Tampere University of Applied Sciences

BIODEGRADABILITY TEST FOR PACKAGING MATERIALS

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ABSTRACT

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Biodegradability Test for Packaging Materials
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Conventional plastics that are used in packages do not biodegrade and cause environmental pollution. Because of this there is a need to replace these with biodegradable packages. Therefore, there has been rise in biodegradable packaging materials and standards to check the biodegradability.

This thesis defines biodegradation and composability, and standards related to them. The difference and misunderstanding between biodegradable and compostable is discussed. Also, biodegradable packaging materials and their trends are also presented.

A design of a test environment for checking the biodegradability of packaging materials is presented according to the standard EN14046 which is followed by the step by step process on doing the experiment. A small laboratory test is conducted to have a basic idea of doing a biodegradability test. The information is essential in constructing a real test environment and doing the test which maybe a next project.

Key words: biodegradable packaging, biodegradable, biodegradation, biodegradability, compostable, bio-plastics, biodegradability test, biodegradability standards

CONTENTS

1 INTRODUCTION

The concern in biodegradability of packages have been rising in present days because of all the problems that the packaging industry has caused. The main problem is that the non-degradable packages have surrounded our planet and the packages will stay there for centuries because of the non-decaying properties of the material. Packages are artificial and when it reaches the natural zone, the animals and in general the environment tries to consume which causes many defaults in our natural pre-existing environment. The packages and plastics that are released into the environment may start to end up in the plates of future generation.

There is a huge business of packaging materials and products, which are designed to influence the costumers to buy their products and catch their attention. The companies of the packaging related business have two main objectives which are to attract their customers and to make it very cost effective. The environmental sustainability side is often on their least priority in the business. (Wu, 2014)

Plastics, which are one of the main packaging problems around the world had been leading the world to major environmental problems. In 2015, the annual global production of plastics was more than 400 million metric tons of which more than third of the production was for packaging sector. In the same year, the waste generated from the global plastics production was about 300 million metric tons of which half of the waste was from the packaging sector. (Geyer et al, 2017)

Plastics are made by the process of polymerization. Polymerization is the process of making plastics combining small molecules into long chains. But sometimes the chains maybe incomplete and the additives and catalyst used to build the plastics material may be released from its surface. It may lead to health and environmental problems, if the plastics are used as wraps for foods. Also, it may end up in marine environment and maybe consumed by living organisms. PVC is the third mostly produced plastic in the world and the monomer of PVC has been found to be carcinogenic also the additive used in PVC is found to be harmful for the reproductive system. The plastics that end up in marine environment are consumed by the animals which can cause pathological and impede reproduction. (Nakatani, 2018)

Research has been done in order to decrease the problems of packaging all around the world. The research done in biodegradable and bio-based polymers are very likely to solve the environmental problems replacing the non-biodegradable packages. The biodegradable and bio-based substances such as PLA, PHB, etc are suitable to produce similar products like conventional plastics, which could replace the plastics markets in the future. (Nakatani, 2018).

As the increase in biodegradable packages increase, there is a need in controlling the biodegradability of those materials. As early as 1990s in US there has been various critics and false claims on the biodegradability of plastics, so the urgent need for bio-degradable standards were advertised (Wilde, 2005). After that there has been various associations and companies responsible for publishing and maintaining the standards on biodegradability.

The thesis contains research on the biodegradation and biodegradable packaging products briefly and their trends on the market. The term biodegradable and compostable, which have caused confusion among the public, has been made clear by explaining the terms and differentiating between them. The main standards used to evaluate the biodegradability of packaging materials are also introduced and categorised systematically. Additionally, a different approach on evaluating the biodegradability was experimented by using the oxitop® device in the laboratory of TAMK to learn the things that should be considered in practice when designing an actual test. Finally, a brief process on designing the test environment for biodegradability test according to the standard EN 14046 is presented.

2 AIMS

The main aims of this thesis are:

To differentiate between biodegradable and compostable packages and make the terms clear to the reader.

To find the relevant standard that are necessary to evaluate the biodegradability of plastics and packages on different mediums and finally finding the specific standards needed for this research.

To conduct a laboratory experiment based on oxitop® device to understand bi-degradability better.

Briefly introducing the process in the construction of a test environment according to the standard EN14046 and suggesting the steps on conducting the test.

3 PACKAGING MATERIALS

After the beginning of twentieth century, there has been vast change in lifestyles of the people in developed countries, which has contributed in significant rise in small packages or more packaging products. Some changes such as reduction in family size, more work schedule, ownership of fridges and microwaves, rise in income, travel, etc contribute to the use of more packaged food and materials. (Emblem and Emblem, 2013)

There is also a huge competition in business market, which plays very crucial role in packaging material quality and quantity. On the modern supermarket place, where there are many products and costumer with very limited time, are attracted to the packages, which are easily to distinguish and are also attractive in its structure and design. The Marks and Spencer was one of the first company to introduce the concept of having secondary packaging material so that it is easier and faster to handle the big bunch of products altogether. Also, the need of barcodes and faster checking out of modern market require secondary packaging or more packaging in today's modern business era. (Emblem and Emblem, 2013)

Protecting the product is one of the most important reason and need to do the packaging. Especially when the product is to be delivered or shipped in long distance place with hard climatic conditions, there is a need for good packages around the products to protect them. Also, health and hygiene are considered very crucial for the business and costumer which brings another need for the packages and packaging products to be healthy and keep the products hygienic. (Emblem and Emblem, 2013)

3.1 Levels of Packaging

Primary packaging, the first stage of packaging, which is in direct contact with the material, product or food whose main aim is to store, preserve and inform the product for the costumer. This should be very crucial to the health and hygiene if it's related to food. These packages usually end up in the dustbin of normal people. For example: beer cans, chips bag, shop bottles, etc. The second stage of packaging, secondary packaging which serves as the storage of primary items in a group to make it easier and faster for moving and selling. This includes mostly corrugated cardboard materials with the display of the brand and sometimes acts as a bag for multiple products. These products are mostly for the supermarkets. For example: beer carry packs, etc. Finally, Tertiary packaging, big packaging which include a group of secondary packaging which are designed especially for protection, handling, shipping or moving the products from one place to another. These products are rarely seen by the costumer and mostly handled by the logistics. (Saxon, 2017).

