reference Standard	Certification and logos	Notes
Industrial composting	EN13432	OK compost TÜV AUSTRIA INDUSTRIAL; kompostierbar; CIC COMPOSTABILE; INDUSTRIAL COMPOSTABLE DIN Geprüft	EN 13432 refers to packaging. In addition, EN 14995 is a similar European standard for compostability of non-packaging products in industrial composting plants.
Well-managed home composting conditions	No European standard	HOME OK compost TÜV AUSTRIA HOME; HOME COMPOSTABLE DIN Geprüft	The OK compost home label builds on a certification scheme developed by TÜV Austria Belgium NV. The DIN-Geprüft Home Compostable label is based on French standard NF T51-800 and/or the Australian standard AS 5810. National standards also exist in Belgium and Italy. A draft European standard exists for plastic carrier bags suitable for treatment in well-managed home composting installations (prEN 17427:2020).
Soil	EN17033	BIODEGRADABLE IN SOIL DIN Geprüft	EN17033 applies to mulch films only.
		SOIL OK bio-degradable TÜV AUSTRIA SOIL	Based on a certification scheme developed by the label provider, but can be compliant with EN 17033 on request by adding two additional ecotoxicity tests.
Freshwater	No European standard	WATER OK bio-degradable TÜV AUSTRIA WATER	Based on a certification scheme developed by the label provider.
Marine water	No European standard	MARINE OK bio-degradable TÜV AUSTRIA MARINE	Based on a certification scheme developed by the label provider, using American standard ASTM D7081 (withdrawn) as a basis.

Certifications and labels are an additional assurance of a product's compostability. Nevertheless, compostability tests are done in conditions far from the reality (e.g. lab-scale or pilot-scale tests) that can enhance the composting process. It can happen that even if products are compostable according to the standard, they don't actually degrade how they should when treated in an industrial composting plant (EEA, 2020).

The main Standards present in Europe are:

- EN 13432 - Requirements for packaging recoverable through composting and biodegradation - Test scheme and evaluation criteria for the final acceptance of packaging.
- EN 14995 - Plastics - Evaluation of compostability - Test scheme and Specifications
- ISO 18606 - Packaging and the environment – Organic Recycling
- ISO 17088 - Specification for compostable plastics

They use different tests (regulated by standards themselves) or have slightly different requirements, but all of them are based on the same criteria: to assess compostability, tests concerning characterisation, biodegradation, disintegration and ecotoxicity must be done (TUV Austria, n.d.).

1. Characterisation: usually volatile solids are tested, together with heavy metals (zinc, copper, cadmium, etc.) concentration.
2. Biodegradation: biodegradability in compost (ISO 14855) or in water (ISO 14851 or ISO14852), and optionally anaerobic tests in water (ISO 14853) or in presence of high solids concentration (ISO 15985) are done. The accepted level of biodegradability is 90% after 6 months at T=56-60°C
3. Disintegration: lab-scale (ISO 20200) or pilot scale (ISO 16929) test under simulated composting conditions. At least 90% disintegration must be reached after 3 months at high temperature (40-70°C range).
4. Ecotoxicity: around the world, different standards are used based on the limit values of toxic substances that can vary moving from a country to another. Usually, the germination test is applied, which consists of cultivating some kinds

of seeds in soil mixed with a specific quantity of the analysed compost (Casale A., 2013).

Several methodologies are implemented to measure compostability or biodegradability of plastic materials. The easiest one is checking if there are still some visible plastic pieces to the naked eyes. Moreover, measuring CO_2 (aerobic conditions) or CH_4 (anaerobic conditions) it is possible to calculate the organic matter converted into these products during the microbial activity. Spectroscopy (IR, NMR and FTIR) is also used to assess the biodegradation process through the changes in the spectrum of bioplastics. Lastly, mass loss is calculated monitoring the mass at the start and at the end of the process, after sieving the sample with meshes of different sizes and drying them (Ruggero F. et al, 2019).

Outside the topic of composting, it is important to note the importance of biodegradability assessment tests in other environments, such as soil and water.

Soil is a rich ecosystem in terms of biodiversity, including an extended population of microorganisms, that actively participate in biodegradation. However, environmental conditions like pH, temperature, moisture and so on, are influenced by natural variables that differ all around the world, affecting the biodegradation process in soil. Currently, there are no European or international official standards on how to determine the biodegradability of plastic materials in soil, thus a mix of techniques is used. Lab tests are performed if the main objective is to measure the intrinsic biodegradability, otherwise the biopolymer is directly buried in soil under natural conditions, and this allows also to observe the possible ecotoxic effects on the environment. The most famous standards for biodegradation of plastic in soil are the American ASTM D5988-18 “standard test method for determining aerobic biodegradation of plastic materials in soil” and the European ISO 17556:2019 “plastics—determination of the ultimate aerobic biodegradability of plastic materials in soil by monitoring the oxygen demand in a respirometer or the amount of carbon dioxide evolved”. To test ecotoxicity, seed germination and plant growth are applied.

The effect of the presence of biodegradable plastics in aquatic systems is a problem that is only been taken into consideration in recent years, so lot of research still needs to be done. This situation represents a threat to human health too, as microplastics are ingested by aquatic organisms and can enter the food chain reaching the human body. The aquatic ecosystem includes a huge variety of habitats and each one of them with markedly distinct environmental conditions that influences plastic biodegradability, making it particularly difficult to predict. Furthermore, biodegradation should be assessed for both fresh water and marine water, and to be even more precise the various strata present in water bodies should be taken into consideration because they are characterized by different behaviours. Up to now, there are no European or international standards focused on studying biodegradation in freshwater systems, and to our knowledge, there is no development for creating one related to inland water bodies. However, regarding marine habitats, some ISO and ASTM standards are available:

- ISO 18830:2016 "plastics—determination of aerobic biodegradation of non-floating plastic materials in a seawater/sandy sediment interface—method by measuring the oxygen demand in closed respirometer"
- ISO 19679:2020 "plastics—determination of aerobic biodegradation of non-floating plastic materials in a seawater/sediment interface—method by analysis of evolved carbon dioxide"
- ISO 14853:2016 "plastics—determination of the ultimate anaerobic biodegradation of plastic materials in an aqueous system—method by measurement of biogas production"
- ISO 23977-1:2020 "plastics—determination of the aerobic biodegradation of plastic materials exposed to seawater—Part 1: method by analysis of evolved carbon dioxide"
- ISO 23977-2:2020 "plastics—determination of the aerobic biodegradation of plastic materials exposed to seawater—Part 2: method by measuring the oxygen demand in closed respirometer"

These standards refers to lab tests, but the actual necessity is to understand how to assess biodegradation in situ, in real-life conditions, so some other new standards trying to do this were introduced: for sea surface ISO 15314:2018 was created, "plastics—methods for marine exposure" and for seafloor and beach scenario ISO 22766:2020 exists, "plastics—determination of the degree of disintegration of plastic materials in marine habitats under real field conditions". As expected, these standards present some limitations, for example the fact that they have problems concerning the replicability of the experiment and they require exorbitant costs (Pyres J. et al., 2022).

2.4 DISINTEGRATION TEST

2.4.1 Lab-scale and pilot-scale disintegration tests

Multiple studies on how to determine the disintegration degree of plastic packaging were made, some following every step of already existing standards (e.g. ISO 20200, ISO 14045, etc.), others changing some conditions.

Mater-Bi, a starch-based polymer, was tested following step by step the ISO standard 14045 using as waste matrix a mixture of food waste and green waste, put inside polypropylene containers (12L) ensuring optimal composting conditions. A kinetic analysis was carried out: process parameters were changed during the experiment to understand how environmental conditions would influence the biodegradability of the polymer, and more in detail its component, which in this case are starch, additives and PBAT (polybutylene adipate terephthalate). The results show that starch and additives undergo a strong degradation during the first days of the composting process, which then continues with a slower rate, but it is not influenced by variations in temperature and moisture. On the other hand, degradation of PBAT is significantly affected by these parameters, particularly by water content that can prevent the transformation of the biopolymer into stable organic matter. Since starch is a natural polysaccharide, it is readily biodegradable and its degradation rate will obviously be faster than the one of a more complex and synthetic polymer, as in the case of PBAT. All these data were collected thanks to the application of a synergic approach based on the use of different instruments and techniques, namely TGA (thermogravimetric analysis), FTIR (Fourier transformed infrared spectroscopy) and SEM (scanning electron microscope) (Ruggero F. et al., 2020).

The degradation of the same material, cut into two different sizes, 50-70 mm and 20-30 mm, was tested in small-scale composting heaps (30kg of organic waste each). Another heap containing only organic waste was present. Predictably, the initial dimensions of the plastic samples impacted the fragmentation process. At the end, the compost coming from all the heaps met the requirements for good quality compost (in Italy). The results obtained for the heaps with compostable plastics displayed no substantial difference compared to the one with only organic waste, so bioplastics do not seem to impact final quality compost. However, at the end of the test, the degradation of bioplastics did not meet the requirements set by the standard for compostability (Lavagnolo M. et al., 2020).

In another study Mater-BI is tested together with other materials to allow a comparison between their different degradation processes. The other materials are PBAT Ecoflex and PLA (polylactic acid), the last one present in its rigid form. The composting test was carried out following a modified version of the standard EN ISO 14855: three replicates for each materials containing mature compost as inoculum were placed in cylindrical glass reactors (5L) for a maximum of 60 days. As usual, optimal composting conditions were assured during the test. MB and PBAT degradations were almost identical; this means that PBAT is degraded both as pure material and as part of MB, but also that starch present in MB does not contribute much in the degradation of MB. The degradation degree of PLA was low, opposed to other previous studies that were performed using film material, meaning that thickness is an important aspect to consider in disintegration. More in-depth analysis on PBAT showed a 50% loss in molecular weight and its tendency to break on the surface: this indicates that depolymerization and physical degradation started, which can result in the formation of nanoplastics. In addition, a loss of transparency of PLA was observed, probably due to the loss of crystallinity. The researchers concluded that further time to reach complete degradation was needed, highlighting the discrepancy between the standards and the industrial composting conditions, and that studies should be made on the presence of microplastics and nanoparticles found in the final compost. Recirculation of macro-residues of bioplastic after a refining treatment could be a solution to apply in industrial composting plan in order to reach a proper degree of degradation (Ruggero F. et al., 2020). These studies are fundamental to understand how the presence of compostable packaging influence the final quality of compost.

Following a modified version of standard EN 14806 ‘‘Packaging – Preliminary evaluation of the disintegration of packaging materials under simulated composting conditions in a laboratory scale test" and EN ISO 20200 "Plastics – Determination of the degree of disintegration of plastic materials under simulated composting conditions in a laboratory–scale test", four different samples (HD-PE "100% biodegradable", two products of Mater-Bi and generic compostable bio-based material), were tested for 12 weeks. The aim of the research was to find how compostable packaging behaves in home composting processes. To simulate as much as possible conditions that represent the reality of home composting, the composition of the inoculum of synthetic waste was changed from the one described in the standards. Under laboratory conditions, mater-Bi and biobased samples disintegrated almost completely (degree of disintegration ranged from 93.5% to 98.7%), while the HD-PE that was labelled as "100% biodegradable" failed to disintegrate. On the other hand, when the same samples were put in home composting bins for 12 months, only Mater-Bi had undergone some disintegration (6.7%-14.1%). These results suggest that materials should also be tested in real conditions that differ from those in a laboratory. To determine the compost quality, phytotoxicity tests were carried out using composts resulting from the disintegration of the materials tested under laboratory conditions. Low germination values were found, due to the low pH of the tested compost (Adamcová D. et al., 2019).

Compostable bags of Mater-Bi were also tested in home and community composting facilities: static and electrochemical composters. The EC (electrochemical composter) used at the beginning of the test had to be changed with one of another kind because bags wrapping around moving parts caused clogging of the machine. In 72 days, the EC was able to degrade up to 90% of the material, which then completed its disintegration during the curing phase. Regarding the results obtained using SC (static composter), even though the process highly depends on external conditions, the disintegration degree of bioplastic was comparable to the one obtained in the EC. The researchers concluded that these specific compostable bags are suited for home composting, specifying that only a few bags were added to a large quantity of biowaste, so further studies where all organic waste is collected in bags should be applied to replicate a more realistic situation (Cafiero L. et al., 2020).

Following standard EN 14806, a lab-scale study in Tampere University of Applied Science was performed to observe the behaviour of three tested materials (S1, S2 and S3) obtained starting from a fibre base sheet coated with three different coating materials. The materials were cut in pieces of 25 mm x 25 mm, mixed with wet synthetic waste (representing the solid matrix chosen for the test) and then placed in polypropylene vessels with a 3.6 L volume each. During the test, the usual parameters were controlled and monitored. The test lasted 91 days. From visual observations it was detected that S1 and S3 degraded more easily than S2. Moisture content reached values up to 60% and, even though this didn't cause anaerobic conditions to develop, it could be the reason that led to a slow composting process, deducted also from an high C/N ratio at the end of the test. The degree of disintegration of the tested materials was compared to the one of the same fibre base sheet coated with starch. The latter degraded faster, proving that starch, acting as food and attracting the microorganisms, may enhance the disintegration process of the material. Another comparison was made observing the behaviour of a non-coated base material, which had the highest level of disintegration among all the tested samples, showing that coating layers do affect the degradability of materials. Further studies should be made to determine the boosting effect on degradation of other cellulose, hemicellulose or plant-based coating substances (Nguyen B., 2020).

In industrial facilities treating compostable materials, it can happen that an anaerobic process precedes the composting process. In the case of pure cellulose acetate, for example, it was demonstrated that a significant disintegration could be achieved and that the main contribution was related to the anaerobic digestion. In addition, microplastics were found in the liquid phase of the digestate, but their possible environmental impact was not considered in this study (Gadaleta G. et al., 2022).

Other than lab-scale trials, pilot-scale tests are widely used because they emulate a situation that's more similar to what happens in real scale plants. A Japanese study on disintegration of PLA film-laminated paper plates following ISO 16929 and ISO 14855 is an example of pilot-scale testing. The reactors used have a volume of 180 L each, with an aeration system and other auxiliary equipment to ensure optimal conditions, operating for a maximum of 80 days. At the end of the experiment, about 94.8% of the PLA decomposed and a germination test was done, which showed that the compost-treated product with the PLA sample did not induce harmful effect on plants, demonstrating its suitability as a good compost product (Kawashima N. et al., 2021).

In the Soil Science laboratory of Tampere University of Applied Science, a pilot-scale study on disintegration was implemented following standard EN 14045 using composting bins, to study the disintegration of a packaging material (not specified). Here biowaste from different sources was used as inoculum for the test; it was observed that it was containing also other materials, such as plastic and metal packaging, that were removed from the most part during preliminary operations before the lab test. Then C/N ratio, moisture content, pH and volatile solids were analysed to check if they complied with the requirements given by the standard procedure. The tested material was cut in 10 cm x 10 cm squares and put inside some nets together with a specific quantity of inoculum, and subsequently the nets were buried in the middle of the bins with the remaining part of the biowaste. The test lasted 12 weeks during which the different parameters affecting compost were regularly checked and the content of the containers was mixed following a specific schedule. The tested material started to show the first signs of disintegration after 1 month from the start of the test ad by the end of the test the samples were significantly reduced in sized. The calculation of disintegration degree proved that the packaging material almost totally disintegrated, fulfilling the requirements of standard EN 13432. It's important to notice that it was not easy to replicate the test following perfectly all the instructions given by the standard, also because they were not always clear and difficult to interpret, so the test may present some errors that can reflect on the final evaluation (Nguyen T., 2020).

Commercial PLA degradation degree was also observed in a comparative study using both lab-scale and pilot-scale testing methodology, following respectively the usual standards ISO 20200 and ISO 16929. At the end of both tests the PLA samples had a disintegration degree of 100%. Nevertheless, the sample under pilot-scale conditions degraded faster than the one observed in the lab-scale test (Intaraksa et al., 2013).

2.4.2 lab-scale vs plant-scale disintegration test

Lab-scale disintegration tests, like ISO 20200, are the most diffused to assess the disintegration degree of compostable packaging because they are easier to perform compared to pilot-scale tests, such as the one described by the standard ISO 16929, and full-scale tests (there is no standard that regulates them). However, lab-scale tests present some criticalities that can lead to an incorrect evaluation of the actual capacity of a material to degrade. First of all, these standard procedures set optimal conditions that don't represent the real situation of a treatment plant; the suggested temperature and duration of the process are much greater than those of real full-scale plants, where the compostable materials are then supposed to be treated. This discrepancy can lead to a disintegration that meets the requirements set by the standard in the controlled lab environment, but not in treatment waste facilities. Other than this, the laboratory standard procedure does not consider all the possible variables that can change during the process inside a treatment plant, whose dynamic is a much more complex mechanism and cannot be reproduced in small-scale reactors. Another problem is represented by the fact that composting plants have to deal with treating bioplastics and biodegradable waste from separate collection at the same time: biowaste is heterogeneous and has a high biodegradability, while certain biopolymers have a low biodegradability rate. Therefore, certain bioplastic materials labelled as compostable because they fulfil the requirements under standard method testing conditions, may eventually not biodegrade under the expected treatment conditions nor under uncontrolled natural conditions, when improperly disposed of.

To reach a solution further research should focus on studying how to modify the standard procedures for lab-scale testing to better emulate real-life situations. Optimal conditions (e.g. high temperature and long period of time) suggested in the standards should be changed to move towards less than favourable conditions. Parameters should vary over time, as well microorganisms responsible for biodegradation since they are susceptible to environmental changes. It should also be noted that the disintegration degree calculated using a 2mm sieve is not sufficient when dealing with materials destined to be treated in a composting plant. Bioplastic microparticles that can be found in the final compost enter agricultural soils and may have some negative effects. In order to avoid this, studies on their possible toxicity must be done (Folino et al., 2023).