4 BIODEGRADABLE PACKAGING PRODUCTS AND MATERIALS

The world has already witnessed various problems that the non-degradable packaging products made of plastic have brought upon us. So, in the present days the need for bio-degradable packaging products is increasing with increasing in packaging especially food packaging which has the largest demand of bio-degradable products. Bio-degradable materials are mostly composed of natural renewable resources or bio-based materials. Biodegradable packaging products are divided into bio-plastics and fibre-based packaging.

4.1 Bio-plastics

Out of 335 million tonnes of plastics that are produced annually around the globe, bio-plastics represent about one percent. The production of bio-plastic is increasing with its demand, which is expected to increase from 2.11 million tonnes in 2018 to approximately 2.62 million tonnes in 2023. Packaging field is the largest consumer of bio-plastics and about 65% of the total is used for packages. (European Bioplastics, 2018)

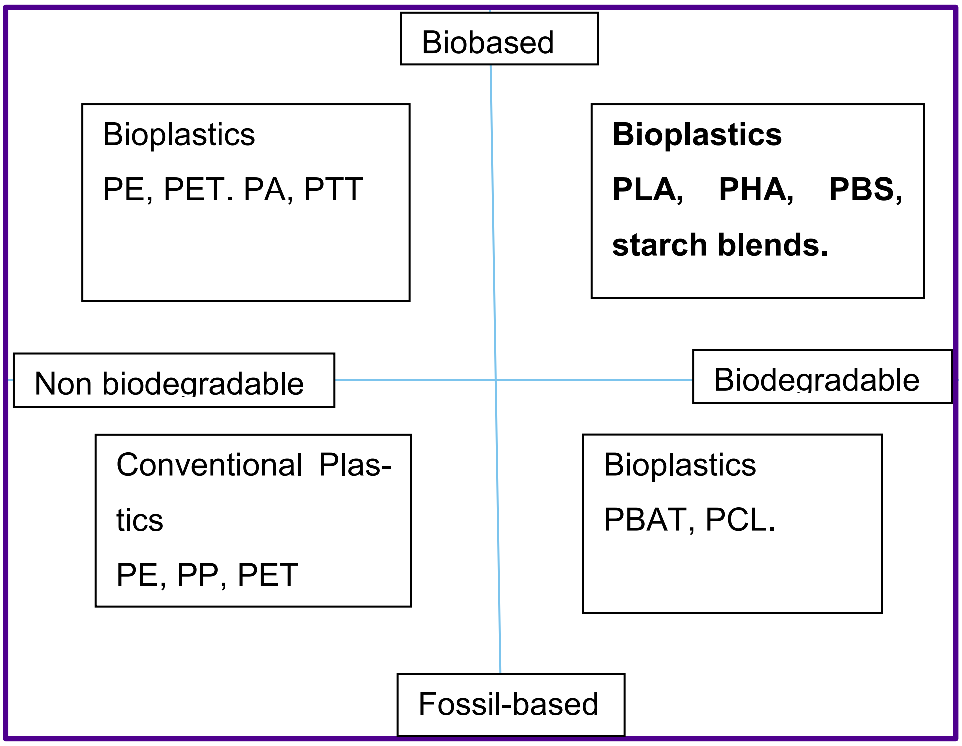

Figure 1 Different categories of plastics transcribed from; (European Bioplastics n.d)

But, not all the bio-plastics are considered biodegradable. So, careful understanding is needed in biodegradation when the term bio-plastic is used. The below figure is helpful for understanding the different sources of plastics and their properties.

From the above figure 2, the bio-plastics can be divided into biodegradable and non-biodegradable bioplastics.

4.1.1 Biodegradable bio-plastics

Biodegradable bio-plastics are categorised based on their ability for organic recycling and it can be tested and possibly certified compostable, if approved by international standards such as EN 13432.

From the chart “Global production capacities bioplastics” in the website of European bioplastics, it’s clear that the biodegradable bio-plastics are produced not even 50% of that of total bio-plastics produced. Bio-plastic trend is increasing in the coming years where bio-degradable bio-plastics are expected to be equal in production with non-bio-degradable bio-plastics. PLA based bio-plastics are the most used biodegradable bioplastics in the world, which is followed by starch blends. It is expected that PLA materials will overtake starch blends in future. With the increase in demand for bio-degradable bioplastics, the global production of bio-degradable bio-plastics is expected to increase by approximately 25% by 2023. (European Bioplastics, 2018)

European Bioplastics classified biodegradable bio-plastics into two categories based on the extraction of the raw materials which are; bio-based biodegradable bioplastics and fossil-based biodegradable bioplastics which are described below briefly.

4.1.2 Bio-based biodegradable bioplastics (PLA, PHA, PBS, starch blends)

Some of the bio-based biodegradable bioplastics are introduced briefly below.

PLA

An aliphatic polyester extracted from renewable resources, such as corn starch, tapioca roots, sugarcane, etc and is soluble in chlorinated solvents, hot benzene, tetrahydrofuran and dioxane. Mostly Poly-L.lactide (PLLA) and poly-D-lactide(PDLA) are blended together to improve performance which increases the crystallinity and temperature stability .(Kumbar et al, 2014). In an aerobic controlled composting condition, PLA which consists of Lactic acid is bio-degraded into water and CO2 (Mahalik and Nambiar, 2010)

PLA is used as Packaging material because of its low glass transition temperature, low thermal stability, high brittleness and low crystallization rate and is used in rigid containers for water, juice and yogurt (Mallegni et al, 2018).

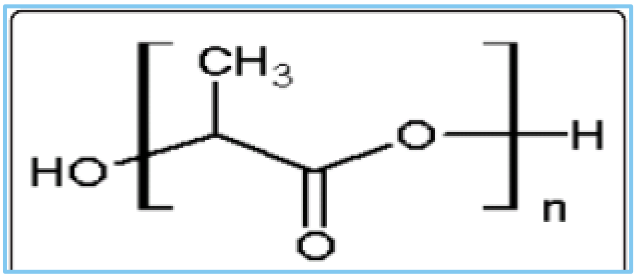

Figure 2 Chemical Structure of Polylactic acid (PLA) (Mahapatro et al, 2011)

Biofutura, a company from the Netherlands is making many products such as coffee cups, bottles, straws from PLA. Another German company Huhtamaki is also making products based on PLA.