Due to the issues just explained above, a lot of industrial composting plants in different countries don't accept biodegradable plastic products, because of their insufficient decomposition at the end of the treatment under normal operating conditions. A test was conducted to compare the behaviours of bioplastics under lab-scale and on-field conditions. The material tested were a PLA-based biodegradable plastic bland used for rigid packaging, and a PBS-based biodegradable plastic bland developed for soft packaging. As standard ISO suggests, for the lab-scale test a synthetic waste was prepared as the composting medium. Regarding the full-scale test, the samples were taken to the Bützberg Biogas and Composting Plant located in Tangstedt, Germany. The bioplastic samples were put inside some bags and cages to make the visual inspection and other analysis easier. The tested materials entered the plant at the start of the composting phase, after pre-treatment and anaerobic digestion of the biowaste, and stayed inside the plant for a total of 3 weeks (which is the duration of the composting stage in this specific plant). At the end of the lab-scale test, the PBS-based material fulfilled the disintegration requirements, while the PLA-based material did not. The resulting degradation of the samples tested inside the plant was much lower compared to the results of the lab-scale process taken during the fourth week. Even though some signals of degradation were observed (e.g. change in colour and brittleness), the analysed polymers have a slow degradation rate, and because of this they are not suitable to be treated in this facility. As already demonstrated, the composting media used in the laboratory experiment is homogeneous, has smaller particles and higher organic content, and this enhance the sample-medium contact surface and the biological activity. Moreover, the material inside the lab-scale reactors is mixed more frequently in respect to the composting medium inside the treatment plant (Chong ZK. Et al., 2022).

On this matter, a research project commissioned and funded by the Dutch Ministry of Economic Affairs and Climate Policy has been carried out by Wageningen Food & Biobased Research. Nine different types of compostable plastics were selected for the study: compostable bags coming from two different producers, PLA plant pots, starch and biodegradable polyester pots, used tea bags, coffee pads and coffee capsules, and finally fruit labels. The objective of the study is to evaluate the degree of disintegration of the selected products under plant conditions and timeframe. The materials were mixed with separately collected organic waste and put inside some nets. The chosen plant operated using composting tunnels for 11 days, regularly aerating and adding water to the waste. After the first cycle, the PLA plant pots were not found in both the replicates, while all the other products were still visible, even though they appeared to have a lower mechanical strength. The samples were recirculated inside the plant to undergo a second round of treatment. After this, the disintegration degree of the materials was evaluated. Only the tea bags and fruit labels were completely degraded, while a substantial amount of the other products was recovered after the sieving procedure. This means that some of the tested products need more time to properly disintegrate in respect to the resident time of this treatment plant. A solution could be to re-circulate the residue fractions after sieving more than one time. Coffee capsules are the product that presents an higher risk of visual contamination: due to their bright colours, fragments will be visible in the final compost material. It's important to say that the results of this research present some errors, as some material was lost when some of the nets broke, and some samples were not perfectly cleaned from impurities before being weighted (Van Der Zee & Molenveld, 2020).

Finally, a complementary research after a lab disintegration test was done in order to assess the actual behaviour of two thermoplastic cellulose acetate-based bioplastics, one in pure and one in composite form, inside a treatment plant. The plant selected for the experimentation is the Biogas-und Kompostwerk Bützberg, located in Hamburg, which uses a combined anaerobic and composting treatment. The samples were cut in 10 cm x 10 cm pieces, put in nets with 1kg of waste and fed to the plant. Three different scenarios were studied: degradation after dry anaerobic treatment only, degradation after composting (aerobic treatment) only and degradation after a combination of the two. The results of the test showed that for both samples:

- The disintegration degree after anaerobic digestion was between a range of 37-50%
- The disintegration degree after composting was under 20%
- The disintegration degree after the combined anaerobic-composting treatment reached values between 40-58%

It is clear that the main degradation of the materials occurred during the anaerobic treatment, and composting did not contribute much to their disintegration. Another fundamental finding was that the disintegration degree of the samples under plant-scale conditions was lower than the one calculated after the lab-scale test on the same products, in which the final disintegration degree was estimated to be between 55% and 74%. Thus, it is not prudent to extend the results obtained at the end of a lab test to the full-scale situation (Gadaleta et al., 2023)

CHAPTER 3: MATERIALS AND METHODS

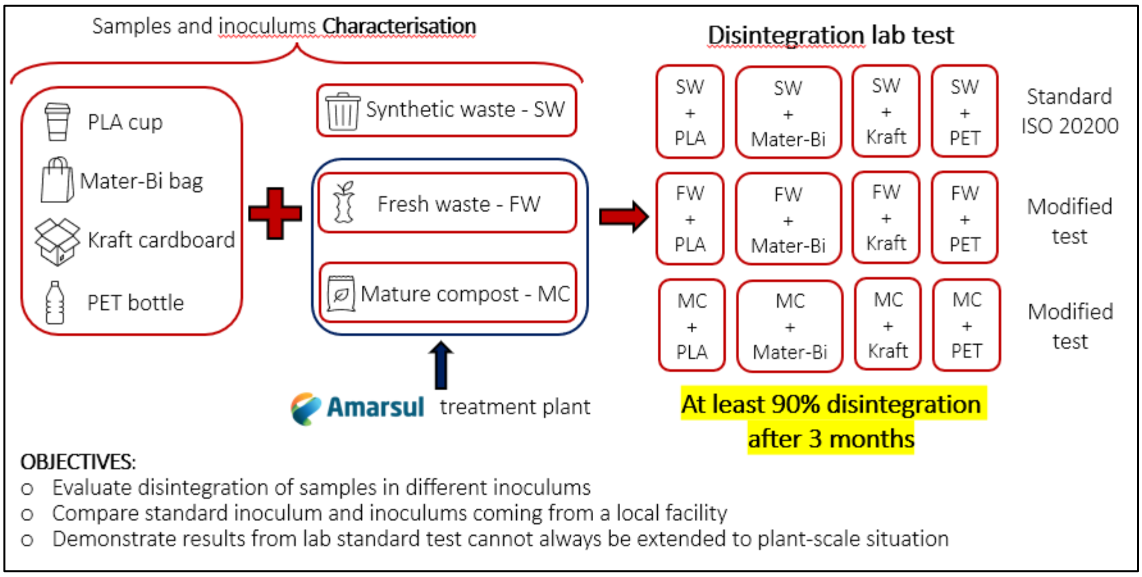

Figure 3.1: summary scheme of the research

The figure above shows a summary scheme of the research, from the objectives of the study to a graphical representation of the lab test.

3.1 MATERIALS

3.1.1 Tested samples

Three different materials were chosen to be tested: Mater-Bi compostable bags (figure 3.1), PLA compostable cups (figure 3.2), and kraft cardboard.

Figure 3.2: Mater-Bi compostable bags

Figure 3.3: PLA compostable cups

Mater-Bi is a bioplastic obtained by PBAT, starch and additives, produced by Novamont, an Italian company and international leader in the bioplastics sector. Mater-Bi film is used to manufacture compostable plastic bags for different purposes, such as the one selected for this test. The label on the bags (figure 3.3) shows the "OK compost HOME" certification by TÜV AUSTRIA, guaranteeing the complete composting of the product also in home composting devices.

Figure 3.4: Mater-Bi compostable bags label

PLA cups are compostable, usually made from starch, odourless and resistant, but not suitable for hot drinks because of their low resistance to high temperatures. Since single-use products made from conventional plastic have been banned from the market, it is expected that this kind of cups will be more and more present in the waste flux. The ones studied in this thesis are made by Safira Packaging Industry and Trade Inc., a Turkish company manufacturing disposable plastic products and selling them worldwide. Their compostability is also declared on the bottom of the cup (figure 3.4).

Figure 3.5: PLA cups label

These two materials were chosen for the test because they are still not accepted by composting plants in Portugal, as there are some uncertainties on the behaviour they would have in the existing facilities. For this reason, studies are being carried out.

Simple kraft cardboard from boxes was also selected because some previous research carried out by the university on PLA/Kraft cups, certified as compostable, showed that their disintegration degree was not sufficient to pass the disintegration test described by Standard ISO 20200. To better understand if this problem was due to kraft behaviour during composting processes, the material will be investigated alone and not aggregated with PLA.

In addition, conventional PET was introduced as a negative control for the experiment. The PET selected comes from 6L water bottles commonly found in Portuguese supermarkets (figure 3.5).

Figure 3.6: PET water bottle

3.1.2 Synthetic waste

The inoculum described by the Standard is a synthetic waste produced mixing different ingredients that are not dangerous and are easily found on the market. Table 3.1 shows the composition of this inoculum.

Table 3.1: Synthetic waste composition (Standard ISO 20200:2015)

Material	**Dry mass (%)**
Sawdust	40
Rabbit-feed	30
Ripe compost	10
Corn starch	10
Saccharose	5
Corn seed oil	4
Urea	1
TOTAL	100

NOTE 1: sawdust must come from untreated wood, preferably from deciduous trees. Sawdust must be sieved with a 5 mm sieve before using it.
NOTE 2: The rabbit feed shall be a commercial product based on alfa-alfa (lucerne) and vegetable meal. If it has a different composition, it must be reported. The protein content shall be around 15% and the cellulose content approximately 20%

The ripe compost indicated in the table as one of the ingredients should be taken from a commercial aerobic composting plant, homogeneous and containing no inert materials, such as plastic, glass, stones or metals. It must be screened using a sieve with a mesh between 0.5 cm and 1 cm. It has to be a good quality compost, rich in microorganisms to ensure the composting process during the test.

The ingredients are weighed and mixed before water is added to reach 55% of humidity (Standard ISO 20200:2015).

The compost used to make the synthetic waste in this case study is a class A certified compost (figure 3.6), produced and sold by “Siro Royal”. Composed of composted pine bark, Sphagnum peat mosses, coco peat, perlite, and some fertilizers, it contains more than 70% of organic matter, and it has already been sieved reaching a particles dimension under 15 mm.

Figure 3.7: compost used to produce the synthetic waste

The rabbit food used is produced and sold by "Rações Selecção" (figure 3.7). Its composition respects the one suggested by the standard: it is based on lucerne and contains 14% of protein and 19% of cellulose.

Figure 3.8: rabbit food used to produce the synthetic waste

3.1.3 Compost and fresh waste

Compost and fresh waste that were used as inoculums for the research were given to the laboratory by Amarsul, a company that collects and manages solid waste produced in the Setúbal peninsula (Alcochete, Almada, Barreiro, Moita, Montijo, Palmela, Seixal, Sesimbra e Setúbal). Table 3.2 shows a classification followed by a brief description of the materials collected from the plant.

Table 3.2: classification and short description of materials collected to use as inoculum for the test

ABBREVIATION	DESCRIPTION
MC	Mature compost (figure 3.8). The material is produced after composting of waste coming from restaurants selective collection of organic waste around the area of Setubal treatment plant. According to the plant, this is a class I (i.e. quality class) compost.
AT	Fresh material after approximately 1 month in the composting tunnel (figure 3.9). This is a mixture of organic waste coming from restaurants selective collection in Setúbal and residential waste collection in the area of Seixal. The latter produces a compost of class IIA. The quality of the compost that is produced mixing waste coming from different selective collections is unknown.
BT	Fresh waste before composting tunnel (figure 3.10).

Before being used in the test, the materials were spread out and put to dry for a couple of days under an aspiration hood in order to make the sieving procedure easier. A 10 mm sieve was used.

Figure 3.9: Mature compost (MC)

Figure 3.10: Fresh material after approximately 1 month in the composting tunnel (AT)

Figure 3.11: Fresh waste before composting tunnel (BT)

3.1.4 Reactors

The reactors are small polypropylene boxes, that can withstand the temperature range set by Standard ISO 20200 without deformations. Each box has a capacity of 7.7 L and dimensions of 36 x 19.5 x 13.5 cm (figure 3.11). A hole of 0.5 cm diameter was made on the two short sides of all the boxes to allow air exchange (figure 3.12). Each reactor is also equipped with a lid to allow hermetic closure (figure 3.13). A total of 45 reactors was prepared for the test, and to distinguish them an identification number was written on the lid and on every side of the containers.

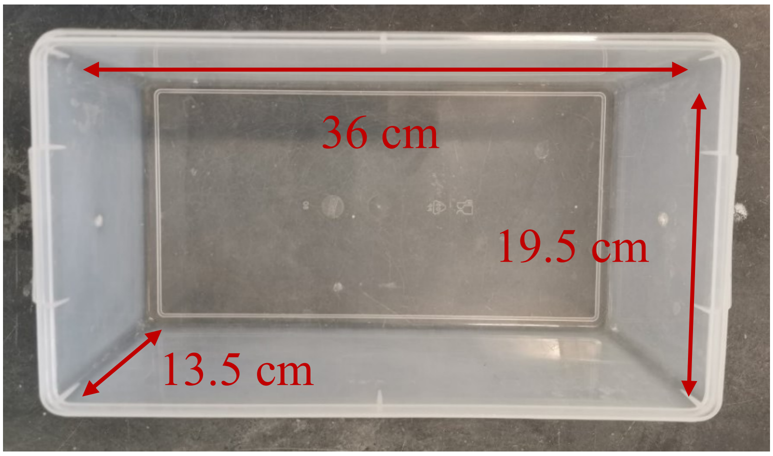

Figure 3.12: photo of the reactor (from the top)

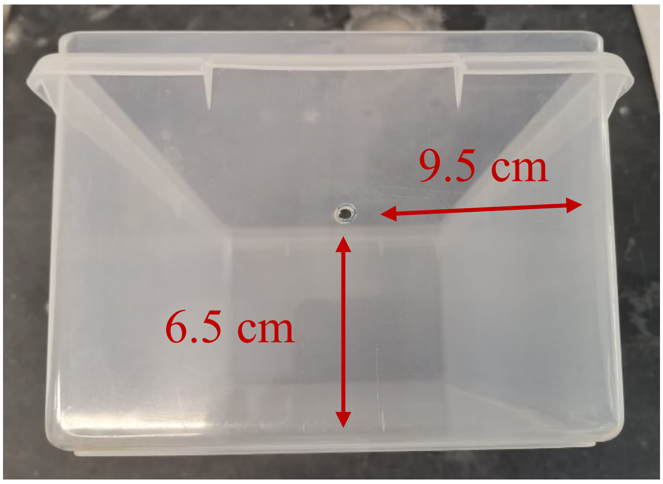

Figure 3.13: photo of the reactor (from the side)

Figure 3.14: lid of the reactor with identification number

3.2 METHODS AND TECHNIQUES

3.2.1 Self-heating test

This test follows the procedure described in "*Determination of Aerobic biological activity – part 2: self-heating test for compost (2008), CEN/TC 223*". Self-heating test has the objective to determine the aerobic biological activity of a compost. if the biological activity is null, then the compost is said to be stabilized. In this study, the self-heating test will be performed on MC (Mature compost), AT (Fresh material after approximately 1 month in the composting tunnel) and BT (Fresh waste before composting tunnel). The results of this test also help to select which material between AT and BT will be more suitable to be applied as a "fresh waste" inoculum in the disintegration test.

First thing, the materials are sieved with a 12.5 mm sieve, then the water content is calculated, and water is added to reach the standard value of 35% of humidity. If the initial moisture of the sample is too high, the compost is left to dry. After this, the samples are put inside Dewar vessels and closed with aluminium foil to avoid heat dispersion. The temperature inside the vessels is monitored by maximum thermometers and temperature probes (figure 3.14). The test lasts for approximately 10 days, during which a maximum temperature will be reached (usually after 2-5 days), and thanks to this value the stability of the material is estimated.

Figure 3.15: Dewar vessels ready for the self-heating test

Anticipating the results of the self-heating test that will be presented in the next chapter, the material that will be selected as fresh waste inoculum (FW), alongside mature compost (MC) and synthetic waste (SW), is AT.

3.2.2 Humidity

Water content for each inoculum is determined following the Standard EN 13040:2007 "*Soil improvers and growing media - Sample preparation for chemical and* *physical tests, determination of dry matter content, moisture content and laboratory compacted bulk density*". A significant quantity of inoculum is weighed and then put inside the oven (figure 3.15) to dry at a constant temperature of 105 ± 2°C till constant mass is reached, meaning that all the water has evaporated. After this, the sample is weighed again, and the humidity is calculated using the following equation:

$$H(\%) = \frac{WM - DM}{WM} \times 100 \tag{3.1}$$

Where:

1) WM (wet matter) = initial weight of the sample
2) DM (dry matter) = final weight of the sample, after water evaporation

Figure 3.16: oven used to dry the samples

3.2.3 Volatile solids

Volatile solids for each inoculum are measured following the procedure described in Standard EN 13039:2011 "*Soil improvers and growing media - Determination of organic matter content and ash*". Usually, the sample is grinded at 0.25 mm, but in this case a 1 mm mesh was used, which is also accepted by the standard. The laboratory device shown in figure 3.16 is used to shred mature compost and synthetic waste. Due to its bigger particles size, fresh waste is grinded using a more complex machine (figure 3.17).

Figure 3.17: grinder used for MC and SW

Figure 3.18: grinder used for FW

After that, a quantity between 0.5 and 5 g of sample is weighed. First it is dried at 105 °C in the oven and then incinerated at 550 ± 25 °C in the muffle (figure 3.20) till constant mass. At the end the weight of the sample (figure 3.21) is taken again, and the content of volatile solids is calculated using the following equation:

$$VS(\%) = \frac{W_i - W_f}{W_i} \times 100 \qquad (3.2)$$

Where:

- W_i (initial weight) = weight of the sample before drying at 105 C°
- W_f (final weight) = weight of the sample after incineration at 550 C°

Figure 3.19: muffle

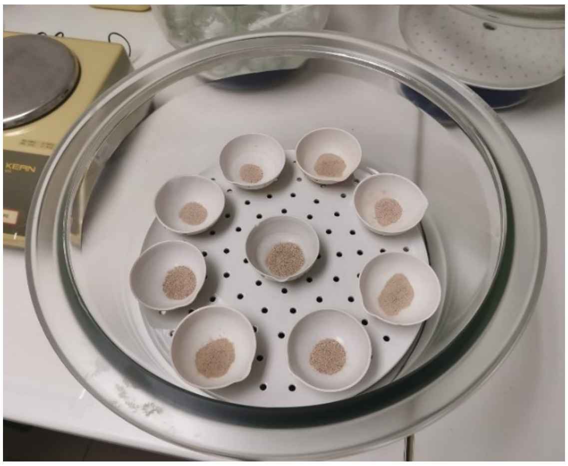

Figure 3.20: samples after incineration

3.2.4 pH

Standard procedure EN 13037:2011 "*Soil improvers and growing media - Determination of pH*" is applied to measure the pH of the different inoculums. First 5 g of material is mixed with 50 mL of distilled water for at least 2 hours. Then it's possible to measure the pH of the solution thanks to a specific electrode.

3.2.5 Electrical conductivity

Standard procedure 13038:2011 "*Soil improvers and growing media - Determination of electrical conductivity*" is applied to measure the electrical conductivity of the different inoculums. First 5 g of material is mixed with 50 mL of distilled water for at least 2 hours. Then it's possible to measure the E.C. of the solution thanks to a specific electrode. It's important to correct the result at the end of the measurement multiplying it by the appropriate coefficient depending on the temperature of the solution.

3.2.6 Heavy metals

Standard procedure EN 13346:2000 "*Characterization of sludges - Determination of trace elements and phosphorus - Aqua regia extraction methods"* is followed to measure the content of heavy metals in the different inoculums. A quantity of 0.5 g of dried and grinded (0.25 mm) sample is mixed with 9 mL of hydrochloric acid and 3 mL of nitric acid. The vessels containing the solution are put in a microwave rotor where the digestion starts. At the end of the cycle, the vessels are left to cool down till ambient temperature to reduce the internal pressure. Next step is filtration of the solution for each vessel, at the end of which the filtrate is collected in other containers and distilled water is added till a total volume of 50 mL is reached. The sample is analysed using ICP-AES (Inductively Coupled Plasma Atomic Emission Spectroscopy – figure 3.22), a heavy metal analyser equipment that identifies and quantifies the elements present in the sample. The sample is turned into an aerosol and then it is injected into the plasma. Thanks to the high temperatures reached, the sample is broken down into atoms that are excited by the same heat and emit wavelengths different for each of the present elements. These wavelengths are separated and captured to be read by a series of detectors (Spectro, n.d.).

Figure 3.21: equipment for "Inductively Coupled Plasma Atomic Emission Spectroscopy"

3.2.7 Standard ISO 20200 and adjustments

The procedure to assess the degradation degree of the materials selected for the test follows the rules set by Standard ISO 20200 of 2015 "*Plastics — Determination of the degree of disintegration of plastic materials under simulated composting conditions in a laboratory-scale test*". Some changes on the standardized method have also been made in order to reach the objective of this thesis. The main one is the introduction of other two inoculums, apart from the one already suggested by the standard (Synthetic Waste), that better represent the waste present in a real composting plant: fresh waste and mature compost. The optimal humidity suggested for the synthetic inoculum is 55%, but this value was found to be not appropriate in the case of MC and FW. Probably due to the presence of inert materials in both inoculums, and the fine granulometry of MC (the plant sieved it using a sieve with a mesh lower than 10 mm as requested by the laboratory), MC and FW have a lower absorption capacity than SW. This is why using 55% of humidity in the preparation of these two inoculums resulted in the production of highly liquid matrixes. New maximum optimal humidity values were experimentally calculated: the optimal water content for MC and FW are respectively 42.76% and 42%.

After preparation of the inoculums, it was observed that the quantity of FW collected from the plant was not enough to fill all 15 reactors. Accordingly, it was decided to exclude from the test reactor 16, that would have contained FW only, and reactor 28, that would have contained FW and conventional PET.

The samples, which in this case are compostable bags, compostable PLA cups, kraft cardboard and conventional PET (negative control), have a thickness lower than 5 mm, so they are cut into squares of approximately 2.5 cm x 2.5 cm (figures 3.23, 3.24 and 3.25), as the standard wants.

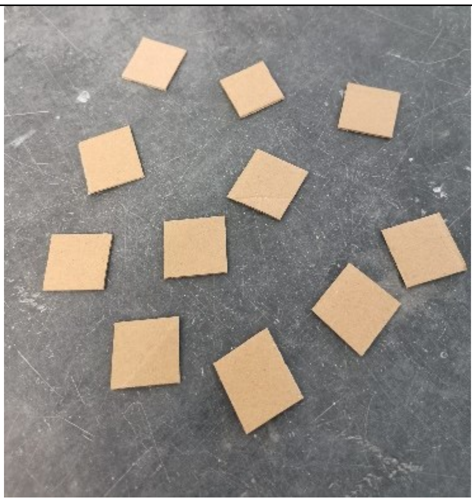

Figure 3.22: Kraft samples

Figure 3.23: Mater-Bi bags samples

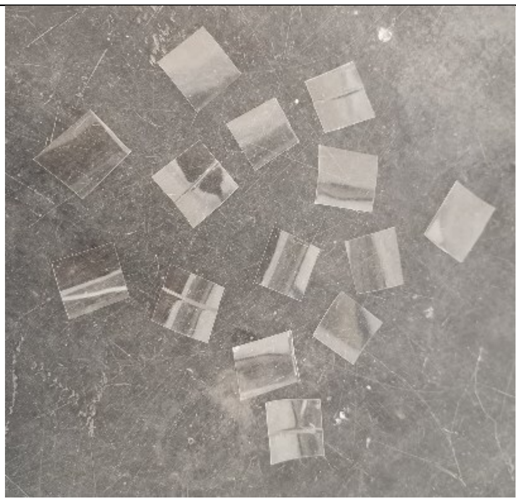

Figure 3.24: PLA cups samples

Then, each reactor is filled with 1 kg of inoculum and a specific quantity of sample between 5 g and 20 g, based on the volume of the sample and the one of the inoculum. In the table below the approximate quantity of investigated material for each type of inoculum is reported.

Table 3.3: quantity of sample for 1 kg of each type of inoculum

Sample / Inoculum	Compostable bags	PLA cups	Kraft cardboard	Conventional PET
Synthetic waste	10 g	20 g	20 g	20 g
Fresh waste	5 g	20 g	20 g	20 g
Mature compost	5 g	20 g	20 g	20 g

In the case of this study, 3 replicates for each combination between the different samples and inoculums are prepared. In addition, it was decided to include in the test also 3 replicates for each inoculum with no biodegradable samples inside, acting as controls. Summing up, a total of 45 boxes was initially prepared, from which reactors number 16 and number 28 were excluded for the reason already explained. Finally, the experimentation will deal with a total of 43 boxes. Tables 3.4 and 3.5 shows the tare (measured without lid on), the types of inoculum and sample contained, the weight of the sample and the total initial weight (Wi = tare + W inoculum + W sample) for each reactor at day 0 of the test period.

Table 3.4: measurements taken at day 0 of the test period (boxes from 1 to 22)

BOX n°	Tare of reactor (g)	Inoculum and sample (type)	Sample (g)	Wi (g)
1	309.42	SW	-	1309.2
2	312.57	SW	-	1312.42
3	312.23	SW	-	1311.96
4	311.83	SW + MB bag	10.14	1320.35
5	306.12	SW + MB bag	10.14	1314.27
6	309.41	SW + MB bag	10.14	1318.45
7	320.76	SW + PLA cup	20.19	1339.66
8	306.39	SW + PLA cup	20.21	1325.81
9	308.28	SW + PLA cup	20.25	1327.28
10	311.17	SW + Kraft	20.25	1349.71
11	313.16	SW + Kraft	20.11	1351.71
12	306.92	SW + Kraft	20.17	1345.81
13	308.57	SW + PET	20.20	1327.95
14	307.03	SW + PET	20.27	1325.88
15	306.66	SW + PET	20.29	1326.66
16	306.22	FW	-	-
17	308.29	FW	-	1308.20
18	304.12	FW	-	1303.79
19	316.46	FW + MB bag	5.07	1329.32
20	307.38	FW + MB bag	5.07	1310.23
21	314.87	FW + MB bag	5.07	1317.43
22	304.10	FW + PLA cup	20.18	1322.78

Table 3.5: measurements taken at day 0 of the test period (boxes from 23 to 45)

BOX n°	Tare of reactor (g)	Inoculum and sample (type)	Sample (g)	Wi (g)
23	302.91	FW + PLA cup	20.14	1321.95
24	302.79	FW + PLA cup	20.26	3121.83
25	314.87	FW + Kraft	20.30	1356.80
26	307.96	FW + Kraft	20.19	1343.60
27	308.12	FW + Kraft	20.25	1345.10
28	308.85	FW + PET	-	-
29	318.44	FW + PET	20.34	1337.88
30	305.82	FW + PET	20.24	1324.51
31	308.47	MC	-	1308.13
32	309.04	MC	-	1308.86
33	308.92	MC	-	1308.99
34	305.94	MC + MB bag	5.07	1310.31
35	315.46	MC + MB bag	5.07	1319.10
36	305.23	MC + MB bag	5.07	1308.79
37	315.37	MC + PLA cup	20.14	1333.20
38	305.16	MC + PLA cup	20.22	1325.46
39	305.17	MC + PLA cup	20.18	1324.40
40	305.73	MC + Kraft	20.26	1344.43
41	305.30	MC + Kraft	20.16	1347.70
42	308.13	MC + Kraft	20.16	1346.76
43	308.57	MC + PET	20.37	1327.58
44	309.55	MC + PET	20.30	1329.03
45	308.72	MC + PET	20.34	1328.68

After all the reactors are prepared, they are closed with the lids and put inside an aerated oven (figure 3.26) at a constant temperature of 58°C. This phase can last from a minimum of 45 days to a maximum of 90 days, and it is called the thermophilic incubation period. Standard ISO 20200 also introduces the possibility to extend the period of the test (mesophilic phase) if necessary, but in the case of this study the experiment will stop at the thermophilic phase.

Figure 3.25: reactors stored at 58°C in the oven

The disposition of the reactors is randomly changed every time they are taken out and put back inside the oven, paying particular attention to always switch the three boxes on the front with different ones.

Water is added, the composting matter is mixed, and the weight of the reactors is recorded following the schedule described in the table below. All the tables recording the mass of the reactors during the test period are reported in Annex I.

Table 3.6: disintegration test schedule (Standard ISO 20200:2015)

Time from start (days)	**Operation**
0	Record initial mass of reactor
1, 2, 3, 4, 7, 9, 11, 14	Weigh reactor and add water to restore the initial mass, if needed. Mix the composting matter
8, 10, 16, 18, 21, 23, 25, 28	Weigh reactor and add water to restore the initial mass, if needed. Do not mix the composting matter
30, 45	Weigh reactor and add water to restore the mass to 80% of initial mass, if needed. Mix the composting matter
From 30 till 60, twice a week	Weigh reactor and add water to restore the mass to 80% of initial mass, if needed. Do not mix the composting matter
From 60 onwards, twice a week	Weigh reactor and add water to restore the mass to 70% of initial mass, if needed. Do not mix the composting matter

At the end of the composting process, all the lids are removed from the reactors and the boxes are put inside the oven to allow the water to evaporate and make the following sieving procedure easier. The material of each container is sieved using 10 mm, 5 mm and 2 mm sieves, collecting the pieces of samples from each fraction if they are visible. Then, the collected fragments are cleaned as much as possible from compost residues and dried. The material that passes through the 2 mm sieve is considered to be disintegrated. The degree of disintegration is finally calculated using the following expression:

$$D = \frac{m_i - m_r}{m_i} \times 100$$

Where: *(3.3)*

- m_i is the initial dry mass of the sample
- m_r is the residual dry mass of the sample (i.e. above 2 mm sieve).

Some requirements must be checked for the test to be valid:

1) The degree of disintegration of the three replicates can differ for no more than 20%

2) Factor R (decrease in total volatile - solids) shall be greater than or equal to 30%

$$R = \frac{[m_i \times DM_i \times VS_i] - [m_f \times DM_f \times VS_f]}{[m_i \times DM_i \times VS_i]} \times 100$$

Where: *(3.4)*

- m_i = initial mass of wet synthetic waste in the reactor
- DM_i = initial dry mass of synthetic waste, expressed as a percentage divided by 100
- VS_i = initial volatile solids content of the synthetic waste, expressed as a percentage divided by 100
- m_f = final mass of the compost
- DM_f = final dry mass of the compost, expressed as a percentage divided by 100
- VS_f = final volatile solids content of the compost, expressed as a percentage divided by 100.

CHAPTER 4: RESULTS AND DISCUSSION

4.1 PHYSICAL AND CHEMICAL CHARACTERIZATION OF SAMPLES AND INOCULUMS (before test)

Physio-chemical characterisation of the materials involved in the test is the first step of every research project. These analyses are fundamental to identify physio-chemical properties and anticipate the materials behaviour during the process under study. Characterisation is an instrument that helps researchers to understand and explain the test final results.

4.1.1 Humidity, pH, electrical conductivity and volatile solids

Tables 4.1 and 4.2 show the characterization of samples and inoculums before the composting test, in terms of humidity (%), pH, electrical conductivity (µS/cm) and volatile solids (%).

Table 4.1: Humidity, pH, electrical conductivity and volatile solids of the samples

SAMPLE	H (%)	pH	E.C. (µS/cm)	VS (%)
Mater-Bi bag	1.41	6.93	11.79	100.00
PLA cup	0.77	6.55	3.40	99.91
Kraft cardboard	7.15	6.64	970.06	86.16
PET bottle	1.02	6.48	12.44	100.00

Looking at table 4.1, kraft carboard stand out from the other samples: it is characterized by a higher water content and electrical conductivity, but mostly its volatile solids percentage is lower compared to the ones of the other samples, meaning that it contains less organic matter.

Table 4.2: Humidity, pH, electrical conductivity and volatile solids of the inoculums

INOCULUM	H (%)	pH	E.C. (μS/cm)	VS (%)
Synthetic Waste	55.81	6.52	871.08	93.59
Fresh Waste	46.56	6.36	3545.50	42.89 **(*)**
Mature Compost	42.28	8.21	3104.56	39.34

Humidity analysis regarding inoculums shown in table xx mirrors what was already explained in the previous chapter, that is the water content of fresh waste and mature compost is below the range to guarantee an optimal composting process. Another relevant information taken by this table is that fresh waste and mature compost contain less than a half of volatile solids compared to synthetic waste.

4.1.2 Heavy metals

The table below shows the results obtained on the heavy metals analysis of the different inoculums.

Table 4.3: Heavy metals analysis of the inoculums

Inoculum	**Cd** [mg/kg]	**Cr** [mg/kg]	**Cu** [mg/kg]	**Ni** [mg/kg]	**Pb** [mg/kg]	**Zn** [mg/kg]
Synthetic Waste	0.544	35.269	14.174	36.827	3.687	31.509
Fresh Waste	1.183	21.699	33.497	9.233	33.147	125.057
Mature compost	3.563	141.669	57.082	171.68	25.379	168.648

According to the "compost quality" system present in Portugal, synthetic waste and fresh waste correspond to a Class I compost, while mature compost present higher values of heavy metals. Because of this, mature compost does not fall into the Class I compost category. The thresholds established by the Portuguese Law Decree (No 103/2015) on Fertiliser Products are listed in table 4.4.

Table 4.4: Compost quality limits set by Portugal – Law Decree (No 103/2015) on Fertiliser Products (DRE, 2015)

Class of quality	**Cd** [mg/kg]	**Cr** [mg/kg]	**Cu** [mg/kg]	**Ni** [mg/kg]	**Pb** [mg/kg]	**Zn** [mg/kg]
Class I	0.7	100	100	50	100	200
Class II	1.5	150	200	100	150	500
Class II A	3	300	400	200	300	100
Class III	5	400	600	200	500	1500

4.2 SELF-HEATING TEST

Table 4.5 expose the maximum temperature reached by fresh waste, fresh waste after 1 month of tunnelling and mature compost during the self-heating test.

Table 4.5: maximum temperature reached during the self-heating test

MATERIAL	T (°C)
Fresh Waste before 1 month of tunnel	60
Fresh Waste after 1 month of tunnel	55
Mature Compost	20

This test was done with the objective to compare the aerobic biological activity of the different materials that were chosen as possible inoculums before the preparation of the test. Fresh Waste before (BT) and after (AT) one month of tunnel reached almost the same maximum temperature. Fresh Waste after 1 month of tunnel was selected for the experiment just because higher quantities of it were available, making it possible to fill as much reactors as possible. Mature Compost instead is more stable than the others, reaching only 20°C, which is to be expected seeing as it was subject to a maturation period.

4.3 PHYSICAL AND CHEMICAL CHARACTERIZATION (after test)

4.3.1 Humidity, volatile solids, pH and electrical conductivity

Tables 4.6 to 4.8 show the physio-chemical characterization of the content of each box after the composting test, in terms of humidity (%), pH, electrical conductivity (µS/cm) and volatile solids (%).

Table 4.6: Humidity, pH, electrical conductivity and volatile solids (Boxes from 1 to 15 – SW inoculum)

BOX n°	sample	H (%)	pH	E.C. (µS/cm)	VS (%)
1	-	57.86	7.04	1873.70	86.55
2	-	58.14	6.90	1980.00	84.43
3	-	56.89	6.99	1560.14	87.45
4	Mater-Bi bag	58.32	6.96	1973.53	86.95
5	Mater-Bi bag	44.88	6.92	2099.88	87.09
6	Mater-Bi bag	62.97	6.90	2281.60	86.18
7	PLA cup	51.39	6.91	1950.48	86.41
8	PLA cup	59.48	6.86	2588.30	85.42
9	PLA cup	56.41	7.03	2090.55	87.25
10	Kraft cardboard	62.49	7.01	2716.11	85.48
11	Kraft cardboard	60.42	6.97	2550.94	84.99
12	Kraft cardboard	58.15	6.95	2355.20	86.88
13	PET bottle	60.77	6.98	2731.26	86.33
14	PET bottle	61.10	7.01	2662.08	86.54
15	PET bottle	58.01	7.03	2241.60	85.91

Table 4.7: Humidity, pH, electrical conductivity and volatile solids (Boxes from 16 to 30 – FW inoculum)

BOX n°	sample	H (%)	pH	E.C. (µS/cm)	VS (%)
16	-	-	-	-	-
17	-	0.72	8.83	2426.38	31.69
18	-	3.36	6.13	4747.92	48.12
19	Mater-Bi bag	0.53	8.95	2364.74	41.82
20	Mater-Bi bag	1.51	6.30	3363.03	50.20
21	Mater-Bi bag	15.10	8.88	2285.00	33.45
22	PLA cup	6.71	5.82	4784.40	51.02
23	PLA cup	5.55	5.79	5121.00	50.58
24	PLA cup	0.63	5.89	4498.72	50.08
25	Kraft cardboard	5.56	6.27	3230.95	49.55
26	Kraft cardboard	26.57	8.73	2257.00	31.68
27	Kraft cardboard	18.09	8.75	3416.68	29.57
28	-	-	-	-	-
29	PET bottle	1.02	6.20	4412.94	44.23
30	PET bottle	18.37	8.79	2760.28	33.90

Table 4.8: Humidity, pH, electrical conductivity and volatile solids (Boxes from 31 to 45 – MC inoculum)

BOX n°	sample	H (%)	pH	E.C. (μS/cm)	VS (%)
31	-	1.24	8.21	5155.70	37.75
32	-	0.60	8.23	5775.18	39.30
33	-	1.13	8.28	4842.42	35.20
34	Mater-Bi bag	3.09	8.32	5359.92	42.18
35	Mater-Bi bag	1.48	8.32	5691.33	39.26
36	Mater-Bi bag	0.51	8.38	4941.37	36.28
37	PLA cup	1.88	8.58	4315.5	39.95
38	PLA cup	0.90	8.51	5410.43	38.93
39	PLA cup	2.72	8.56	5008.35	39.97
40	Kraft cardboard	10.34	8.53	3836.56	43.74
41	Kraft cardboard	0.28	8.47	4393.00	38.83
42	Kraft cardboard	1.58	8.40	5423.30	39.10
43	PET bottle	2.17	8.37	5519.80	37.03
44	PET bottle	0.79	8.34	5608.60	35.98
45	PET bottle	1.16	8.42	5398.76	43.24

Table 4.9 shown below, presents the mean values of humidity (%), pH, electrical conductivity (µS/cm) and volatile solids (%), putting together the results obtained for the three replicates of each combination of inoculum and sample.