PHA

Poly(hydroxtalkanoate)s are considered as eco-friendly and bio-degradable polymeric materials, which is produced in the cell of bacteria by fermentation with sugar or lipids to form linear polyester which is harvested and made into plastic pellets. As there are variety of PHAs, the properties of the material are affected by its chemical structure. Some of popular PHAs in the market are poly(3-hydroxybutyrate) (P3HB), poly(4-hydroxybutyrate) (P4HB) and poly(3-hydroxybutyrate-co-3-hydroxyvalerate) (PHBV). (Greene, 2014)

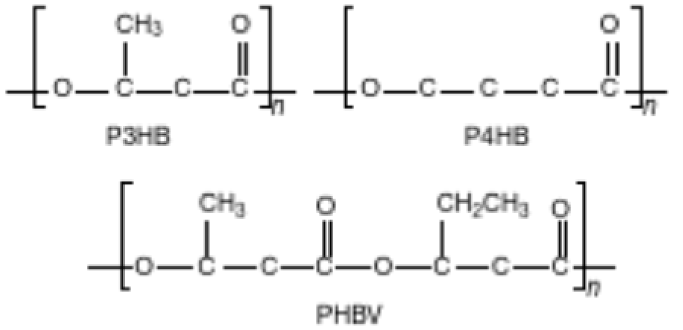

Figure 3 Chemical structure of Structures of poly(3-hydroxybutyrate) (P3HB), poly(4-hydroxybutyrate) (P4HB) and poly(3-hydroxybutyrate-co-3-hydroxyvalerate) (PHBV). (Greene, 2014)

Some of the companies and manufacturer of PHA are Kaneka from Japan and Danimer Scientific from USA.

Starch polymers

Extracted from starch of potato, corn, wheat, cassava or other renewable resources. Starch usually called as thermoplastic starch TSP is biodegradable when all its additives are also biodegradable. Biodegradable TSP include starch, aliphatic polyester, glycerol and water. Starch is a blended combination of the two chemicals: amylose and amylopectin shown below. (Greene, 2014)

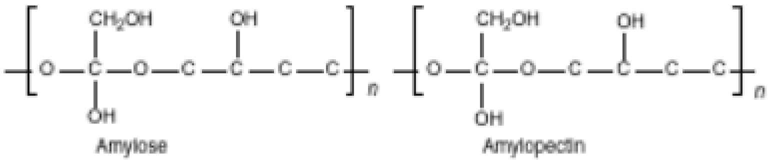

Figure 4 Molecular structure of amylose and amylopectin. (Greene, 201)

Indochine Bio plastiques from Malaysia, Agrana Stärke from Austria are some companies manufacturing starch polymer and starch polymer-based products.

4.1.3 Fossil-based biodegradable bioplastics (PBAT, PCL)

Poly (butylene adipate-co-terephthalate), PBAT is used for making biodegradable bio-plastics and many times as binding agent or composites with other biodegradable materials. PBAT are fully biodegradable and is also considered compostable which have higher tensile strength and higher elongation at break than most of other biodegradable polymers such as PLA and PBS and are comparable with the properties of low-density PE. PBAT has been found more improved in mechanical properties when used as composites with other substances rather than using pure PBAT. (Ferreira et al, 2017)

BASF, a company from Germany is making PBAT and PBAT blends materials. Jinhui Zhaolong High Tech, a Chinese company is also making PBAT materials.

PCL, Polycaprolactone is partially crystalline compound with melting point of 59-64°C, tensile strength of 16 MPa and tensile modulus of 0.4 Gpa, takes more time to degrade (2-5 years) therefore, it can be used to make products, which require longer degradation period (Cameron and Kamvari-Moghaddam, 2012). POLI-FILM GmbH, a German company manufactures PCL based materials.

4.2 Fibre-based packaging

Fibre based materials or pulp material are produced mostly from wood and sometimes produced from agricultural residue such as straws (of wheat, rye, barley, rice), sugarcane bagasse, cotton, flax, bamboo, corn husks, etc. These materials are made into pulps by mechanical, Chemical or combination process, which is the building blocks of fibre-based materials. They are used in variety of packages such as vegetable parchment paper, whiteboard, linerboard, food board, carton board, folded carton board, moulded pulp, packing electronics, household items, shipping boxes, etc. (Crevel, 2016).

There is a huge market in Fibre-based packaging all around the world. Average global production of fibre-based packaging in 2011-2013 was 213 million metric tons which accounted for $261 billion value where China and US were the major producers of fibre-based packaging which produced almost half of the total fibre production. The Production is expected to increase to 500 million tonnes by 2020. (Fibre-based packaging, 2017)

Ecopulp, a company from Finland is making fibre-based products for industries and individuals. Another company from UK, which is making fibre based corrugate and pulp in supermarkets.

5 BIODEGRADATION

Biodegradation is the decaying of organic materials, which is performed by microorganisms present in earth which transforms organic complex compounds into less complex inorganic compounds also toxic contaminants into less toxic or no toxic substances. As bio-degradation is biochemical reaction, which is balanced by microorganisms, where an organic compound is oxidised by electron acceptor, which itself is reduced. In general, the microbial population number is between 1×10^5 to 1×10^7 cell per gram of dry weight. (Eskander and Saleh, 2017). According to Eskander and Saleh (2017), there are some requirements for biodegradation, which are as follows.

1. The presence of the appropriate microorganisms,
2. An energy source such as organic moiety that is used by the organisms for cell maintenance and growth. Organic moiety are functional groups of compounds such as CH_3, Cl that affect biodegradation (Raina and Terry, 2015).
3. A carbon source,
4. An electron acceptor such as O_2, $NO_3{}^{-}$, $SO_4{}^{-2}$, CO_2
5. Nutrients as nitrogen, phosphorus, calcium, magnesium, iron, trace elements, and
6. Acceptable environmental conditions e.g., Appropriate temperature, pH, salinity levels.

Aerobic and anaerobic biodegradation

The process of bio-degradation when oxygen is present, and the microorganisms use oxygen as electron acceptor to oxidize part of carbon of the organic compound into carbon dioxide and another part to create new cell and oxygen is converted into water is called aerobic biodegradation. The by-products in aerobic biodegradation are carbon dioxide, water and an increased population of microorganisms. (Eskander and Saleh, 2017)

The process of bio-degradation takes place when oxygen is not present, the substitute of oxygen includes nitrate, sulphate, iron, manganese or CO_2, which are used by microorganisms. These compounds act as electron acceptor and the by-products include nitrogen gas, hydrogen sulphide, reduced form of metals, and methane depending on the electron acceptor. (Eskander and Saleh, 2017)