Table 4.9: Mean values of humidity, pH, electrical conductivity and volatile solids

inoculum	sample	H (%)	pH	E.C. (µS/cm)	VS (%)
SW	-	57.63	6.98	1804.61	86.14
SW	Mater-Bi bag	55.39	6.93	2118.34	86.74
SW	PLA cup	55.76	6.93	2209.78	86.36
SW	Kraft cardboard	60.35	6.98	2540.75	85.78
SW	PET bottle	59.96	7.01	2544.98	86.26
FW	-	2.04	7.48	3587.15	39.90
FW	Mater-Bi bag	5.71	8.04	2670.92	41.82
FW	PLA cup	4.29	5.83	4801.37	50.56
FW	Kraft cardboard	16.74	7.92	2968.21	36.93
FW	PET bottle	9.69	7.50	3586.61	39.06
MC	-	0.99	8.24	5257.77	37.41
MC	Mater-Bi bag	1.70	8.34	5330.87	39.24
MC	PLA cup	1.84	8.55	4911.43	39.62
MC	Kraft cardboard	4.07	8.47	4550.95	40.56
MC	PET bottle	1.37	8.38	5509.05	38.75

This table is useful to assess more easily the quality of the product obtained at the end of the test. The values of pH in all of the cases fall on the interval of 6 to 9.5, which is typical for the different types of compost commercially available. However, compost with values of E.C. greater than 1500 µS/cm can cause salt stress in plants, inhibiting their growth.

4.4 DISINTEGRATION TEST RESULTS

4.4.1 Visual inspection

At the end of the test, the inoculums present inside the different reactors are visually analysed. Synthetic waste is homogeneous and characterised by a dark brown colour. Fresh waste appears heterogeneous in all the boxes but present some differences in colour: in some boxes it is dark brown, in other boxes it is light brown. This discrepancy could be due to the different percentage of humidity in the inoculum, or also to the formation of Actinomycetes present in high quantities in some of the boxes. Mature Compost looks unchanged in respect to its original appearance.

Some photos were taken to analyse the changes of the different materials during the composting process, at intervals of approximately 15 days from the beginning of the test. During the operations of day 18, 30 and 45 a small quantity of sample was taken from one box for all the different combinations of tested materials and inoculums, and then put back inside. The identification numbers of the boxes under observation are 5, 8, 11, 14, 20, 23, 26, 29, 35, 38, 41 and 44.

DAY 18

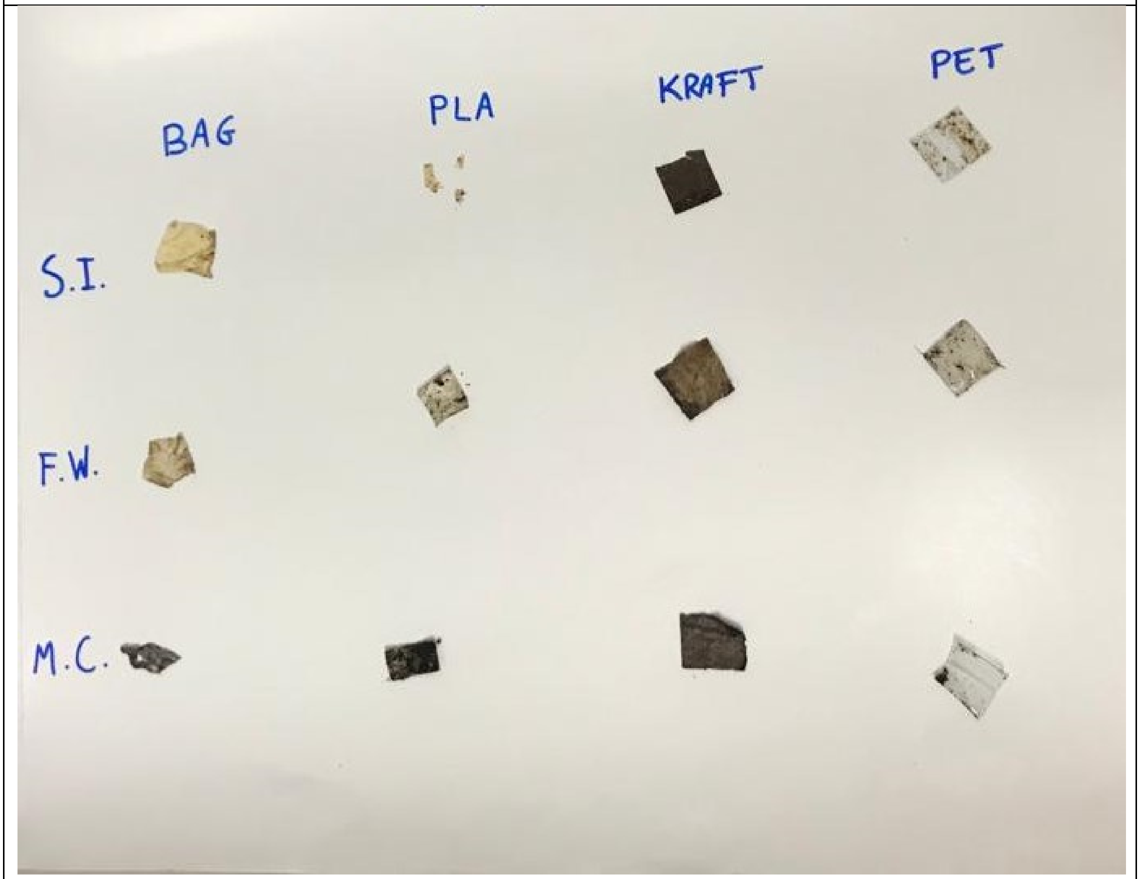

Figure 4.1: samples after 18 days from the start of the test

On day 18 (figure 4.1), all tested materials are still visible. PET doesn't present changes compared to its initial status. Kraft is softer due to the absorption of water, and its layers are separated from each other. Mater-Bi is sticky and has lost its original milky colour and, as seen in the image below, it appears to be yellow/brown. PLA has lost its transparency because of the progressive decrease in crystallinity and has started to significantly disintegrate in Synthetic Waste.

DAY 30

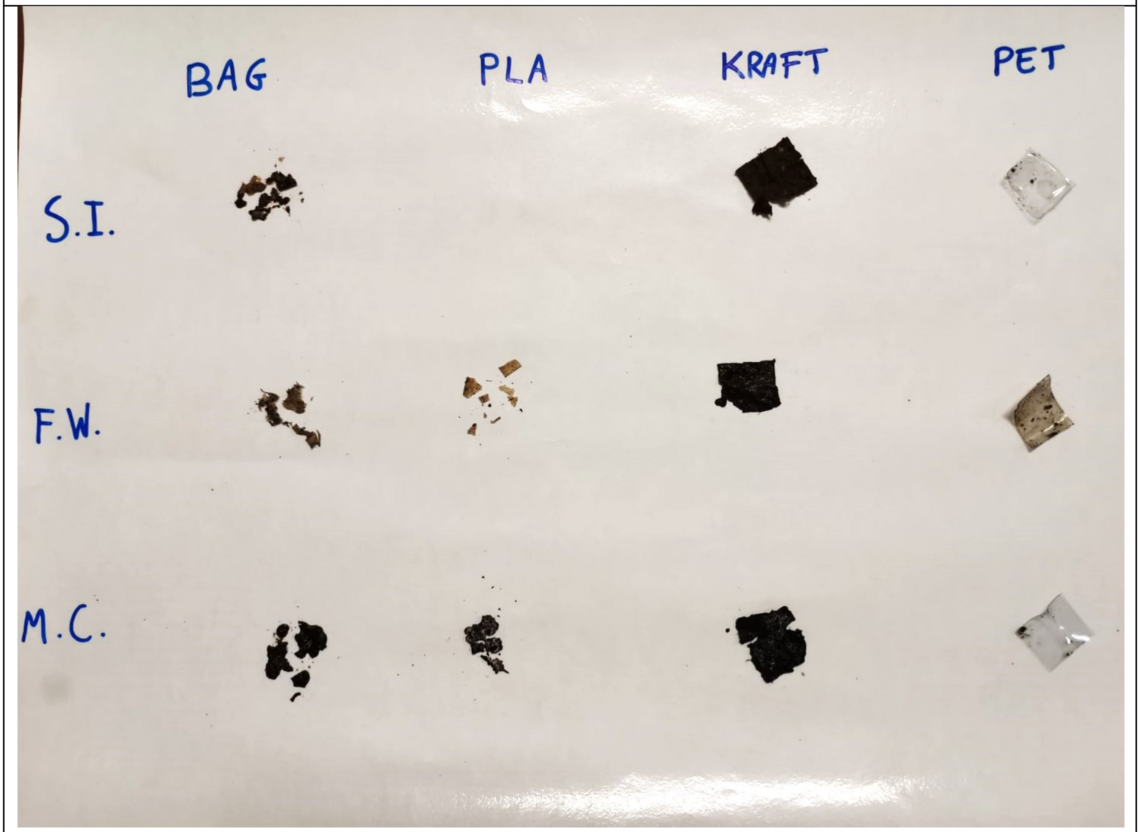

Figure 4.2: samples after 30 days from the start of the test

On day 30 (figure 4.2), PET has not changed. Kraft squares are dirty and slightly broken, but still intact. Both Mater-Bi and PLA have started to disintegrate. It was observed that no visible fragments of PLA were not found in the reactor containing Synthetic Waste.

DAY 45

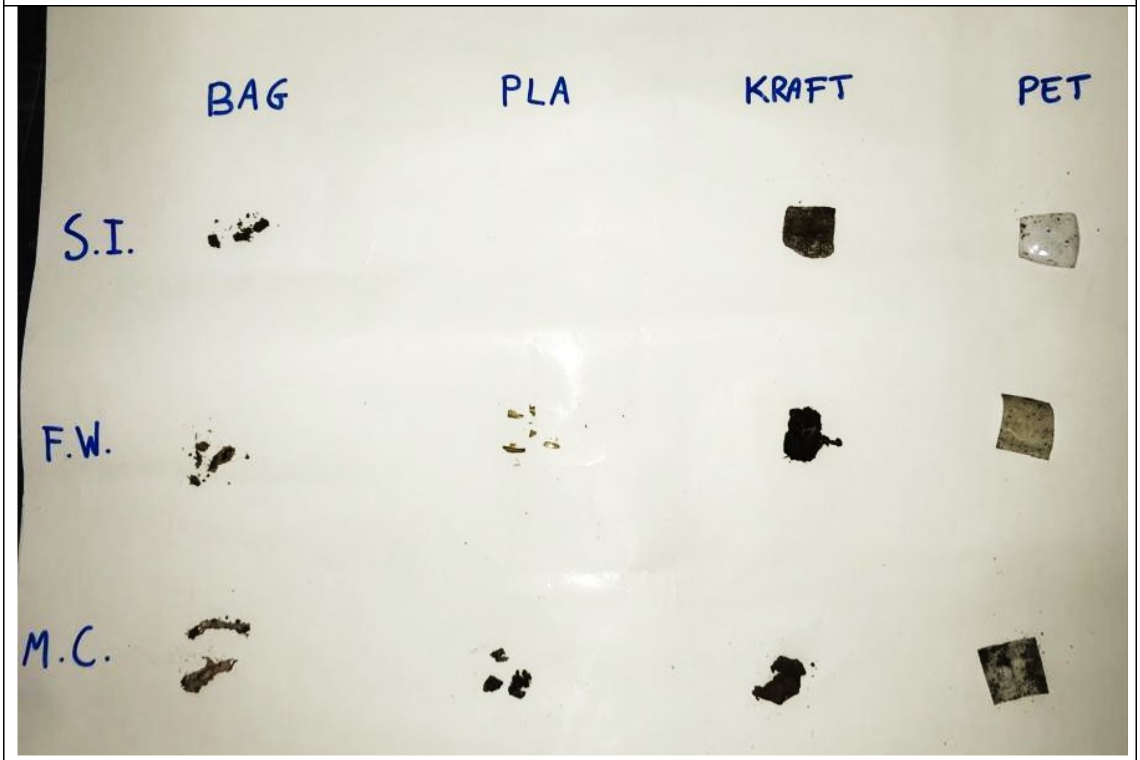

Figure 4.3: samples after 45 days from the start of the test

On day 45 (figure 4.3) PET does not present alterations, except for some deformations due to the high temperatures reached in the oven. Kraft is completely cover with inoculum, crumpled, but mostly intact. PLA and Mater-Bi fragments are getting smaller and less visible to the naked eye as a result of their increasing fragility.

4.4.2 Degree of disintegration

After sieving the content of each reactor using a 2 mm sieve, the residual fragments of sample present in the fraction above 2 mm are manually picked. Unsurprisingly, PET is the easiest material to remove, but concerning the other samples, some problems have to be dealt with. Especially in Fresh Waste and Mature compost, residual pieces of material are hard to detect since inoculum is attached to them and they do not have a distiguishable colour. In addition, they are extremely fragile, therefore if they are not treated carefully they could break in even smaller fragments and get lost again inside the inoculum. Putting together these observations, it is clear that this operation becomes particularly difficult and time consuming.

The next step suggested by Standard ISO 20200 is to wash the recovered samples to be able to measure their exact mass. Washing PET with water is a process done without any difficulties and does not take much time. Mater-Bi residues can not be washed using water otherwise they would melt, and neither can they be cleaned using a brush as they are too fragile and they will surely break. Hence, they are weighed as such. Kraft is the type of material with higher quantities of inoculum attached to it (figure 4.4). This is caused by the fact that the inoculum is accumulated inside the creases of the crumpled pieces of Kraft. The use of water is bypassed in this case too, or else Kraft would melt. As an alternative, brushes and tweezers are used to remove as much residues of inoculum as possible, taking into account that also Kraft is easy to break.

Figure 4.4: Kraft sample recovered from one of the reactors

The recovered samples are then weighed and the disintegration degrees (D) are calculated using formula 3.3 (see chapter "Materials and methods"). Tables 4.10 to 4.12 show the results of this calculation for all the reactors. Obviously D was not determined for the controls as they only contain inoculum.

Table 4.10: degree of disintegration (boxes from 1 to 15 – SW inoculum)

BOX n°	sample	sample recovered	D (%)	Mean value of D (%)
1	-	-		
2	-			
3	-			
4	Mater-Bi bag	-	100.00	100.00
5	Mater-Bi bag	-	100.00	
6	Mater-Bi bag	-	100.00	
7	PLA cup	-	100.00	100.00
8	PLA cup	-	100.00	
9	PLA cup	-	100.00	
10	Kraft cardboard	10.57	43.78	29.19
11	Kraft cardboard	14.76	20.95	
12	Kraft cardboard	14.45	22.84	
13	PET bottle	20.00	-0.03	-0.31
14	PET bottle	20.03	0.17	
15	PET bottle	20.30	-1.08	

Table 4.10 shows the results regarding the reactors containing Synthetic Waste. What is noticeable is that PLA and Mater-Bi residues were not found, resulting in a degree of disintegration equal to 100 %. Kraft presents a mean value of D of 29 %, but it's always important to remember that the value could be overestimated, due to errors in manual picking of the residual material, or underestimated, because of the presence of some

inoculum attached to the residues. As predicted, the initial quantity of PET put inside the reactors was completely removed at the end of the test.

Table 4.11: degree of disintegration (boxes from 16 to 30 – FW inoculum)

BOX n°	sample	sample recovered	D (%)	Mean value of D (%)
16	-	-		
17	-			
18	-			
19	Mater-Bi bag	3.53	29.38	56.66
20	Mater-Bi bag	2.97	40.58	
21	Mater-Bi bag	-	100.00	
22	PLA cup	-	100.00	100.00
23	PLA cup	-	100.00	
24	PLA cup	-	100.00	
25	Kraft cardboard	26.51	-40.65	-39.57
26	Kraft cardboard	33.58	-79.14	
27	Kraft cardboard	18.60	1.07	
28	-	-	-	-3.08
29	PET bottle	20.31	-0.88	
30	PET bottle	21.09	-5.27	

Table 4.11 displays the outcomes of the disintegration process that took place inside the boxes with Fresh Waste inoculum. PLA has completely disintegrated in this case as well. As for Mater-Bi, no residues were found in one of the boxes, while some fragments were detected in the other two. However, it is not certain if these residues are actually Mater-Bi bioplastic considering that Fresh Waste inoculum was already contaminated by other materials, plastics included, when it was collected from the treatment plant. Kraft is characterized by a negative degree of disintegration, which means that all the original

material was collected, but a significant amount of inoculum was still attached to it when it was weighed. This outcome was predictable since Kraft turned out to be most difficult material to manage during the cleaning procedure. PET was again entirely recovered, but with a slightly negative D due as previously explained.

Table 4.12: degree of disintegration (boxes from 31 to 45 – MC inoculum)

BOX n°	sample	sample recovered	D (%)	Mean value of D (%)
31	-	-		
32	-			
33	-			
34	Mater-Bi bag	1.47	70.59	73.39
35	Mater-Bi bag	0.88	82.40	
36	Mater-Bi bag	1.64	67.19	
37	PLA cup	-	100.00	100
38	PLA cup	-	100.00	
39	PLA cup	-	100.00	
40	Kraft cardboard	22.79	-21.16	-23.59
41	Kraft cardboard	22.01	-17.59	
42	Kraft cardboard	24.71	-32.01	
43	PET bottle	20.83	-3.31	-4.05
44	PET bottle	21.06	-4.81	
45	PET bottle	20.84	-4.02	

Finally, table 4.12 exposes the disintegration degree in case of Mature Compost. Also in this final set-up, PLA is 100% disintegrated. Regarding Mater-Bi, some discrepancies between the results in the three reactors are observed, however they are less prominent than those noted in Fresh Waste. Kraft and PET did not disintegrate, and their disintegration degree is negative once more because of cleaning issues.