5.1 Oxitop® device and process:

Oxitop® device is manometric measurement of oxygen consumption where CO_2 produced is absorbed by an absorbing agent, which is some alkaline agent. Oxygen is consumed by the biodegradable organic substances in aerobic condition to produce carbon dioxide. More accurately, the oxygen is consumed by the organisms and same time carbon dioxide is released. Inside a closed jar with large free space, O_2 present in the free space is consumed in the soil and the release of CO_2 which is approximately equimolar is consumed by absorbing alkaline substance. Due to this, the pressure change is only related with the oxygen present in the free space. The pressure inside the jar is negative. The following equation shows the biodegradation process in glucose which is the basis of all biodegradation process. (Platen and Wirtz, 1999)

$$C_6H_{12}O_6 + 6\ O_2 \rightarrow 6\ CO_2 + 6\ H2O \qquad (1)$$

The following equation for general gas equation gives the relation between the change in pressure and oxygen consumption in soil. (Platen and Wirtz, 1999)

$$\Delta p = \Delta n * Re * T * V^{-1} \qquad (2)$$

NaOH is considered best for soil respiration, which reacts with CO_2 as follows:

$$CO_2 + 2\ NaOH \rightarrow Na_2CO_3 + H_2O \qquad (3)$$

In the above equation carbon dioxide is absorbed by NaOH to form sodium carbonate and water.

5.2 Compostability

Compostability of a material is the ability to bio-degrade with the help of bio-organisms in a specific period in a controlled environment. (Science Learning Hub, 2014). “A material is considered compostable if at least 90% of it biodegrades in a specified test within 6 months.” (Piergiovanni and Limbo, 2016).

Composting is increased biodegradation process and if any material undergoes composting than it should not only biodegrade but also should be part of compost that can be used and provide nutrients in the soil (Brussels, 2018). The composting is the process where the biodegradation process of organic material is accelerated by providing optimum environment for the decomposing microorganisms (Composting n.d). The end-product of the composting process is rich in nutrients and it can be used in growing crops, trees and plants (Ross, 2018). According to EPA, there are five main aspects that should be controlled during the composting process which are feedstock and nutrient balance, particle size, moisture content, oxygen flow and temperature (EPA, 2018).

European Standard EN 13432, defines that the compostable materials must have the following characteristics:

1. Biodegradability value in a controlled composting condition in standard method of carbon dioxide evolution should be at least 90% in less than 6 months.

2. Disintegration test from a standard method should be 10% of the initial mass for 3-month experiment.

3. There should not be any negative impact on the quality of compost.

4. Heavy metals should be according to the defined values in the norm.

If any material that is biodegradable can also break any of the requirement from the European Standard EN 13432. Hence, proper test by a recognised standard is needed to prove composability of a material. From the above discussion we can also say that all the compostable materials can be termed as biodegradable, but all the biodegradable materials cannot be termed as compostable because of the specific time frame and condition required for obtaining the compostable properties.

The European bioplastics is one of the organisations that is responsible for providing the trademark for compostable materials in many countries in Europe. The certification proves that the product is industrially compostable according to European Standard EN 14342. The logo is called the seeding logo and looks identical to the figure below.

compostable

Figure 5 Compostable seeding logo. Permission taken for the use of logo (European bioplastics, 2016)

5.3 Biodegradable and compostable

The term biodegradable has been misused by various companies and the term is misleading among the public. Technically, all the products will bio-degrade at the certain point, so the term biodegradable for packaging products is pointless. So specific time frame should be given to specify the actual biodegradability of the material. Even the petroleum-based plastics can bio-degrade after hundreds of years and leave harmful products (What's the differences between biodegradable and bio-based plastics? 2017). For any product or material to be biodegradable, the time frame, the level of biodegradability and the required condition should be taken care and it is not logical to claim biodegradability without any specification of standard (European bioplastics n.d). According to Maarten van der Zee (2014), the term biodegradation has different meaning in different fields of study. In the field of medicine, degradation of drugs into macromolecules and in field of environment, fragmentation, loss of mechanical properties and degradation through the action of living organisms has been all termed as biodegradation (Zee, 2014).

On the other hand, there are oxo-plastics or oxo-degradable plastics, which are causing the same confusion in the public. "Oxo-plastics or oxo-degradable plastics are conventional plastics which include additives to accelerate the fragmentation of the material into very small pieces, triggered by UV radiation or heat exposure". Although there has been claims that the plastics after undergoing the fragmentation into small pieces and they will biodegrade gradually without leaving toxic chemicals, but the research suggests that the oxo-degradation will lead to micro-plastic which will eventually end up in food chain. (Brussels, 2018).

Based on the above research, the differences between biodegradable and compostable can be more simplified in the table below.

Table 1 Difference between biodegradable and compostable.

Biodegradable	Compostable
All the biodegradable materials are not compostable.	All the compostable materials can be also called biodegradable.
All the material in the world are technically biodegradable including petroleum-based plastics, metals, etc.	Only a small number of materials are compostable which should follow a frame work of internationally recognised standard such as EN-14342.
Biodegradation occurs in natural environment.	Composting is done under a controlled condition of moisture, heat, etc.
Biodegradation of a material does not necessarily mean a specific time frame.	Any material to be compostable should undergo testing and should meet frame work of international standards which also have fixed time frame.
The residue of the biodegradable material may be toxic to the environment.	The residue of the compostable materials can be used as compost and is non-toxic to the environment.

6 STANDARDS FOR TESTING BIODEGRADABILITY

The standards for checking biodegradability can be found in Finnish standards association (SFS) page. SFS is the standard that is adopted or developed in Finland. EN is European standard that is adopted or developed in the European Committee for standardization (CEN) or CENELEC and ISO is international standard developed in ISO.

There are many standards if we go outside our range, but we are concerned about the standards that are adopted by SFS, CEN and OECD because of our region and location and relevance. Some of the well-established standards that are not mentioned are (ASMT), (DIN) the German Deutches Institute für Normung, Japan’s Biodegradable Plastic Society (BPS).

Packaging and packaging waste directive (94/62/EC):

The Packaging and packaging waste directive was purposed on 09.09.1993 and entered into force in 31.12.1994 by The European Parliament and of the Council (Farmer, 2012). The directive was purposed to reduce and recover the packaging waste and match the same process and maintain the same regulation all over EU. The directive has been amended and revised several times; the last amendment of the directive was on 29 April 2015. (Packaging and Packaging Waste, 2018)

Some of the important standards are listed in table below based on the medium of the experiment process.

Table 2 Standards based on soil medium.