PLA disintegrated completely in all the reactors, meaning its degradation degree is 100%. This result is consistent with the outcomes of a previous research conducted by Intaraksa et al. (2013) on samples with the same characteristics and following the same standard procedure. Ruggero et al. (2021) experiment resulted in a complete degradation of Mater-Bi, which is the same result obtained in this test in the case of SW inoculum. Its degree of disintegration decreased consistently in MC and even more in FW, but this could have been anticipated seeing as their biological activity is weaker and their humidity during the test was always maintained under optimal levels for composting processes. Kraft was entirely recovered from almost all the reactors, with no significative degradation. López Alvarez et al. (2009) pointed out that paper-like materials contain complex organic compounds, such as lignocellulose or fatty acids, which retard biodegradation, making Kraft a non-compostable substance.

4.4.3 Validity check of the results

Following the calculation of the various disintegration degrees, the validity of the results must be checked following Standard ISO 20200 directives. Index R, calculated using formula 3.4 (see chapter "Materials and methods"), should be higher than 30%, and the degree of disintegration of the three replicates should not differ for more than 20%.

The outcomes of the test involving the use of standardised synthetic waste is considered valid. Looking at table 4.13, R is well above 30% for every reactor, so the first requirement is met. Then, concerning the variability between replicates values of D, they are all under 20%, except for Kraft, which variability is 22.83%. Since this value is just slightly above 20%, the test can still be assumed valid.

Observing the parameters in the case of fresh waste (table 4.14), it is immediately noticeable that index R in most cases has a negative value. This is due to the fact that volatile solids during the test have not registered a significative reduction; on the contrary, the final quantity of volatile solids in some reactors is even higher than the volatile solids content measured before the beginning of the test. What happened here could be caused by the composition of the waste, by it's final humidity or by the presence of compostable material particles, that are characterized by a considerable amount of volatile solids, making them able to raise the volatile solids of the compost under analysis. In addition, the VS content measured both before and after the test may not be representative of the entire inoculum due to its excessive heterogeneity. This aspect influence also the calculation of the variation of D, which is much higher than the requested value of 20%.

The situation of the reactors containing Mature Compost (table 4.15) is different once again. The inoculum, despite presenting impurities such as glass or plastic, it is more homogeneous than Fresh Waste. In fact, the variability of the disintegration degree is under the predetermined threshold of 20%. The volatile solids reduction, and consequently index R, is still too low to consider the test valid. This could mean that Mature Compost was not active enough to be used alone as the solid matrix of a composting test.

The validity parameters just described are listed in the tables below.

Table 4.13: parameters to check the validity of the test (boxes from 1 to 15)

BOX n°	R	Max D variation (%)
1	53.79	-
2	52.85	
3	48.00	
4	50.58	0
5	52.13	
6	55.03	
7	54.83	0
8	54.44	
9	50.80	
10	56.06	22.83
11	55.30	
12	53.37	
13	54.42	1.25
14	53.29	
15	52.98	

Table 4.14: parameters to check the validity of the test (boxes from 16 to 30)

BOX n°	R	Max D variation (%)
16	-	-
17	42.53	
18	-11.92	
19	18.66	70.66
20	-17.16	
21	38.72	
22	-22.83	0
23	-22.27	
24	-20.58	
25	-12.14	80.21
26	46.33	
27	48.18	
28	-	4.39
29	-3.33	
30	36.57	

Table 4.15: parameters to check the validity of the test (boxes from 31 to 45)

BOX n°	R	Max D variation (%)
31	6.34	-
32	2.12	
33	12.22	
34	-5.16	15.21
35	3.37	
36	10.14	
37	1.62	0
38	4.02	
39	1.17	
40	7.56	14.42
41	8.12	
42	7.27	
43	8.55	1.50
44	11.16	
45	-7.69	

CHAPTER 5: CONCLUSIONS AND FUTURE STUDIES

5.1 CONCLUSION

The present work aimed at understanding the behaviour of different materials, some of them certified as compostable, simulating composting process conditions in a laboratory environment. A disintegration test, which is one of the steps to assess compostability, was carried out on selected materials. The experiment followed the procedure described in Standard ISO 20200:2015 "Determination of the degree of disintegration of plastic materials under simulated composting conditions in a laboratory-scale test", introducing some adjustments to implement a test that better represented a real composting process. To do so, a specific treatment plant located in the area of Lisbon was involved in the study. Material from different stages of the composting process was taken for the research: waste after 1 month of tunnel (denominated as Fresh Waste - FW) and the final product of the whole process (denominated Mature Compost - MC) were selected to be used as inoculums in the laboratory test. The materials selected for the experiment were Mater-Bi bags, PLA cups, Kraft cardboard and lastly conventional PET, chosen as negative control. The test essentially followed the procedure described in Standard ISO 20200, allowing some alterations to adapt it at the use of non-standardised inoculums.

The test proceeded for about 90 days, at the end of which the final product of the composting process contained in each reactor was characterised, and the disintegration degree was calculated. The results obtained for Mater-Bi and PLA processed in reactors containing SW (Synthetic Waste) are valid in terms of requirements set by ISO 20200, and are comparable to the ones obtained in previous studies. On the other hand, Kraft disintegration degree is not high enough to meet the requirements set by the Standard, and this is probably due to the fact that cellulosic materials, as stated in other scientific articles, need more time to degrade.

The situation is different for the samples processed in reactors containing FW and MC. Except for PLA that completely disintegrate in both inoculums coming from the facility, Mater-bi and Kraft materials gave different results as opposed to the ones obtained by disintegration test using standard inoculum SW. The degree of disintegration calculated for Mater-Bi in FW and MC is lower than the value observed for the same material in reactors containing SW. The same can be said for Kraft: its disintegration when processed together with MC or FW is basically 0%, lower than the one determined in the case of Kraft treated with SW. The negative aspect is that the results calculated at the end of the test in FW and MC reactors do not pass the validity check conditions set by the standard, probably due the presence of strong differences between FW and MC compared with SW, which affected the whole composting process. However, this does not mean that the whole experiment did not contribute to enhancing knowledge on the matter. Some results may be not valid for according to ISO 20200, but the test proved that what happens inside each treatment plant differs from the ideal situation described in the standard, depending on the type of waste and operational conditions (temperature, timelines, moisture, etc.) of the specific facility under analysis. And what makes the situation even more difficult is that every facility works differently. Consequently, since the degradation process in a composting plant is influenced by a wide range of variables, the behaviour of a potentially compostable material can differ for each facility. Carrying out tests like the one described in this thesis, taking into considerations faults and issues found along the experiment and summarised in this paper, could help each treatment facility to predict at what degree the material will disintegrate. After this assessment, those in charge of the plant can make their considerations and decide if a material can be accepted, if there is the need to change process parameters or some technologies, or if it’s more appropriate to send it to another treatment.

5.2 FUTURE STUDIES

The procedure presented challenges and errors, some of which could have been avoided if dealt with differently. A critic aspect that characterized the whole test was the use of inoculums coming from a treatment plant dealing with mixed waste. Even mature compost, which came from an operative line that treats food waste from restaurant selective collection, contained small pieces of glass and plastic. This resulted in solid matrixes that were already contaminated from the beginning by other substances. A large amount of impurities were manually taken out from the inoculums, but a part of them was not detected. Fresh waste inoculum was also highly heterogeneous, containing big pieces of waste of different origins, and difficult to be analysed as a whole. Summing up, these were not the best inoculums to use in this kind of test, as they negatively affected some important conditions, one of them being water retention capacity correlated to the optimal water content, essential for a successful composting process. Moreover, in the reactors containing fresh waste and mature compost, the quantity of tested sample was excessive in proportion to their specific volume. A lower quantity should have been selected according to this, to allow a better dispersion and easier burying of the pieces of samples. Additionally, the collection of residues at the end of the test is a complex and time-consuming procedure, done manually by the researchers that have to detect every piece visible to the naked eye, with the possibility of estimation errors. Considering that samples have no recognisable colour, and that inoculum is attached to them, it is not easy to find the residual material, even more so if the inoculum contains impurities such as in this case. The residues are also difficult to clean due to their fragility, so their final weight was overestimated, affecting the calculation of the disintegration degree. To partially overcome this problem, it is possible to estimate the quantity of inoculum attached to the residues using TGA (Thermal gravimetric analysis) and subtract it from the total weight loss (F. Ruggero, 2020).

The undersieve (< 2mm) of each reactor was collected and stored for future analysis on the presence of microplastic in compost, a study carried out by the same laboratory of NOVA FCT University. Parts of bioplastics can still be present in compost as micro and nano plastics. This matter is of increasing concern, and it will probably get more attention in the future. In addition, a germination test can be done to assess and compare the phytotoxicity of the solid matrixes before and after the disintegration test. Finally, the next step should involve a full-scale test implemented directly inside the treatment plant, to compare its results with the ones obtained in the lab. Unluckily, plant-scale tests are rarely taken into consideration because of their high costs, the lack of a specific methodology to carry out this kind test and the risk to contaminate the final compost that will be put on the market. However, they could play and important role in the near future facing the problem of a growing compostable packaging industry and consequently its increasing waste flux.

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ANNEX I:

Boxes weight registered throughout the test

Day 1 (Add H2O + Mix)			
	W initial (g)	Wi (g)	Wf (g)
1	**1309.20**	1288.7	1309.4
2	**1312.42**	1285.4	1313.8
3	**1311.96**	1296.8	1312.8
4	**1320.35**	1305.6	1320.7
5	**1314.27**	1284.1	1314.5
6	**1318.45**	1296.0	1318.7
7	**1339.66**	1306.8	1340.0
8	**1325.81**	1309.2	1326.4
9	**1327.28**	1304.9	1327.6
10	**1349.71**	1334.8	1349.9
11	**1351.71**	1342.1	1351.8
12	**1345.81**	1337.3	1346.0
13	**1327.95**	1278.0	1327.5
14	**1325.88**	1299.4	1326.2
15	**1326.66**	1228.9	1326.7
16	**-**	-	-
17	**1308.20**	1242.8	1308.6
18	**1303.79**	1284.9	1304.1
19	**1319.32**	1303.9	1319.6
20	**1310.23**	1296.3	1310.3
21	**1317.43**	1296.4	1317.8
22	**1322.78**	1315.5	1322.8
23	**1321.95**	1305.6	1322.0
24	**1321.83**	1308.3	1321.9
25	**1356.80**	1344.6	1357.0
26	**1343.60**	1333.5	1343.9
27	**1345.10**	1333.0	1345.1
28	**-**	-	-
29	**1337.88**	1322.4	1338.2
30	**1324.51**	1297.5	1324.6
31	**1308.13**	1295.4	1308.4
32	**1308.86**	1290.5	1309.1
33	**1308.99**	1293.2	1309.5
34	**1310.31**	1301.9	1310.5
35	**1319.10**	1308.0	1329.5
36	**1308.79**	1300.7	1309.2
37	**1333.20**	1320.1	1333.4
38	**1325.46**	1313.8	1325.5
39	**1324.40**	1310.3	1324.7
40	**1344.43**	1327.1	1345.0
41	**1347.70**	1319.6	1347.9
42	**1346.76**	1333.4	1346.9
43	**1327.58**	1316.2	1327.7
44	**1329.03**	1352.8	1329.6
45	**1328.68**	1320.0	1328.9

Day 2 (Add H_2O + Mix)			
Box nº	W initial (g)	Wi (g)	Wf (g)
1	**1309.20**	1301.3	1309.8
2	**1312.42**	1289.5	1312.7
3	**1311.96**	1304.1	1311.9
4	**1320.35**	1314.5	1320.4
5	**1314.27**	1209.1	1315.2
6	**1318.45**	1292.7	1318.7
7	**1339.66**	1301.2	1339.9
8	**1325.81**	1307.7	1325.8
9	**1327.28**	1285.6	1327.9
10	**1349.71**	1230.4	1349.7
11	**1351.71**	1315.4	1351.5
12	**1345.81**	1311.8	1347.3
13	**1327.95**	1284.4	1327.4
14	**1325.88**	1259.1	1325.2
15	**1326.66**	1283.0	1326.3
16	**-**	-	-
17	**1308.20**	1273.9	1308.4
18	**1303.79**	1288.2	1303.9
19	**1319.32**	1290.9	1319.1
20	**1310.23**	1290.7	1310.5
21	**1317.43**	1258.6	1352.4
22	**1322.78**	1309.4	1322.5
23	**1321.95**	1314.1	1321.4
24	**1321.83**	1266.3	1321.5
25	**1356.80**	1337.7	1356.3
26	**1343.60**	1301.0	1344.0
27	**1345.10**	1329.5	1345.2
28	**-**	-	-
29	**1337.88**	1253.1	1337.8
30	**1324.51**	1305.9	1324.1
31	**1308.13**	1277.5	1308.4
32	**1308.86**	1278.3	1308.4
33	**1308.99**	1280.5	1308.2
34	**1310.31**	1276.8	1310.2
35	**1319.10**	1284.6	1319.6
36	**1308.79**	1285.1	1308.1
37	**1333.20**	1305.1	1333.6
38	**1325.46**	1296.3	1325.2
39	**1324.40**	1298.3	1324.0
40	**1344.43**	1327.4	1344.0
41	**1347.70**	1321.1	1347.0
42	**1346.76**	1297.8	1346.2
43	**1327.58**	1312.5	1327.4
44	**1329.03**	1320.9	1329.9
45	**1328.68**	1294.1	1328.8

Day 3 (Add H2O + Mix)			
Box nº	W initial (g)	Wi (g)	Wf (g)
1	**1309.20**	1282.1	1309.8
2	**1312.42**	1294.6	1312.9
3	**1311.96**	1295.7	1313.5
4	**1320.35**	1310.9	1320.6
5	**1314.27**	1299.8	1314.9
6	**1318.45**	1301.1	1319.8
7	**1339.66**	1277.4	1341.8
8	**1325.81**	1321.7	1326.7
9	**1327.28**	1295.7	1327.6
10	**1349.71**	1309.9	1349.9
11	**1351.71**	1313.6	1352.2
12	**1345.81**	1323.4	1346.0
13	**1327.95**	1303.9	1327.9
14	**1325.88**	1305.6	1326.7
15	**1326.66**	1276.9	1329.5
16	**-**	-	-
17	**1308.20**	1284.5	1308.5
18	**1303.79**	1289.5	1304.0
19	**1319.32**	1295.1	1319.6
20	**1310.23**	1278.4	1310.3
21	**1317.43**	1291.3	1317.7
22	**1322.78**	1312.8	1323.7
23	**1321.95**	1295.0	1322.3
24	**1321.83**	1285.5	1322.2
25	**1356.80**	1346.6	1356.9
26	**1343.60**	1312.7	1343.6
27	**1345.10**	1319.1	1345.3
28	**-**	-	-
29	**1337.88**	1301.4	1338.5
30	**1324.51**	1307.1	1324.8
31	**1308.13**	1303.4	1308.5
32	**1308.86**	1291.4	1309.0
33	**1308.99**	1289.2	1309.0
34	**1310.31**	1288.5	1310.5
35	**1319.10**	1268.2	1329.5
36	**1308.79**	1298.2	1309.0
37	**1333.20**	1319.9	1336.6
38	**1325.46**	1307.6	1325.7
39	**1324.40**	1293.0	1324.6
40	**1344.43**	1334.1	1346.0
41	**1347.70**	1328.9	1348.0
42	**1346.76**	1331.1	1346.9
43	**1327.58**	1313.2	1328.2
44	**1329.03**	1318.4	1329.5
45	**1328.68**	1320.1	1329.3

Day 4 (Add H_2O + Mix)			
Box nº	W initial (g)	Wi (g)	Wf (g)
1	**1309.20**	1234.6	1309.0
2	**1312.42**	1285.8	1312.3
3	**1311.96**	1303.8	1311.3
4	**1320.35**	1310.6	1320.6
5	**1314.27**	1296.8	1314.8
6	**1318.45**	1309.6	1318.9
7	**1339.66**	1300.1	1339.8
8	**1325.81**	1298.8	1326.0
9	**1327.28**	1295.4	1327.8
10	**1349.71**	1323.3	1349.8
11	**1351.71**	1318.9	1351.3
12	**1345.81**	1332.0	1345.5
13	**1327.95**	1320.5	1327.4
14	**1325.88**	1309.3	1325.2
15	**1326.66**	1285.0	1326.4
16	**-**	-	-
17	**1308.20**	1267.4	1308.1
18	**1303.79**	1269.4	1303.6
19	**1319.32**	1293.8	1319.5
20	**1310.23**	1289.4	1310.0
21	**1317.43**	1290.0	1317.5
22	**1322.78**	1292.3	1322.4
23	**1321.95**	1308.3	1321.5
24	**1321.83**	1298.9	1321.9
25	**1356.80**	1336.9	1356.1
26	**1343.60**	1314.6	1343.2
27	**1345.10**	1323.7	1345.1
28	**-**	-	-
29	**1337.88**	1316.6	1337.2
30	**1324.51**	1295.4	1324.3
31	**1308.13**	1290.7	1308.2
32	**1308.86**	1273.8	1308.7
33	**1308.99**	1291.9	1308.1
34	**1310.31**	1293.1	1310.1
35	**1319.10**	1295.2	1319.3
36	**1308.79**	1298.0	1308.0
37	**1333.20**	1321.1	1333.8
38	**1325.46**	1352.5	1325.5
39	**1324.40**	1294.5	1324.3
40	**1344.43**	1321.1	1344.4
41	**1347.70**	1272.1	1347.3
42	**1346.76**	1305.6	1346.1
43	**1327.58**	1314.5	1327.6
44	**1329.03**	1294.3	1329.4
45	**1328.68**	1306.2	1328.9

Day 7 (Add H_2O + Mix)			
Box nº	W initial (g)	Wi (g)	Wf (g)
1	**1309.20**	1282.9	1309.9
2	**1312.42**	1287.8	1312.6
3	**1311.96**	1285.5	1312.7
4	**1320.35**	1295.8	1320.4
5	**1314.27**	1278.0	1314.7
6	**1318.45**	1255.1	1319.1
7	**1339.66**	1025.5	1340.0
8	**1325.81**	1209.9	1326.0
9	**1327.28**	1263.7	1328.3
10	**1349.71**	1236.2	1349.8
11	**1351.71**	1331.8	1351.9
12	**1345.81**	1283.1	1346.4
13	**1327.95**	1262.6	1328.2
14	**1325.88**	1282.0	1326.0
15	**1326.66**	1077.7	1328.7
16	**-**	-	-
17	**1308.20**	1156.1	1309.4
18	**1303.79**	1233.4	1304.6
19	**1319.32**	1236.5	1320.3
20	**1310.23**	1204.4	1310.3
21	**1317.43**	1221.8	1318.3
22	**1322.78**	1237.1	1322.8
23	**1321.95**	1247.4	1322.0
24	**1321.83**	1233.1	1321.8
25	**1356.80**	1317.3	1356.9
26	**1343.60**	1279.4	1344.8
27	**1345.10**	1327.1	1345.3
28	**-**	-	-
29	**1337.88**	1264.1	1338.0
30	**1324.51**	1234.0	1324.8
31	**1308.13**	1296.2	1309.2
32	**1308.86**	1236.3	1310.2
33	**1308.99**	1292.4	1309.6
34	**1310.31**	1153.7	1310.8
35	**1319.10**	1280.7	1319.1
36	**1308.79**	1258.4	1309.2
37	**1333.20**	1296.1	1333.8
38	**1325.46**	1261.0	1326.0
39	**1324.40**	1289.5	1325.2
40	**1344.43**	1221.3	1344.6
41	**1347.70**	1177.8	1347.7
42	**1346.76**	1289.4	1347.0
43	**1327.58**	1262.9	1327.6
44	**1329.03**	1255.3	1329.9
45	**1328.68**	1141.1	1329.4

Day 8 (Add H2O)			
Box nº	W initial (g)	Wi (g)	Wf (g)
1	**1309.20**	1303.8	1310.1
2	**1312.42**	1291.8	1313.1
3	**1311.96**	1305.6	1312.5
4	**1320.35**	1304.0	1322.8
5	**1314.27**	1297.5	1315.0
6	**1318.45**	1292.4	1320.7
7	**1339.66**	1323.3	1340.1
8	**1325.81**	1298.4	1326.1
9	**1327.28**	1309.8	1328.1
10	**1349.71**	1318.4	1350.8
11	**1351.71**	1338.2	1352.1
12	**1345.81**	1318.1	1346.2
13	**1327.95**	1312.7	1329.1
14	**1325.88**	1280.7	1326.5
15	**1326.66**	1308.1	1327.2
16	**-**	-	-
17	**1308.20**	1281.8	1309.7
18	**1303.79**	1276.0	1304.4
19	**1319.32**	1290.4	1320.1
20	**1310.23**	1292.6	1311.7
21	**1317.43**	1233.2	1318.2
22	**1322.78**	1293.6	1323.4
23	**1321.95**	1278.2	1322.3
24	**1321.83**	1296.0	1322.2
25	**1356.80**	1344.5	1358.0
26	**1343.60**	1226.1	1344.1
27	**1345.10**	1313.5	1346.4
28	**-**	-	-
29	**1337.88**	1320.6	1338.2
30	**1324.51**	1310.6	1325.4
31	**1308.13**	1295.4	1309.7
32	**1308.86**	1298.8	1309.2
33	**1308.99**	1303.1	1309.3
34	**1310.31**	1220.0	1311.2
35	**1319.10**	1285.7	1320.5
36	**1308.79**	1289.3	1309.3
37	**1333.20**	1326.3	1334.2
38	**1325.46**	1289.6	1326.4
39	**1324.40**	1295.1	1325.8
40	**1344.43**	1327.1	1345.3
41	**1347.70**	1334.5	1348.4
42	**1346.76**	1337.6	1347.8
43	**1327.58**	1307.6	1328.1
44	**1329.03**	1322.3	1330.0
45	**1328.68**	1306.2	1329.2

Day 9 (Add H2O + Mix)			
Box nº	W initial (g)	Wi (g)	Wf (g)
1	**1309.20**	1295.5	1310.9
2	**1312.42**	1298.1	1316.9
3	**1311.96**	1293.9	1312.0
4	**1320.35**	1304.0	1326.5
5	**1314.27**	1302.4	1317.5
6	**1318.45**	1304.0	1323.9
7	**1339.66**	1329.3	1339.8
8	**1325.81**	1309.4	1326.5
9	**1327.28**	1323.9	1328.0
10	**1349.71**	1335.5	1350.2
11	**1351.71**	1326.9	1355.4
12	**1345.81**	1328.7	1346.3
13	**1327.95**	1299.1	1330.4
14	**1325.88**	1310.9	1326.1
15	**1326.66**	1234.4	1327.1
16	**-**	-	-
17	**1308.20**	1286.4	1309.7
18	**1303.79**	1273.3	1305.3
19	**1319.32**	1281.5	1320.2
20	**1310.23**	1291.0	1311.3
21	**1317.43**	1297.3	1318.4
22	**1322.78**	1307.3	1322.8
23	**1321.95**	1301.3	1322.6
24	**1321.83**	1301.8	1322.3
25	**1356.80**	1347.8	1357.4
26	**1343.60**	1334.1	1322.9
27	**1345.10**	1314.8	1346.5
28	**-**	-	-
29	**1337.88**	1325.0	1338.9
30	**1324.51**	1315.1	1325.1
31	**1308.13**	1248.9	1308.6
32	**1308.86**	1278.7	1309.8
33	**1308.99**	1265.7	1309.5
34	**1310.31**	1301.1	1311.2
35	**1319.10**	1299.8	1320.2
36	**1308.79**	1292.5	1309.1
37	**1333.20**	1286.3	1335.0
38	**1325.46**	1312.5	1326.3
39	**1324.40**	1312.2	1325.7
40	**1344.43**	1331.2	1345.1
41	**1347.70**	1328.3	1348.1
42	**1346.76**	1331.8	1347.5
43	**1327.58**	1311.3	1331.1
44	**1329.03**	1319.5	1329.2
45	**1328.68**	1311.7	1329.5

Day 10 (Add H2O)			
Box nº	W initial (g)	Wi (g)	Wf (g)
1	**1309.20**	1304.9	1311.0
2	**1312.42**	1261.8	1313.8
3	**1311,96**	1306.1	1313.6
4	**1320.35**	1315.9	1321.5
5	**1314.27**	1308.5	1315.6
6	**1318.45**	1309.2	1319.5
7	**1339.66**	1293.2	1341.0
8	**1325.81**	1310.1	1326.3
9	**1327.28**	1316.1	1329.7
10	**1349.71**	1323.6	1350.1
11	**1351.71**	1347.5	1352.5
12	**1345.81**	1328.1	1346.3
13	**1327.95**	1307.7	1330.4
14	**1325.88**	1285.2	1326.1
15	**1326.66**	1284.6	1328.8
16	**-**	-	-
17	**1308.20**	1282.5	1311.3
18	**1303.79**	1285.4	1304.8
19	**1319.32**	1292.0	1320.8
20	**1310.23**	1283.8	1312.6
21	**1317.43**	1261.1	1319.1
22	**1322.78**	1296.7	1324.0
23	**1321.95**	1299.7	1322.5
24	**1321.83**	1316.7	1322.9
25	**1356.80**	1328.8	1357.8
26	**1343.60**	1319.1	1346.8
27	**1345.10**	1332.3	1346.1
28	**-**	-	-
29	**1337.88**	1326.0	1338.4
30	**1324.51**	1262.5	1326.4
31	**1308.13**	1292.7	1310.4
32	**1308.86**	1293.9	1309.8
33	**1308.99**	1290.0	1309.3
34	**1310.31**	1287.3	1312.9
35	**1319.10**	1281.6	1320.2
36	**1308.79**	1297.9	1309.2
37	**1333.20**	1286.7	1334.3
38	**1325.46**	1302.6	1326.2
39	**1324.40**	1310.7	1325.2
40	**1344.43**	1330.1	1346.2
41	**1347.70**	1336.0	1348.5
42	**1346.76**	1314.0	1347.8
43	**1327.58**	1322.0	1328.1
44	**1329.03**	1309.3	1330.3
45	**1328.68**	1311.7	1329.6

Day 11 (Add H_2O + Mix)			
Box nº	W initial (g)	Wi (g)	Wf (g)
1	**1309.20**	1297.8	1310.0
2	**1312.42**	1298.8	1312.6
3	**1311.96**	1308.9	1312.6
4	**1320.35**	1315.0	1321.9
5	**1314.27**	1300.8	1314.3
6	**1318.45**	1301.4	1318.8
7	**1339.66**	1321.4	1344.6
8	**1325.81**	1308.1	1326.2
9	**1327.28**	1309.2	1328.3
10	**1349.71**	1340.5	1350.7
11	**1351.71**	1301.8	1352.2
12	**1345.81**	1339.1	1345.8
13	**1327.95**	1295.6	1329.9
14	**1325.88**	1294.1	1326.3
15	**1326.66**	1319.1	1327.5
16	**-**	-	-
17	**1308.20**	1269.8	1309.5
18	**1303.79**	1363.1	1304.3
19	**1319.32**	1299.4	1320.6
20	**1310.23**	1280.9	1311.4
21	**1317.43**	1297.2	1318.9
22	**1322.78**	1306.0	1323.7
23	**1321.95**	1310.3	1323.3
24	**1321.83**	1291.9	1322.7
25	**1356.80**	1331.5	1357.8
26	**1343.60**	1326.9	1343.9
27	**1345.10**	1288.8	1345.4
28	**-**	-	-
29	**1337.88**	1322.0	1338.0
30	**1324.51**	1326.9	1324.8
31	**1308.13**	1296.9	1309.0
32	**1308.86**	1295.4	1309.2
33	**1308.99**	1299.6	1309.3
34	**1310.31**	1288.3	1311.7
35	**1319.10**	1303.4	1319.1
36	**1308.79**	1295.6	1309.0
37	**1333.20**	1305.6	1334.2
38	**1325.46**	1308.1	1327.0
39	**1324.40**	1310.4	1325.7
40	**1344.43**	1327.9	1345.4
41	**1347.70**	1331.5	1348.7
42	**1346.76**	1321.4	1348.9
43	**1327.58**	1298.1	1328.7
44	**1329.03**	1313.9	1330.4
45	**1328.68**	1319.4	1329.3

Day 14 (Add H2O + Mix)			
Box nº	W initial (g)	Wi (g)	Wf (g)
1	**1309.20**	1231.1	1309.4
2	**1312.42**	1247.0	1313.0
3	**1311.96**	1269.9	1313.2
4	**1320.35**	1277.7	1322.0
5	**1314.27**	1295.1	1315.5
6	**1318.45**	1262.4	1319.3
7	**1339.66**	1308.5	1339.9
8	**1325.81**	1384.8	1325.9
9	**1327.28**	1366.5	1328.1
10	**1349.71**	1324.2	1350.1
11	**1351.71**	1326.6	1351.8
12	**1345.81**	1306.1	1346.4
13	**1327.95**	1255.6	1329.1
14	**1325.88**	1267.3	1326.8
15	**1326.66**	1310.7	1326.8
16	**-**	-	-
17	**1308.20**	1233.1	1309.6
18	**1303.79**	1224.1	1304.3
19	**1319.32**	1233.1	1319.6
20	**1310.23**	1204.4	1311.9
21	**1317.43**	1286.3	1318.5
22	**1322.78**	1240.2	1323.6
23	**1321.95**	1249.1	1322.3
24	**1321.83**	1284.9	1322.9
25	**1356.80**	1299.9	1357.6
26	**1343.60**	1233.1	1344.3
27	**1345.10**	1277.1	1346.7
28	**-**	-	-
29	**1337.88**	1295.4	1337.9
30	**1324.51**	1287.3	1324.8
31	**1308.13**	1225.7	1308.4
32	**1308.86**	1269.2	1309.1
33	**1308.99**	1255.9	1309.3
34	**1310.31**	1108.4	1310.6
35	**1319.10**	1261.3	1319.3
36	**1308.79**	1266.5	1308.8
37	**1333.20**	1258.6	1333.9
38	**1325.46**	1286.4	1326.0
39	**1324.40**	1148.6	1324.5
40	**1344.43**	1329.1	1345.4
41	**1347.70**	1078.0	1347.9
42	**1346.76**	1278.1	1346.9
43	**1327.58**	1254.7	1327.6
44	**1329.03**	1226.6	1329.4
45	**1328.68**	1298.5	1329.2

Day 16 (Add H2O)			
Box nº	W initial (g)	Wi (g)	Wf (g)
1	**1309.20**	1261.3	1309.4
2	**1312.42**	1372.8	1313.4
3	**1311.96**	1342.3	1312.4
4	**1320.35**	1302.9	1320.8
5	**1314.27**	1281.4	1315.5
6	**1318.45**	1372.1	1321.2
7	**1339.66**	1323.5	1340.1
8	**1325.81**	1304.0	1326.3
9	**1327.28**	1296.3	1328.2
10	**1349.71**	1322.5	1350.6
11	**1351.71**	1311.4	1354.1
12	**1345.81**	1322.4	1346.6
13	**1327.95**	1289.5	1328.4
14	**1325.88**	1285.3	1326.2
15	**1326.66**	1280.3	1327.2
16	**-**	-	-
17	**1308.20**	1255.8	1309.1
18	**1303.79**	1269.3	1304.0
19	**1319.32**	1249.4	1319.8
20	**1310.23**	1269.2	1312.1
21	**1317.43**	1103.1	1318.7
22	**1322.78**	1297.6	1326.3
23	**1321.95**	1282.7	1322.3
24	**1321.83**	1284.8	1322.1
25	**1356.80**	1340.8	1357.8
26	**1343.60**	1283.0	1344.1
27	**1345.10**	1323.3	1345.4
28	**-**	-	-
29	**1337.88**	1326.1	1338.4
30	**1324.51**	1258.7	1324.7
31	**1308.13**	1285.5	1308.7
32	**1308.86**	1295.5	1310.7
33	**1308.99**	1284.7	1309.8
34	**1310.31**	1254.6	1311.0
35	**1319.10**	1274.3	1320.1
36	**1308.79**	1144.4	1309.3
37	**1333.20**	1279.0	1334.1
38	**1325.46**	1309.1	1326.1
39	**1324.40**	1276.7	1325.6
40	**1344.43**	1338.8	1346.0
41	**1347.70**	1262.4	1348.8
42	**1346.76**	1293.5	1347.2
43	**1327.58**	1298.6	1328.2
44	**1329.03**	1267.7	1329.5
45	**1328.68**	1276.4	1329.1

Day 18 (Add H_2O)			
Box nº	W initial (g)	Wi (g)	Wf (g)
1	**1309.20**	1287.9	1310.2
2	**1312.42**	1275.4	1312.8
3	**1311.96**	1297.8	1312.1
4	**1320.35**	1276.6	1320.9
5	**1314.27**	1297.8	1314.8
6	**1318.45**	1287.6	1318.8
7	**1339.66**	1308.9	1340.2
8	**1325.81**	1283.4	1326.0
9	**1327.28**	1268.1	1327.6
10	**1349.71**	1328.2	1349.8
11	**1351.71**	1331.1	1352.1
12	**1345.81**	1307.3	1346.2
13	**1327.95**	1147.3	1328.1
14	**1325.88**	1289.0	1326.2
15	**1326.66**	1299.0	1327.1
16	**-**	-	-
17	**1308.20**	1192.8	1309.0
18	**1303.79**	1243.9	1305.3
19	**1319.32**	1266.8	1320.0
20	**1310.23**	1287.5	1310.5
21	**1317.43**	1307.5	1318.7
22	**1322.78**	1249.5	1323.2
23	**1321.95**	1281.2	1323.4
24	**1321.83**	1301.0	1322.2
25	**1356.80**	1295.2	1357.0
26	**1343.60**	1141.7	1344.1
27	**1345.10**	1317.7	1345.2
28	**-**	-	-
29	**1337.88**	1315.1	1338.4
30	**1324.51**	1275.4	1324.8
31	**1308.13**	1284.4	1308.5
32	**1308.86**	1284.1	1309.5
33	**1308.99**	1246.6	1309.1
34	**1310.31**	1219.5	1310.4
35	**1319.10**	1275.5	1320.0
36	**1308.79**	1301.0	1309.4
37	**1333.20**	1283.4	1333.6
38	**1325.46**	1247.0	1325.8
39	**1324.40**	1274.2	1325.4
40	**1344.43**	1328.6	1345.5
41	**1347.70**	1263.4	1348.1
42	**1346.76**	1309.3	1347.3
43	**1327.58**	1305.8	1328.1
44	**1329.03**	1301.6	1329.2
45	**1328.68**	1307.5	1328.9

Day 21 (Add H_2O)			
Box nº	W initial (g)	Wi (g)	Wf (g)
1	**1309.20**	1283.4	1309.5
2	**1312.42**	1252.2	1312.6
3	**1311.96**	1298.0	1315.8
4	**1320.35**	1296.1	1320.8
5	**1314.27**	1291.2	1314.6
6	**1318.45**	1254.8	1318.9
7	**1339.66**	1325.1	1340.4
8	**1325.81**	1301.6	1326.4
9	**1327.28**	1312.1	1329.1
10	**1349.71**	1206.6	1350.0
11	**1351.71**	1311.3	1352.7
12	**1345.81**	1280.5	1345.9
13	**1327.95**	1304.0	1328.9
14	**1325.88**	1292.0	1326.4
15	**1326.66**	1247.0	1327.6
16	**-**	-	-
17	**1308.20**	1229.7	1309.9
18	**1303.79**	1277.8	1304.6
19	**1319.32**	1218.4	1319.9
20	**1310.23**	1273.4	1311.2
21	**1317.43**	1279.0	1317.8
22	**1322.78**	1236.0	1323.0
23	**1321.95**	1236.7	1323.6
24	**1321.83**	1303.6	1322.9
25	**1356.80**	1262.6	1357.0
26	**1343.60**	1249.2	1344.3
27	**1345.10**	1284.5	1346.3
28	**-**	-	-
29	**1337.88**	1310.2	1338.1
30	**1324.51**	1225.6	1325.6
31	**1308.13**	1252.4	1313.0
32	**1308.86**	1247.6	1309.3
33	**1308.99**	1285.6	1309.8
34	**1310.31**	1187.6	1310.5
35	**1319.10**	1272.5	1319.9
36	**1308.79**	1278.8	1309.1
37	**1333.20**	1217.7	1333.3
38	**1325.46**	1258.5	1326.5
39	**1324.40**	1272.1	1324.7
40	**1344.43**	1276.5	1352.9
41	**1347.70**	1228.0	1348.1
42	**1346.76**	1238.3	1351.7
43	**1327.58**	1106.4	1328.5
44	**1329.03**	1305.1	1329.7
45	**1328.68**	1144.2	1331.5