1)	ISO 17556-2012 Plastics - Determination of the ultimate aerobic biodegradability of plastic materials in soil by measuring the oxygen demand in a respirometer or the amount of carbon dioxide evolved.	Incubated at 20-25°C in a respirometer. Duration maximum 6 months. O_2 or CO_2 is measured.
2)	OECD: 304A Inherent Biodegradability in Soil.	Biometer flask apparatus. Alkali absorption and liquid scintillation counting. Incubated at 22°C ± 2°C. Maximum 64 days. CO_2 is measured.
3)	OECD: 307 Aerobic & Anaerobic Transformation in Soil	Incubated in the dark in biometer-type flasks or in flow-through systems under controlled laboratory conditions (at constant temperature and soil Moisture) for 120 days.

Table 3 Standards based on composting

1)	SFS-EN 14045 *Packaging. Evaluation of the disintegration of packaging materials in practical oriented tests under defined composting condition.*	pilot-scale aerobic composting test under defined conditions with bio waste and compost for 12 weeks. Disintegration is measured by sieving of the compost and the calculation of a mass balance.
2)	SFS-EN 14046 *Packaging. Evaluation of the ultimate aerobic biodegradability of packaging materials under controlled composting conditions. Method by analysis of released carbon dioxide.*	Ultimate aerobic biodegradability of packaging materials based on organic compounds under controlled composting conditions. CO_2 is measured for calculating degree of biodegradation.
3)	SFS-EN 13432 *Packaging. Requirements for packaging recoverable through composting and biodegradation. Test scheme and evaluation criteria for the final acceptance of packaging.*	The standard provides frame work to support organic recovery and the composability of plastics.
4)	SFS-EN 14995 *Plastics. Evaluation of composability. Test scheme and specifications.*	Provides framework to support organic recovery and the composability of plastics. The standard is identical with SFS-EN 13432.
5)	SFS-EN ISO 20200 *Plastics. Determination of the degree of disintegration of plastic materials under simulated composting conditions in a laboratory-scale test*	Determining degree of disintegration in laboratory scale. Time period is 90 days in air circulation oven at 25°C ± 2°C. Disintegration is measured by sieving of the compost and the calculation of a mass balance.
6)	SFS-EN ISO 14855-1 *Determination of the ultimate aerobic biodegradability of plastic ma-*	Solid-phase respirometry test system. Controlled condition and constant temperature of 58 °C ± 2 °C for

	terials under controlled composting conditions. Method by analysis of evolved carbon dioxide. Part 1: General method	maximum 6 months. Biodegradability is determined by CO_2 production. Disintegration and loss of mass is also evaluated.
7)	SFS-EN ISO 14855-2 *Determination of the ultimate aerobic biodegradability of plastic materials under controlled composting conditions. Method by analysis of evolved carbon dioxide. Part 2: Gravimetric measurement of carbon dioxide evolved in a laboratory-scale test.*	The controlled condition is same as of SFS-EN ISO 14855-1:en, the difference is that this standard provides gravimetric setting for small-scale experiment in annex A and B of the standard.

Table 4 Standards based on aqueous medium.

1)	SFS-EN 14047 *Packaging. Determination of the ultimate aerobic biodegradability of packaging materials in an aqueous medium. Method by analysis of evolved carbon dioxide.*	The standard is identical and comparable with SFS-EN ISO 14852:2018.
2)	SFS-EN 14048 *Packaging. Determination of the ultimate aerobic biodegradability of packaging materials in an aqueous medium. Method by measuring the oxygen demand in a closed respirometer.*	The standard is identical and comparable with SFS-EN ISO 14851:en.
3)	SFS-EN ISO 14851 *Determination of the ultimate aerobic biodegradability of plastic materials in an aqueous medium. Method by measuring the oxygen demand in a closed respirometer.*	The test determines the degree of biodegradation by BOD evaluation inside a closed respirometer. Incubated in constant temperature between 20 to 25 °C ± 1. Test mixture consists of optimized inorganic test medium and activated sludge or a suspension of active Soil or compost as the inoculum.
4)	SFS-EN ISO 14852:2018 *Determination of the ultimate aerobic biodegradability of plastic materials in an aqueous medium. Method by analysis of evolved carbon dioxide.*	The method uses the medium, test environment and time same as SFS-EN ISO 14851:en, the determination of biodegradation is done by evaluation of CO_2 production.

5)	SFS-EN ISO 14853 *Plastics. Determination of the ultimate anaerobic biodegradation of plastic materials in an aqueous system. Method by measurement of biogas production.*	Biodegradability is determined by using biogas production (CH4 + CO_2) in anaerobic aqueous medium of digested sludge. Maximum time for the experiment is 90 days.

The main concern in this thesis is on the biodegradability or composting of packaging material. The standard that engages on the biodegradability and composting of packaging materials are further described below.

SFS-EN 13432
Packaging. Requirements for packaging recoverable through composting and biodegradation. Test scheme and evaluation criteria for the final acceptance of packaging

Taking care of the requirement of the organic recovery option from directive (94/62/EC), this standard has got the framework to support that this standard and other four standard can be used to support that the packaging product have been made according to the fulfilment of the requirements. The organic recovery includes aerobic composting and anaerobic bio gasification of packaging in municipal or industrial biological waste treatment facilities. The standard’s main aim is to verify if the packaging product is organically recovered or not by using the tools and additional standard provided in it. The standard suggests that EN ISO 14855, EN ISO 14851 and EN ISO 14852 could be used during different tests in the laboratory. If the standards provide legit information on the ultimate biodegradability of a packaging material in the controlled aerobic composting test other identical standards shall be used for laboratory purposes. (SFS-EN 13432, 2001)

SFS-EN 14045

Packaging. Evaluation of the disintegration of packaging materials in practical oriented tests under defined composting conditions

As the heading of the standard already says that this standard is used in evaluating the disintegration of packaging material. Also, this standard helps in visual perception and documenting photos and their contribution in the whole process. The time frame for the experiment is given 12 weeks. The temperature, pH, moisture content and gas composition with the composting material should be monitored and should fulfil some certain criteria. The test would require a composting bin of minimum 140 L. In the end of the process, the test material mixture should be sieved over 2 and 10 mm and the disintegration of the test material is evaluated. (SFS-EN 14045. 2003)

SFS-EN 14046

Packaging. Evaluation of the ultimate aerobic biodegradability of packaging materials under controlled composting conditions. Method by analysis of released carbon dioxide

This standard provides aerobic biodegradability and carbon dioxide released method to evaluate the biodegradability of packaging material. This standard requires 9 small vessels which should be chosen according to the size of test material used. From those 6 vessels, 3 are used for test material, 3 for reference substance and 3 for blank control. Those vessels with the required components and testing are than subjected to environment with temperature of 58 ± 2 C and aeration free from carbon dioxide. The most vital thing in this standard is maintaining the temperature and measuring the released CO_2 from the test material. The released CO_2 is analysed to find the biodegradability of the test material. (SFS-EN 14046, 2003)

7 TEST ENVIRONMENT FOR BIODEGRADABILITY

As I discussed in the literature about different biodegradable materials and many different standards to check the biodegradability of the standards, there is a need of designing the real environment where the biodegradability can be tested. Tampere university of applied science (TAMK) has been doing research and projects based on the designing of the test environment. This is also a part of research as a thesis chapter on giving the recommendations and suggestions on designing the test environment and step by step process for testing biodegradability.

First, I am using Oxitop® soil respiration method to test the biodegradation process of a test material in a normal condition in Laboratory. The purpose was to see what things in practice are necessary while doing a biodegradability test as it provides pressure curve which can be discussed about what happened in the biodegradation process. The Oxitop® soil respiration method also gives an idea and understanding of the whole biodegradation process which is very essential to consider in designing a biodegradability test environment.

7.1 Laboratory process (Oxitop®)

First, mature compost was collected from a domestic compost bin. The mature compost was then grinded into fine particles to be mixed with Planting soil (Puutarhamulta) bought from market. The ratio of the compost and soil mixture was 1:2 by mass. The mixture was finely mixed by hand.

Six jars of Oxitop® vessels of 500 mL for soil respiration was selected and filled with 50g of the prepared soil. In three of the jars test material was placed while other three were left as blanks for further comparison. 40 mL of the absorbing agent which was 1M NaOH was placed inside the holder.

The oxitop®-C sensor was screwed and tightened with lid and clips. The oxitop® OC 110 controller was used for collecting the pressure data from the sensor. After 14 days of the experiment time, the data from the controller was exported in computer and worked into curves using excel.

7.1.1 Results from Oxitop®

After 14 days of the experiment, the data was collected in oxitop® OC 110 controller and after working in excel, the curve was obtained as the figure below.

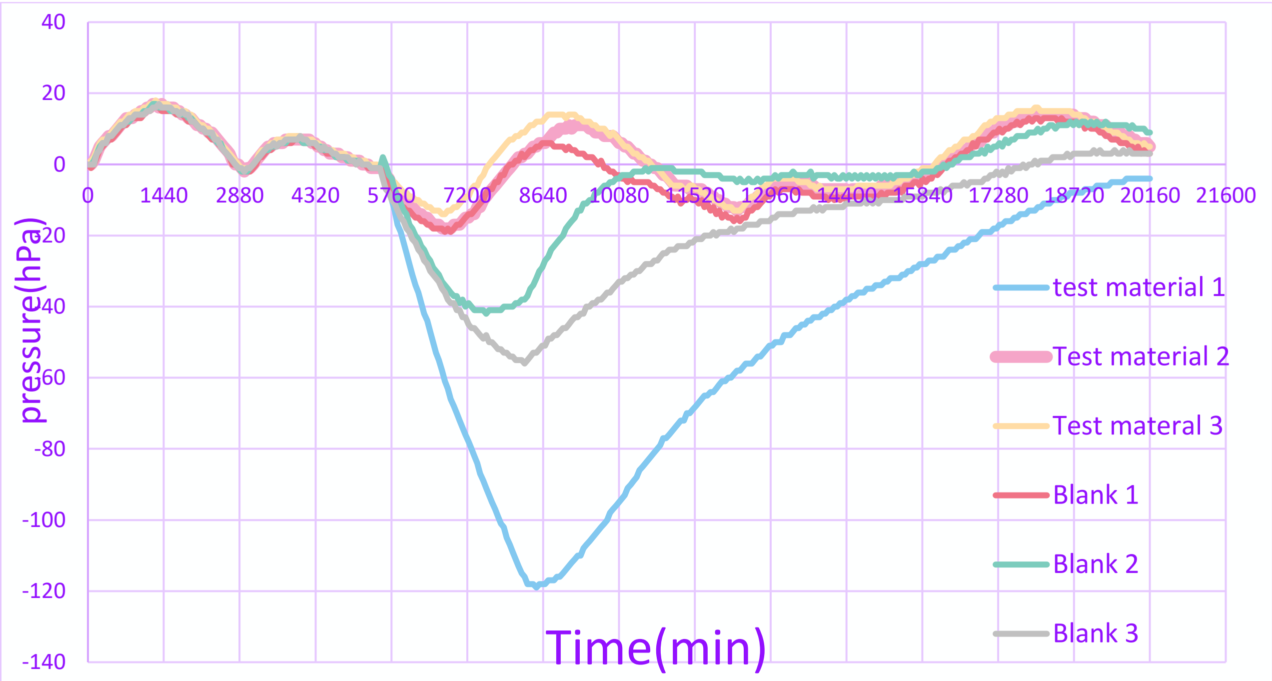

Figure 6 Pressure vs time curve of the oxitop experiment process.

The figure above that we got after working the data from oxitop sensor into the excel have six different curves separated with different colour and names in the legend. Each curve are for the individual oxitop glass jar whose names are given in the legend above in the figure.

From the figure, the result is very unusual in this case of measurement. According to the principle of the experiment, there should be significant decrease in pressure and the curve should be gradually going down. The curve going down indicates the biodegradation process happening by the consumption of CO2 leading to the decrease in pressure. The curve seems to be going down or decreasing in pressure can be seen for around 5 days for some jars but later the curves go up that means that the pressure inside the vessel is increasing. The curve going up, which indicates there is increase in pressure inside the oxitop vessel.

7.2 Design of a biodegradability test environment

The goal of this chapter is to suggest the process in designing the test environment for biodegradable or compostable packages. EN 14342 is the standard which defines the frame work and criteria for composting conditions and acceptance of the packages. But to do a real composting test, another standard is required which should be based on composting conditions. One of the well-recognised standards in this case is EN 14046, whose principle is to evaluate the ultimate biodegradability of the test material by the measurement of CO_2 under controlled composting conditions.

From the annex A (principle of the test system with released carbon dioxide) in the standard EN 14046, there is a basic interpretation of the model. The model shows all the components for designing the test environment. However, the test model only shows with one composting vessel which is not very clear to imagine the real model. So, I have drawn a basic model where all the composting vessels are present with all the necessary parts.

Carefully analysing test model in annex A of the standard, the test environment has been divided into five main parts. These five main parts are the material or system that needs to be designed using engineering programs and tools or maybe be purchased ready-made from the market. The model would look quite like the figure below.