Day 23 (Add H_2O)			
Box nº	W initial (g)	Wi (g)	Wf (g)
1	**1309.20**	1302.5	1309.3
2	**1312.42**	1278.9	1312.6
3	**1311.96**	1298.2	1312.1
4	**1320.35**	1311.0	1322.0
5	**1314.27**	1280.7	1324.5
6	**1318.45**	1303.1	1329.0
7	**1339.66**	1281.6	1340.5
8	**1325.81**	1276.8	1326.0
9	**1327.28**	1309.6	1327.4
10	**1349.71**	1324.5	1350.3
11	**1351.71**	1311.2	1352.9
12	**1345.81**	1223.5	1346.5
13	**1327.95**	1309.7	1328.5
14	**1325.88**	1304.9	1326.1
15	**1326.66**	1309.6	1327.0
16	**-**	-	-
17	**1308.20**	1111.9	1308.8
18	**1303.79**	1233.4	1307.0
19	**1319.32**	1228.4	1319.5
20	**1310.23**	1267.2	1311.3
21	**1317.43**	1284.4	1318.7
22	**1322.78**	1287.9	1324.2
23	**1321.95**	1281.1	1322.0
24	**1321.83**	1274.5	1322.5
25	**1356.80**	1335.3	1347.0
26	**1343.60**	1324.6	1344.8
27	**1345.10**	1285.1	1346.2
28	**-**	-	-
29	**1337.88**	1314.2	1338.0
30	**1324.51**	1277.8	1324.6
31	**1308.13**	1263.1	1309.2
32	**1308.86**	1262.9	1309.8
33	**1308.99**	1259.8	1310.5
34	**1310.31**	1271.3	1310.5
35	**1319.10**	1372.7	1319.4
36	**1308.79**	1278.8	1308.8
37	**1333.20**	1294.7	1333.8
38	**1325.46**	1276.3	1325.6
39	**1324.40**	1237.3	1324.6
40	**1344.43**	1168.8	1344.5
41	**1347.70**	1295.2	1347.8
42	**1346.76**	1290.8	1346.9
43	**1327.58**	1295.9	1327.8
44	**1329.03**	1282.4	1329.2
45	**1328.68**	1306.1	1329.3

Day 25 (Add H_2O)			
Box nº	W initial (g)	Wi (g)	Wf (g)
1	**1309.20**	1273.2	1309.6
2	**1312.42**	1301.3	1312.5
3	**1311.96**	1302.7	1314.6
4	**1320.35**	1293.4	1321.5
5	**1314.27**	1286.2	1314.7
6	**1318.45**	1302.3	1318.6
7	**1339.66**	1306.3	1339.7
8	**1325.81**	1292.3	1326.3
9	**1327.28**	1279.0	1327.8
10	**1349.71**	1297.8	1349.9
11	**1351.71**	1334.0	1351.7
12	**1345.81**	1308.2	1346.4
13	**1327.95**	1302.4	1328.0
14	**1325.88**	1293.4	1326.1
15	**1326.66**	1193.2	1327.0
16	**-**	-	-
17	**1308.20**	1255.0	1312.0
18	**1303.79**	1295.6	1305.8
19	**1319.32**	1257.6	1320.4
20	**1310.23**	1284.5	1310.3
21	**1317.43**	1276.0	1317.7
22	**1322.78**	1274.6	1322.9
23	**1321.95**	1275.0	1322.2
24	**1321.83**	1285.7	1322.3
25	**1356.80**	1323.3	1357.0
26	**1343.60**	1313.1	1344.0
27	**1345.10**	1293.1	1345.3
28	**-**	-	-
29	**1337.88**	1288.9	1338.2
30	**1324.51**	1362.5	1324.8
31	**1308.13**	1298.3	1308.4
32	**1308.86**	1266.9	1309.2
33	**1308.99**	1293.5	1309.1
34	**1310.31**	1241.1	1311.0
35	**1319.10**	1250.8	1319.6
36	**1308.79**	1244.9	1311.4
37	**1333.20**	1252.8	1333.5
38	**1325.46**	1277.1	1327.4
39	**1324.40**	1296.4	1324.5
40	**1344.43**	1297.0	1344.6
41	**1347.70**	1306.8	1347.8
42	**1346.76**	1216.5	1347.5
43	**1327.58**	1317.4	1327.7
44	**1329.03**	1303.4	1329.3
45	**1328.68**	1235.3	1329.4

Day 28 (Add H_2O)			
Box nº	W initial (g)	Wi (g)	Wf (g)
1	**1309.20**	1278.4	1309.5
2	**1312.42**	1289.9	1312.4
3	**1311.96**	1294.6	1311.2
4	**1320.35**	1308.7	1320.7
5	**1314.27**	1286.7	1314.9
6	**1318.45**	1394.6	1318.8
7	**1339.66**	1181.2	1339.6
8	**1325.81**	1275.4	1325.3
9	**1327.28**	1241.6	1327.5
10	**1349.71**	1332.2	1349.6
11	**1351.71**	1322.0	1351.2
12	**1345.81**	1303.9	1345.9
13	**1327.95**	1231.0	1327.8
14	**1325.88**	1228.0	1325.6
15	**1326.66**	1181.0	1326.6
16	**-**	-	-
17	**1308.20**	1093.5	1308.7
18	**1303.79**	1236.3	1303.7
19	**1319.32**	1151.1	1319.6
20	**1310.23**	1024.0	1310.0
21	**1317.43**	1260.7	1317.6
22	**1322.78**	1271.1	1322.6
23	**1321.95**	1223.8	1321.4
24	**1321.83**	1174.8	1321.7
25	**1356.80**	1336.5	1356.2
26	**1343.60**	1236.1	1343.5
27	**1345.10**	1199.4	1345.7
28	**-**	-	-
29	**1337.88**	1269.5	1337.5
30	**1324.51**	1063.5	1324.8
31	**1308.13**	1277.2	1308.6
32	**1308.86**	1274.2	1308.4
33	**1308.99**	1232.5	1308.7
34	**1310.31**	1199.3	1310.1
35	**1319.10**	1273.0	1319.4
36	**1308.79**	1257.3	1308.1
37	**1333.20**	1143.5	1333.8
38	**1325.46**	1264.9	1325.6
39	**1324.40**	1200.9	1324.4
40	**1344.43**	1241.9	1344.1
41	**1347.70**	1032.6	1347.7
42	**1346.76**	1254.4	1346.6
43	**1327.58**	1217.0	1327.1
44	**1329.03**	1245.0	1329.3
45	**1328.68**	1220.7	1328.2

Day 31 (Add H2O + Mix)			
Box nº	80% W initial (g)	Wi (g)	Wf (g)
1	**1047.36**	1287.5	1286.3
2	**1049.94**	1300.4	1299.5
3	**1049.57**	1304.6	1304.1
4	**1056.28**	1222.0	1219.5
5	**1051.21**	1275.0	1273.0
6	**1054.76**	1288.9	1287.6
7	**1071.73**	1265.6	1264.7
8	**1060.65**	1288.1	1287.7
9	**1061.82**	1297.8	1297.2
10	**1079.77**	1318.5	1316.3
11	**1081.37**	1342.8	1341.5
12	**1076.65**	1301.3	1299.1
13	**1062.36**	1277.1	1276.7
14	**1060.70**	1256.8	1256.4
15	**1061.33**	1312.9	1311.8
16	**-**	-	-
17	**1046.56**	1236.5	1235.2
18	**1043.03**	1261.3	1260.4
19	**1055.46**	1275.2	1272.9
20	**1048.18**	1284.0	1282.5
21	**1053.94**	1258.3	1254.7
22	**1058.22**	1299.3	1297.8
23	**1057.56**	1276.5	1274.6
24	**1057.46**	1279.1	1277.2
25	**1085.44**	1308.2	1307.5
26	**1074.88**	1328.2	1324.6
27	**1076.08**	1243.2	1241.8
28	**-**	-	-
29	**1070.30**	1315.3	1314.1
30	**1059.61**	1296.0	1294.2
31	**1046.50**	1155.5	1154.4
32	**1047.09**	1250.6	1250.0
33	**1047.19**	1262.1	1261.6
34	**1048.25**	1235.1	1233.9
35	**1055.28**	1297.6	1296.2
36	**1047.03**	1257.4	1256.0
37	**1066.56**	1298.9	1297.2
38	**1060.37**	1266.9	1265.7
39	**1059.52**	1285.3	1283.4
40	**1075.54**	1305.0	1303.6
41	**1078.16**	1288.5	1285.5
42	**1077.41**	1312.6	1310.4
43	**1062.06**	1297.1	1295.7
44	**1063.22**	1312.7	1312.3
45	**1062.94**	1174.3	1174.0

Day 35 (Add H2O)			
Box nº	80% W initial (g)	Wi (g)	Wf (g)
1	**1047.36**	1254.8	-
2	**1049.94**	1281.6	-
3	**1049.57**	1282.0	-
4	**1056.28**	1131.8	-
5	**1051.21**	1220.3	-
6	**1054.76**	1197.9	-
7	**1071.73**	1060.7	1071.9
8	**1060.65**	1131.3	-
9	**1061.82**	1261.3	-
10	**1079.77**	918.8	1080.2
11	**1081.37**	1282.7	-
12	**1076.65**	1143.6	-
13	**1062.36**	1203.9	-
14	**1060.70**	1192.8	-
15	**1061.33**	1129.1	-
16	**-**	-	-
17	**1046.56**	1169.7	1180.1
18	**1043.03**	1126.0	1155.9
19	**1055.46**	856.0	1055.8
20	**1048.18**	1208.7	1220.9
21	**1053.94**	1229.5	-
22	**1058.22**	1135.1	1156.8
23	**1057.56**	1130.5	1161.8
24	**1057.46**	873.3	1058.3
25	**1085.44**	1211.5	1241.2
26	**1074.88**	1150.8	1170.4
27	**1076.08**	905.7	1077.0
28	**-**	-	-
29	**1070.30**	1246.8	1272.5
30	**1059.61**	1254,.9	-
31	**1046.50**	1033.0	1062.8
32	**1047.09**	1091.2	1121.9
33	**1047.19**	1122.9	1159.6
34	**1048.25**	1093.4	1121.2
35	**1055.28**	1181.2	1208.5
36	**1047.03**	1113.8	1138.3
37	**1066.56**	1163.6	1198.2
38	**1060.37**	1118.9	1146.2
39	**1059.52**	1209.0	1234.1
40	**1075.54**	1105.1	1140.0
41	**1078.16**	1184.8	1199.7
42	**1077.41**	1107.6	1137.4
43	**1062.06**	1217.3	-
44	**1063.22**	1158.1	1175.3
45	**1062.94**	1135.110	1156.0

Day 38 (Add H2O)			
Box nº	80% W initial (g)	Wi (g)	Wf (g)
1	**1047.36**	1195.8	-
2	**1049.94**	1218.0	-
3	**1049.57**	1273.1	-
4	**1056.28**	1090.6	-
5	**1051.21**	1178.3	-
6	**1054.76**	1167.2	-
7	**1071.73**	980.1	1072.8
8	**1060.65**	1047.0	1060.7
9	**1061.82**	1217.6	-
10	**1079.77**	929.4	1080.3
11	**1081.37**	1248.8	-
12	**1076.65**	1076.8	-
13	**1062.36**	1168.1	-
14	**1060.70**	1126.5	-
15	**1061.33**	1025.9	-
16	**-**	-	-
17	**1046.56**	1018.8	1048.3
18	**1043.03**	1037.8	1044.2
19	**1055.46**	842.2	1057.1
20	**1048.18**	1065.0	1081.5
21	**1053.94**	1151.2	-
22	**1058.22**	1118.8	1138.6
23	**1057.56**	1019.4	1061.2
24	**1057.46**	933.6	1057.8
25	**1085.44**	1126.7	1154.0
26	**1074.88**	1077.3	1137.8
27	**1076.08**	1051.4	1099.0
28	**-**	-	-
29	**1070.30**	1208.4	-
30	**1059.61**	1164.2	1193.4
31	**1046.50**	954.8	1047.5
32	**1047.09**	1055.2	1102.0
33	**1047.19**	1079.0	1101.1
34	**1048.25**	1082.7	1117.7
35	**1055.28**	1095.0	1129.7
36	**1047.03**	1012.5	1090.5
37	**1066.56**	962.7	1068.1
38	**1060.37**	1043.5	1089.4
39	**1059.52**	1082.1	1110.9
40	**1075.54**	1065.3	1104.4
41	**1078.16**	941.9	1078.8
42	**1077.41**	972.6	1079.5
43	**1062.06**	1186.4	-
44	**1063.22**	1123.1	-
45	**1062.94**	1042.2	1064.6

Day 42 (Add H_2O)			
Box nº	80% W initial (g)	Wi (g)	Wf (g)
1	**1047.36**	1155.1	-
2	**1049.94**	1130.8	-
3	**1049.57**	1114.1	-
4	**1056.28**	1033.4	1059.0
5	**1051.21**	1060.0	-
6	**1054.76**	1055.7	-
7	**1071.73**	990.3	1072.2
8	**1060.65**	1014.5	1062.3
9	**1061.82**	1093.5	-
10	**1079.77**	996.4	1081.2
11	**1081.37**	1201.3	-
12	**1076.65**	1023.3	1078.1
13	**1062.36**	1108.1	-
14	**1060.70**	1008.3	1061.1
15	**1061.33**	961.4	1061.9
16	**-**	-	-
17	**1046.56**	1004.6	1047.0
18	**1043.03**	942.7	1044.1
19	**1055.46**	987.8	1056.9
20	**1048.18**	1061.8	-
21	**1053.94**	1015.2	1056.3
22	**1058.22**	964.0	1060.8
23	**1057.56**	897.7	1058.5
24	**1057.46**	932.0	1058.0
25	**1085.44**	898.1	1085.9
26	**1074.88**	914.3	1075.0
27	**1076.08**	952.0	1078.8
28	**-**	-	-
29	**1070.30**	1145.8	-
30	**1059.61**	926.7	1069.0
31	**1046.50**	987.1	1049.3
32	**1047.09**	881.9	1048.7
33	**1047.19**	990.5	1047.8
34	**1048.25**	906.6	1048.9
35	**1055.28**	1029.3	1063.6
36	**1047.03**	1060.8	1097.6
37	**1066.56**	993.7	1067.9
38	**1060.37**	902.8	1062.9
39	**1059.52**	1042.0	1062.4
40	**1075.54**	1059.3	1078.6
41	**1078.16**	933.4	1078.5
42	**1077.41**	979.9	1079.3
43	**1062.06**	1008.7	1062.2
44	**1063.22**	986.7	1064.4
45	**1062.94**	947.9	1063.0

Day 45 (Add H2O + Mix)			
Box nº	80% W initial (g)	Wi (g)	Wf (g)
1	**1047.36**	1084.2	1083.1
2	**1049.94**	1093.1	1090.9
3	**1049.57**	1089.3	1087.6
4	**1056.28**	1034.8	1056.3
5	**1051.21**	1000.9	1051.5
6	**1054.76**	960.6	1054.8
7	**1071.73**	890.7	1072.2
8	**1060.65**	968.1	1060.7
9	**1061.82**	1055.9	1062.1
10	**1079.77**	1051.6	1076.8
11	**1081.37**	1156.5	1154.5
12	**1076.65**	971.4	1076.7
13	**1062.36**	914.5	1062.4
14	**1060.70**	906.6	1062.3
15	**1061.33**	923.7	1063.9
16	**-**	-	-
17	**1046.56**	905.2	1046.6
18	**1043.03**	936.2	103.4
19	**1055.46**	820.6	1055.8
20	**1048.18**	979.8	1049.1
21	**1053.94**	912.6	1058.4
22	**1058.22**	1012.5	1059.2
23	**1057.56**	929.1	1058.1
24	**1057.46**	1010.1	1057.7
25	**1085.44**	996.3	1085.7
26	**1074.88**	1007.9	1075.0
27	**1076.08**	1008.8	1076.9
28	**-**	-	-
29	**1070.30**	990.5	1071.0
30	**1059.61**	921.2	1059.9
31	**1046.50**	1027.0	1047.0
32	**1047.09**	988.1	1047.5
33	**1047.19**	968.0	1049.4
34	**1048.25**	972.9	1048.7
35	**1055.28**	877.2	1055.5
36	**1047.03**	986.3	1047.4
37	**1066.56**	953.0	1066.7
38	**1060.37**	929.0	2060.4
39	**1059.52**	1045.5	1084.1
40	**1075.54**	1026.7	1144.5
41	**1078.16**	903.7	1078.6
42	**1077.41**	934.5	1077.8
43	**1062.06**	1001.2	1063.2
44	**1063.22**	976.0	1063.3
45	**1062.94**	947.7	1063.0

Day 49 (Add H2O)			
Box nº	80% W initial (g)	Wi (g)	Wf (g)
1	**1047.36**	1009.1	1047.7
2	**1049.94**	1049.4	1050.0
3	**1049.57**	1053.6	-
4	**1056.28**	1021.4	1057.2
5	**1051.21**	1018.3	1051.7
6	**1054.76**	911.5	1055.2
7	**1071.73**	872.6	1071.8
8	**1060.65**	897.7	1060.7
9	**1061.82**	1002.7	1062.3
10	**1079.77**	996.5	1080.9
11	**1081.37**	1068.0	1082.9
12	**1076.65**	1003.8	1077.0
13	**1062.36**	997.1	1062.7
14	**1060.70**	848.4	1061.0
15	**1061.33**	961.3	1061.7
16	**-**	-	-
17	**1046.56**	995.4	1047.2
18	**1043.03**	862.3	1043.2
19	**1055.46**	812.7	1056.3
20	**1048.18**	906.8	1049.1
21	**1053.94**	886.1	1054.2
22	**1058.22**	932.9	1058.4
23	**1057.56**	996.6	1059.0
24	**1057.46**	864.7	1058.6
25	**1085.44**	1011.6	1087.0
26	**1074.88**	946.8	1075.2
27	**1076.08**	895.2	1077.1
28	**-**	-	-
29	**1070.30**	1030.9	1071.9
30	**1059.61**	971.7	1061.3
31	**1046.50**	879.0	1047.0
32	**1047.09**	941.7	1047.9
33	**1047.19**	995.9	1048.3
34	**1048.25**	914.7	1049.0
35	**1055.28**	916.6	1055.7
36	**1047.03**	905.0	1049.9
37	**1066.56**	901.2	1067.1
38	**1060.37**	907.4	1061.1
39	**1059.52**	1054.0	1086.8
40	**1075.54**	1096.8	-
41	**1078.16**	879.4	1078.4
42	**1077.41**	941.3	1077.8
43	**1062.06**	977.0	1062.7
44	**1063.22**	950.3	1064.5
45	**1062.94**	978.2	1063.2