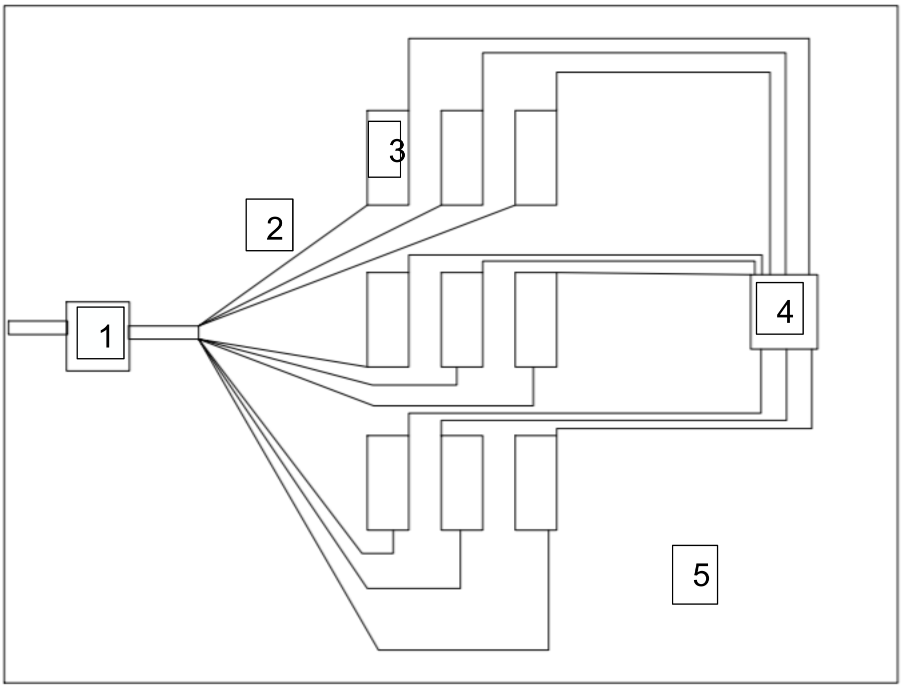

Figure 7 Interpretation of the biodegradability test environment according to SFS-EN 14046. From the figure,

1 is the air circulation system

2 is air tight tubes

3 is composting vessels

4 is Co2 determination system

5 is test environment(heating)

7.2.1 Air circulation system

According to the standard EN 14046, “synthetic air free from carbon dioxide or compressed air is supplied at constant low pressure and if compressed air is used the carbon dioxide is removed by passing through a suitable carbon dioxide absorption system”. So, the air that goes into the composting vessels should be CO_2 free. For removing carbon dioxide, sodium hydroxide or barium hydroxide solution can be used as absorbing agent.

Therefore, a pressurised-air system, which would provide CO_2 free air and water saturated in optimum flow rate and ensure enough aerobic condition to each of the composting vessels is necessary.

In NOTE 2 of section 8.3, it is mentioned that if CO_2 is measured directly, then CO_2 free air is not necessary, and CO_2 is measured from both inlet and outlet and subtracted inlet from outlet to get the CO_2 produced from biodegradation process in composting vessels.

7.2.2 CO2 determination system

This is another crucial part in the design of the test environment. CO_2 is measured from the exhaust pipe coming out from each of the composting vessels. In 7.2 section of the standard, two methods of CO_2 determination are suggested:

1. Device for direct carbon dioxide determination, if infrared analyser or gas chromatography is used than also measurement of gas flow is needed. TOC is another option.

2. Determination of DIC after complete absorption in a basic solution.

7.2.3 Composting vessels

In the section “8.3 start-up of the test” suggest that in typical case the composting vessel of volume 3 L should be used while in the section “7.2 composting vessels” also says that a minimum of 2 L composting vessel should fulfil the requirement shown in the standard.

Nine static composting vessels are required to start the test from which equal three vessels are required for test material, reference substance and blank con-trol each.

7.2.4 Test environment (Heating)

The test environment inside the vessels should be (58 ± 2) ºC, under diffused light and free from vapours. Heating system and monitoring is needed here to ensure the temperature.

7.2.5 Air tight tubes

The composting vessels should contain inlet and outlet gas tight tubes. Inlet contains CO_2 free water saturated air that passed into each of the composting. The air should be passed from the bottom of the vessel to ensure the enough aeration and even distribution into the compost. And outlet leads to CO_2 measuring system that measures the CO_2 production in the biodegradation process from the composting vessels.

After the construction of the design is ready, the biodegradability test is ready to perform.

7.3 Process

The step by step process while performing the test according to the standard EN 14046 is given below.

7.3.1 Preparation and measurements

The preparation is started by taking a healthy Inoculum from an operating composting plant (2 to 4 months old). If not, then from municipal solid bio-waste can be used. Removing the large particles from the inoculum and sieving the compost on 0,5 cm to 1 cm screen.

After taking a healthy inoculum, the following parameters should be measured and maintained before starting the test.

Total dry solids and volatile solid contents of the compost inoculum

Total dry solids should be between 50 % and 55% of wet solids and volatile solid contents of the compost inoculum should be more than 15% of wet solids or 30% of the dry solids. The parameters can be adjusted by adding water or aerating dry air into compost.

TOC of test material and reference material(cellulose)

TOC should be measured as g TOC per g total dry solids and minimum of 50 g total dry solids containing 20 g TOC per vessel is required.

pH

pH should be measured by making a mixture of 1-part inoculum and 5 parts of de-ionized water, shaking and measuring immediately. After than the pH should be between 7,0 and 9,0.

Total organic carbon, Total Nitrogen, fatty acid (optional): For Checking more characteristics of compost inoculum.

TDS and TVS of test material and reference material (optional) for comparing with the last results and calculating weight loss.

Carbon/nitrogen ratio

It is recommended that the Carbon/nitrogen ratio of the test mixture to be 10 and 40. It can be maintained by adding urea in the test mixture. The calculation to be done for total carbon by TOC of compost inoculum and total nitrogen by Kjeldahl method ISO 5663.

Now, the following steps are followed which are transcribed from the standard.

- Investigating the test material and making it suitable for the experiment which is by cutting into 2 cm * 2 cm as suggested by SFS EN14046.

- Starting test by making the 3 vessels for test material, reference substance and blank control ready. Same amount of compost in each vessel. Filling vessel ¾th of the vessel for manual shaking of the compost. According to SFS EN14046, if the vessel is 3L then 600 g of inoculum and 100 g of test mixture should be added to the vessel after measuring TSD of both materials.