Day 52 (Add H2O)			
Box nº	80% W initial (g)	Wi (g)	Wf (g)
1	**1047.36**	1018.8	1047.9
2	**1049.94**	1022.0	1051.9
3	**1049.57**	1029.9	1064.0
4	**1056.28**	1033.4	1056.4
5	**1051.21**	1011.9	1051.4
6	**1054.76**	955.6	1056.2
7	**1071.73**	961.1	1072.1
8	**1060.65**	1031.7	1061.7
9	**1061.82**	856.9	1062.1
10	**1079.77**	972.5	1079.8
11	**1081.37**	961.4	1081.7
12	**1076.65**	967.8	1076.6
13	**1062.36**	963.2	1064.2
14	**1060.70**	996.3	1062.5
15	**1061.33**	928.0	1061.9
16	**-**	-	-
17	**1046.56**	973.6	1047.2
18	**1043.03**	935.4	1043.1
19	**1055.46**	924.5	1056.0
20	**1048.18**	913.2	1050.5
21	**1053.94**	1015.2	1054.6
22	**1058.22**	949.4	1058.8
23	**1057.56**	1000.0	1058.0
24	**1057.46**	945.1	1058.2
25	**1085.44**	1011.4	1087.6
26	**1074.88**	1007.1	1075.7
27	**1076.08**	1011.9	1076.6
28	**-**	-	-
29	**1070.30**	976.1	1072.0
30	**1059.61**	999.1	1059.7
31	**1046.50**	961.6	1046.6
32	**1047.09**	944.5	1048.6
33	**1047.19**	1031.6	1048.7
34	**1048.25**	928.4	1048.6
35	**1055.28**	948.7	1055.3
36	**1047.03**	930.0	1047.6
37	**1066.56**	984.8	1055.3
38	**1060.37**	1037.0	1061.3
39	**1059.52**	955.8	1060.0
40	**1075.54**	888.6	1075.8
41	**1078.16**	963.9	1079.2
42	**1077.41**	972.8	1078.6
43	**1062.06**	1004.5	1063.3
44	**1063.22**	935.5	1063.8
45	**1062.94**	938.3	1063.6

Day 57 (Add H_2O)			
Box nº	80% W initial (g)	Wi (g)	Wf (g)
1	**1047.36**	940.7	1047.7
2	**1049.94**	955.3	1050.7
3	**1049.57**	1023.7	1050.1
4	**1056.28**	999.6	1056.5
5	**1051.21**	862.1	1051.5
6	**1054.76**	803.8	1055.4
7	**1071.73**	778.4	1072.3
8	**1060.65**	905.6	1068.5
9	**1061.82**	1003.3	1061.9
10	**1079.77**	951.8	1079.7
11	**1081.37**	1015.7	1082.0
12	**1076.65**	1001.3	1077.5
13	**1062.36**	954.0	1062.8
14	**1060.70**	841.8	1061.2
15	**1061.33**	816.0	1062.6
16	**-**	-	-
17	**1046.56**	844.9	1047.1
18	**1043.03**	015.7	1043.2
19	**1055.46**	792.8	1055.5
20	**1048.18**	915.0	1049.3
21	**1053.94**	957.9	1054.4
22	**1058.22**	966.1	1058.5
23	**1057.56**	939.3	1062.0
24	**1057.46**	861.5	1057.8
25	**1085.44**	879.5	1085.5
26	**1074.88**	883.4	1075.1
27	**1076.08**	976.8	1079.6
28	**-**	-	-
29	**1070.30**	955.9	1070.5
30	**1059.61**	901.6	1060.0
31	**1046.50**	1019.1	1047.3
32	**1047.09**	882.1	1049.0
33	**1047.19**	943.8	1047.3
34	**1048.25**	908.8	1048.4
35	**1055.28**	1000.3	1056.0
36	**1047.03**	992.6	1047.2
37	**1066.56**	1024.7	1067.4
38	**1060.37**	880.4	1061.4
39	**1059.52**	888.5	1059.5
40	**1075.54**	923.2	1075.6
41	**1078.16**	882.0	1078.4
42	**1077.41**	958.9	1078.0
43	**1062.06**	913.4	1062.9
44	**1063.22**	994.0	1063.3
45	**1062.94**	972.1	1063.6

Day 60 (Add H2O)			
Box nº	80% W initial (g)	Wi (g)	Wf (g)
1	**1047.36**	999.0	1047.9
2	**1049.94**	977.4	1051.2
3	**1049.57**	996.9	1049.8
4	**1056.28**	1028.2	1056.5
5	**1051.21**	1029.5	1052.0
6	**1054.76**	995.3	1055.0
7	**1071.73**	957.0	1072.1
8	**1060.65**	1030.3	1060.9
9	**1061.82**	959.0	1063.2
10	**1079.77**	997.0	1079.9
11	**1081.37**	1017.6	1081.6
12	**1076.65**	812.8	1076.8
13	**1062.36**	1015.8	1062.5
14	**1060.70**	934.9	1067.7
15	**1061.33**	993.9	1061.5
16	**-**	-	-
17	**1046.56**	949.6	1046.9
18	**1043.03**	973.0	1043.2
19	**1055.46**	899.3	1’56.4
20	**1048.18**	995.3	1049.5
21	**1053.94**	907.7	1054.1
22	**1058.22**	933.2	1052.3
23	**1057.56**	948.1	1058.0
24	**1057.46**	932.9	1057.6
25	**1085.44**	959.8	1087.7
26	**1074.88**	839.6	1075.5
27	**1076.08**	1015.9	1077.1
28	**-**	-	-
29	**1070.30**	956.1	1071.1
30	**1059.61**	993.6	1059.6
31	**1046.50**	1000.2	1046.8
32	**1047.09**	1005.2	1048.5
33	**1047.19**	997.5	1047.6
34	**1048.25**	878.8	1048.4
35	**1055.28**	960.6	1055.4
36	**1047.03**	1008.1	1050.6
37	**1066.56**	926.9	1067.0
38	**1060.37**	956.7	1061.0
39	**1059.52**	934.6	1059.7
40	**1075.54**	1055.7	1075.7
41	**1078.16**	1028.1	1078.7
42	**1077.41**	993.7	1077.8
43	**1062.06**	996.0	1062.2
44	**1063.22**	984.3	1063.5
45	**1062.94**	1020.6	1063.3

Day 63 (Add H2O)			
Box nº	70% W initial (g)	Wi (g)	Wf (g)
1	**916.44**	1020.1	-
2	**918.69**	1019.2	-
3	**918.37**	1030.8	-
4	**924.25**	1018.7	-
5	**919.99**	949.9	-
6	**922.92**	1004.0	-
7	**937.76**	773.0	938.7
8	**928.07**	989.8	-
9	**929.10**	1027.4	-
10	**944.80**	982.0	-
11	**946.20**	995.8	-
12	**942.07**	1010.4	-
13	**929.57**	962.8	-
14	**928.12**	915.5	929.2
15	**928.66**	939.3	.
16	**-**	-	-
17	**915.74**	898.6	916.2
18	**912.65**	911.1	912.8
19	**923.52**	950.6	-
20	**917.16**	990.2	-
21	**922.20**	1029.0	-
22	**925.95**	938.0	-
23	**925.37**	1044.8	-
24	**925.28**	1003.9	-
25	**949.76**	972.3	-
26	**940.52**	962.1	-
27	**941.57**	1015.7	-
28	**-**	-	-
29	**936.52**	921.6	936.7
30	**927.16**	957.4	-
31	**915.69**	1002.2	-
32	**916.20**	983.4	-
33	**916.29**	966.2	-
34	**917.22**	953.2	-
35	**923.37**	977.3	-
36	**916.15**	918.2	-
37	**933.24**	972.3	-
38	**927.82**	919.5	928.2
39	**927.08**	964.0	-
40	**941.10**	957.4	-
41	**943.39**	1006.1	-
42	**942.73**	957.7	-
43	**929.31**	895.9	930.1
44	**930.32**	983.0	-
45	**930.08**	945.1	-

Day 67 (Add H_2O)			
Box nº	70% W initial (g)	Wi (g)	Wf (g)
1	**916.44**	944.4	-
2	**918.69**	889.8	919.7
3	**918.37**	982.6	-
4	**924.25**	983.0	-
5	**919.99**	894.8	920.6
6	**922.92**	895.2	923.3
7	**937.76**	901.2	938.1
8	**928.07**	855.6	928.7
9	**929.10**	984.8	-
10	**944.80**	819.2	945.2
11	**946.20**	941.4	946.4
12	**942.07**	884.0	942.2
13	**929.57**	898.4	929.8
14	**928.12**	827.4	928.9
15	**928.66**	917.1	928.8
16	**-**	-	-
17	**915.74**	884.3	916.2
18	**912.65**	847.3	912.9
19	**923.52**	866.2	923.7
20	**917.16**	887.8	917.6
21	**922.20**	839.7	922.7
22	**925.95**	864.6	926.3
23	**925.37**	863.8	925.9
24	**925.28**	890.9	925.5
25	**949.76**	878.8	950.1
26	**940.52**	909.2	940.8
27	**941.57**	940.2	941.9
28	**-**	-	-
29	**936.52**	911.3	936.7
30	**927.16**	814.8	927.3
31	**915.69**	889.5	916.3
32	**916.20**	883.4	916.7
33	**916.29**	903.7	917.2
34	**917.22**	874.9	917.8
35	**923.37**	876.8	925.1
36	**916.15**	884.9	917.1
37	**933.24**	889.9	933.7
38	**927.82**	879.4	928.1
39	**927.08**	867.8	929.2
40	**941.10**	896.9	941.1
41	**943.39**	889.8	943.8
42	**942.73**	911.4	943.1
43	**929.31**	899.0	929.8
44	**930.32**	923.1	931.8
45	**930.08**	894.3	930.6

Day 70 (Add H_2O)			
Box nº	70% W initial (g)	Wi (g)	Wf (g)
1	**916.44**	911.1	916.6
2	**918.69**	848.8	920.2
3	**918.37**	935.5	-
4	**924.25**	963.7	-
5	**919.99**	881.6	920.1
6	**922.92**	826.4	923.6
7	**937.76**	883.6	938.0
8	**928.07**	897.8	928.4
9	**929.10**	911.3	929.7
10	**944.80**	878.0	944.9
11	**946.20**	824.0	947.3
12	**942.07**	925.7	942.2
13	**929.57**	889.9	930.4
14	**928.12**	853.5	929.9
15	**928.66**	646.9	929.1
16	**-**	-	-
17	**915.74**	754.3	916.3
18	**912.65**	842.5	913.2
19	**923.52**	815.1	924.0
20	**917.16**	851.6	918.5
21	**922.20**	862.8	923.0
22	**925.95**	880.6	927.3
23	**925.37**	870.0	926.8
24	**925.28**	860.3	925.6
25	**949.76**	880.9	949.7
26	**940.52**	809.9	940.7
27	**941.57**	839.2	942.0
28	**-**	-	-
29	**936.52**	879.4	936.8
30	**927.16**	905.4	929.0
31	**915.69**	886.2	916.9
32	**916.20**	877.6	917.5
33	**916.29**	885.7	917.1
34	**917.22**	876.9	917.5
35	**923.37**	891.3	924.2
36	**916.15**	870.8	916.8
37	**933.24**	882.7	934.7
38	**927.82**	872.6	928.3
39	**927.08**	888.7	928.0
40	**941.10**	882.7	941.7
41	**943.39**	902.2	944.3
42	**942.73**	882.5	944.0
43	**929.31**	893.6	931.9
44	**930.32**	895.7	931.1
45	**930.08**	904.3	930.4

Day 73 (Add H2O)			
Box nº	70% W initial (g)	Wi (g)	Wf (g)
1	**916.44**	855.1	917.0
2	**918.69**	871.3	919.9
3	**918.37**	885.1	918.6
4	**924.25**	953.8	-
5	**919.99**	837.0	920.4
6	**922.92**	888.5	923.1
7	**937.76**	776.2	939.3
8	**928.07**	870.6	929.0
9	**929.10**	902.0	930.2
10	**944.80**	811.6	945.5
11	**946.20**	879.7	947.6
12	**942.07**	895.1	942.6
13	**929.57**	880.6	930.8
14	**928.12**	670.9	929.0
15	**928.66**	849.0	929.3
16	**-**	-	-
17	**915.74**	800.4	917.2
18	**912.65**	840.5	913.5
19	**923.52**	828.9	926.6
20	**917.16**	853.2	918.1
21	**922.20**	823.1	922.7
22	**925.95**	861.2	928.7
23	**925.37**	860.2	926.2
24	**925.28**	859.9	926.0
25	**949.76**	897.0	950.3
26	**940.52**	900.7	941.0
27	**941.57**	749.9	952.9
28	**-**	-	-
29	**936.52**	887.4	937.8
30	**927.16**	786.6	927.8
31	**915.69**	891.8	916.3
32	**916.20**	878.7	917.5
33	**916.29**	893.4	916.6
34	**917.22**	871.9	918.2
35	**923.37**	876.5	924.7
36	**916.15**	877.1	916.7
37	**933.24**	923.7	936.1
38	**927.82**	862.7	929.0
39	**927.08**	883.8	927.5
40	**941.10**	897.1	942.6
41	**943.39**	898.1	944.0
42	**942.73**	888.2	944.7
43	**929.31**	902.3	929.7
44	**930.32**	903.1	933.6
45	**930.08**	896.9	931.9

Day 78 (Add H2O)			
Box nº	70% W initial (g)	Wi (g)	Wf (g)
1	**916.44**	855.6	916.1
2	**918.69**	811.3	918.5
3	**918.37**	682.1	918.8
4	**924.25**	931.8	-
5	**919.99**	877.9	919.2
6	**922.92**	620.5	922.8
7	**937.76**	810.9	937.3
8	**928.07**	741.5	928.0
9	**929.10**	728.0	924.4
10	**944.80**	849.3	944.5
11	**946.20**	833.5	946.3
12	**942.07**	752.3	942.6
13	**929.57**	851.6	929.1
14	**928.12**	828.4	928.3
15	**928.66**	791.8	928.3
16	**-**	-	-
17	**915.74**	727.3	915.7
18	**912.65**	844.0	912.7
19	**923.52**	854.1	923.9
20	**917.16**	847.9	917.8
21	**922.20**	758.6	922.6
22	**925.95**	867.8	925.3
23	**925.37**	905.6	925.8
24	**925.28**	857.0	925.2
25	**949.76**	874.2	949.6
26	**940.52**	735.3	940.8
27	**941.57**	883.0	941.4
28	**-**	-	-
29	**936.52**	880.3	936.6
30	**927.16**	762.8	927.8
31	**915.69**	877.2	915.3
32	**916.20**	887.1	916.4
33	**916.29**	874.8	916.0
34	**917.22**	871.9	917.9
35	**923.37**	873.5	923.9
36	**916.15**	875.8	916.9
37	**933.24**	875.3	933.2
38	**927.82**	871.8	927.1
39	**927.08**	867.5	927.7
40	**941.10**	874.8	941.4
41	**943.39**	880.3	943.0
42	**942.73**	880.7	942.8
43	**929.31**	890.3	929.5
44	**930.32**	899.2	930.9
45	**930.08**	894.7	930.5

Day 81 (Add H2O)			
Box nº	70% W initial (g)	Wi (g)	Wf (g)
1	**916.44**	900.0	913.7
2	**918.69**	613.4	919.1
3	**918.37**	906.0	918.6
4	**924.25**	922.8	924.6
5	**919.99**	850.6	921.5
6	**922.92**	884.5	923.1
7	**937.76**	852.9	938.9
8	**928.07**	857.7	928.7
9	**929.10**	891.9	929.6
10	**944.80**	912.1	945.0
11	**946.20**	901.8	946.8
12	**942.07**	899.4	942.2
13	**929.57**	872.5	929.8
14	**928.12**	811.0	929.5
15	**928.66**	881.8	931.0
16	**-**	-	-
17	**915.74**	831.4	916.3
18	**912.65**	861.1	913.1
19	**923.52**	768.1	924.1
20	**917.16**	864.0	917.3
21	**922.20**	863.5	923.1
22	**925.95**	862.7	927.0
23	**925.37**	911.2	930.0
24	**925.28**	889.4	925.7
25	**949.76**	901.3	950.3
26	**940.52**	855.6	941.7
27	**941.57**	850.0	942.0
28	**-**	-	-
29	**936.52**	899.8	937.1
30	**927.16**	813.3	927.6
31	**915.69**	903.7	918.1
32	**916.20**	876.9	916.6
33	**916.29**	895.7	917.1
34	**917.22**	883.4	919.7
35	**923.37**	909.6	924.2
36	**916.15**	882.0	917.6
37	**933.24**	879.5	934.5
38	**927.82**	894.0	928.4
39	**927.08**	887.4	929.8
40	**941.10**	885.0	941.8
41	**943.39**	892.7	943.9
42	**942.73**	894.5	942,9
43	**929.31**	892.1	930.4
44	**930.32**	901.4	930.6
45	**930.08**	899.2	930.3

Day 84-87 (End of test: Drying till constant mass)			
Box nº	W day 84 (g)	W day 86 (g)	W day 87 (g)
1	833.4	533.3	530.2
2	864.2	546.9	543.5
3	882.5	561.3	558.1
4	874.9	552.4	546.5
5	717.7	535.5	533.0
6	891.6	528.0	525.0
7	765.0	539.3	536.7
8	850.3	529.2	526.8
9	842.6	545.6	541.2
10	898.9	534.2	531.6
11	887.5	545.6	540.5
12	861.7	552.3	539.1
13	884.7	541.3	534.6
14	900.0	549.9	537.7
15	865.9	547.3	541.5
16	-	-	-
17	726.8	726.7	723.8
18	855.5	838.9	837.0
19	767.1	766.6	762.7
20	852.1	845.6	843.9
21	808.1	736.2	733.6
22	894.6	856.7	855.0
23	888.8	858.8	856.3
24	857.4	856.2	853.9
25	902.5	870.9	869.8
26	878.5	729.7	726.9
27	825.1	732.5	731.6
28	-	-	-
29	879.1	875.9	873.5
30	851.4	752.4	751.2
31	878.8	871.7	/
32	877.9	874.5	/
33	881.7	875.2	/
34	891.1	873.0	/
35	882.8	874.4	/
36	871.4	868.5	/
37	883.9	873.2	/
38	870.1	865.0	/
39	881.8	866.1	/
40	873.6	874.9	/
41	878.0	876.4	/
42	890.1	880.9	/
43	901.9	889.0	/
44	895.2	890.6	/
45	901.5	894.6	/

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