- Fitting the composting vessels into the test environment and connecting tubes for supplying the required air and another tube for co2 determination.

- The oxygen concentration in the vessel should not drop below 6%. So, adjusting of air flow rate in the chamber is suggested in EN 14046(8.3).

7.3.2 Incubation period

As suggested in the standard EN 14046(8.4), if direct/momentaneous measurement of CO_2 is used than the measurement can be done twice a day during biodegradation phase and once a day during plateau phase. However, if dissolved inorganic carbon (DIC) is used than once per day during biodegradation phase and twice per week during plateau phase. Other important steps to be considered during the incubation period are listed below.

- Shaking of compost vessel weekly for making the compost healthy by removing the vessel from its CO_2 measurement system.

- Taking photos/videos of the test material weekly after shaking process and noting its structure, moisture content, colour, fungal development, smell of the exhaust air.

According to the standard (8.4), the time for the composting process is at least 45 days with constant temperature of 58°C ± 2°C, which can be extended if the biodegradation of the test material is not reaching plateau phase and can be shortened if the plateau phase is reached earlier. However, it should not exceed the composting limit of 6 months.

At the last of section 8.4, it is suggested that regular measuring of pH is necessary which is done by preparing a mixture of 1 part of compost inoculum with 5 parts of de-ionised water. If pH is less than 7,0 ± 0,2, biodegradation could be disturbed due to acidification. Volatile fatty acids should be checked and if 2 g of volatile fatty acid per kg of TDS has been formed than the test is considered invalid.

For determining weight loss which is option part of the test "note in 8.2" and "annex C" of the standard can be followed and is optional.

7.3.3 Calculation

Calculating theoretical amount of carbon dioxide (ThCO2) (in g per vessel)

$$Th\,CO_2 = M_t \times C_t \times \frac{44}{12} \qquad (4)$$

Where, M_t is total dry solids (TDS) of the test material introduced in the composting vessels at the start of the test (g); C_t is relative amount of total organic carbon in the total dry solids of test material (g/g);
44 and 12 are the molar and atomic masses of carbon dioxide and carbon.

Finally, the percentage degree of biodegradation is calculated using the following formula.

Percentage degree of biodegradation:

$$D_t = \frac{(CO_2)_t - (CO_2)_b}{Th\,CO_2} \times 100 \quad (5)$$

$(CO_2)_t$ is the accumulated amount of carbon dioxide released by each compost vessel (g/vessel); $(CO_2)_b$ is the mean accumulated amount of carbon dioxide released by the blank controls (g/vessel) ; $Th\,CO_2$ is the theoretical amount of carbon dioxide of the test material in the test vessels (g/vessel).

7.3.4 Expression of result

An example of a form sheet in annex E of SFS EN14046 guides for compiling the tables containing measured and calculated data of test material, the reference substance and blank control for each day of measurement. Making 9 carbon dioxide release curve as a function of time for all the vessels, and 6 bio-degradation curves for test material and reference substance in percentage as a function of time. The curve example given in annex B will be followed. In 9.4, it is suggested to calculate the average percentage of biodegradation if the deviation is less than 20% but if its more than that then values of each compost to be used separately.

Finally, describing the quality of test materials by comparing the test material structure, photos, etc taken previously.

7.3.5 Checking validity at the end and reporting:

- The percentage of biodegradation of reference substance to be more than 70% after 45 days.
- Deviation of biodegradation for the refence substance in different vessels is less than 20 absolute points at the end of test.
- CO_2 produced in compost inoculum in blank control produced after 10 days should be more than 50 and less than 150 mg per g of volatile solids (mean values).

Finally, the reporting should be done as per the instruction provided in the standard section 11 (test report).

8 DISCUSSION AND CONCLUSIONS

Although the biodegradability and composability of the packaging materials are discussed in the universities, schools and other big organisations, there are many big profit organisations that dominate the market and have caused main problems for the plastic and packaging pollution around the world. Also, there is difference between bioplastics, biodegradable bioplastics and fossils-based bioplastics, which is causing misunderstanding between the people and sound same for the common public. This should be made clear by the universities, government and related companies to the public.

The understanding of the difference between compostable and biodegradable is also main issue these days. There are many products that claim biodegradable, but they do not specify the real-time frame, which is necessary. The institutions and the costumers that use these products should be aware and understand the difference. There are logos that represent the biodegradability and composability which are recognised by the international standards which should be clear in the eye of public for separating the good and bad packaging.

More research in the products that replaces plastics, would be very crucial in creating plastic free packages. Materials such as PHA, PBA, starch, fibres, etc which are already mentioned above, should be in focus and the companies should be pressured to focus and change their production into these products which are identifies as biodegradable or compostable.

There are so many standards around the world which define biodegradability of packaging materials or plastics. Due to many standards, it is very difficult to find the relevant standard while doing a specific test. Internationally harmonised standard, which is for biodegradability and composting in different mediums is necessary. This would make clear and easier for conducting a biodegradability test.

The laboratory experiment which was done using oxitop device did not become successful because the result in the curve showed very unusual result which could be the effect of leaking of the air from the lid of the vessel as it was supposed to be air tight. If another experiment was conducted, the curve would make better formation. As the main aim of the experiment was to know the biodegradation process and things while conducting a biodegradability experiment, the result was not considered much importance.

When designing the test environment for biodegradability test requires the combination of engineering, chemistry and biology. The test design requires an air circulation system, which should be able to provide CO_2 free, right moisture and right flow inside the composting vessel, which is challenging task to maintain throughout the experiment. Also, there are other aspects such as CO_2 measurement and right temperature which need good engineering. This would be a project itself in constructing a design which would require the participants having background of engineering, chemistry and biology.

There is major concern in packaging waste due to plastics which end up in landfills, grounds and water sources, this has caused pressure to find another way for substituting the plastics because of which biodegradable plastics are getting popular these days. And after the growth of biodegradable plastics, another problem started when the companies started to use biodegradable and caused misunderstanding. Therefore, there are many standards to evaluate the biodegradability which should be used to define the biodegradability of biodegradable packaging products. These standards require a real environment to test the biodegradability, so one of the main concerns is to build the test environment to test the biodegradability of so-called biodegradable packages and plastics. Therefore, this thesis has also provided the steps on designing the test environment for conducting biodegradability test according to standard SFS-EN 14046. A further project or research would be required to build a real environment for the test which can also be called as a continuation of this thesis and this thesis would be very helpful for that project.

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