TAMPERE UNIVERSITY OF APPLIED SCIENCES

STUDY ON DEGRADABILITY OF WOOD COMPOSITE PACKAGING PRODUCT IN INDUSTRIAL COMPOST

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ABSTRACT

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Study on Degradability of Wood Composite Packaging Product in Industrial Compost
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The commissioner of this thesis Sulapac Ltd. has developed wood based composite packaging material, which meets the quality requirements of the cosmetic packaging. The main aim of this bachelor´s thesis was to test how this wood based composite packaging material degrades in the industrial composting facility. The second aim of the study was to compare the results to the requirements of the standards, which regulate packaging materials. The study was implemented in the industrial composting facility of Kekkilä Ltd. in Lappeenranta, by Tampere University of Applied Sciences.

Sulapac Ltd. provided wood composite sample jars, which were put to the stainless-steel capsules, with the compost material and were buried into the industrial compost for 12 weeks. Changes on the samples were examined and photographed, in the beginning, two times during and after the composting period. Dry mass of the samples was measured and disintegration of the samples was defined at every time of the investigation. Content of the capsules with disintegrated samples was sieved after test. Volatile solid content was measured from the samples.

According to the results, samples were disintegrating in the industrial composting facility. After 17 days of composting, 16 samples out of 24 were disintegrated to pieces and dry mass could not be measured. During the 12 weeks test period, 21 out of the 24 samples were disintegrated to pieces the size of 1-2 mm, six control plastic samples remained unchanged. Sieving test results support the assumption that 90 % disintegration of the samples, to the smaller than 2 mm size of the particles, may be reachable in 12 weeks in industrial composting environment. Volatile solid content of the sample mass was over 50%.

Results show that the wood composite material goes through significant changes in the industrial composting facility. Samples meet the SFS-EN 13432 standard requirements for disintegration and volatile solid content. The results of the Bachelor's Thesis can be used as a basis for a possible further study in which the biodegradability and compostability of packaging are tested in accordance with the SFS-EN 13432 standard.

Key words: wood composite, degradability, composting

TIIVISTELMÄ

Tampereen ammattikorkeakoulu.
Energia- ja ympäristötekniikan koulutusohjelma.

SIHVONEN PETRI
Puukomposiittipakkausten hajoamistutkimus teollisessa kompostissa.

Opinnäytetyö, 39 sivua, josta liitteitä 3 sivua
Huhtikuu 2018

Luodakseen vaihtoehdon muovin käyttämiselle kosmetiikkapakkauksissa, Sulapac Oy on kehittänyt puupohjaisen komposiittimateriaalin, joka täyttää kosmetiikkapakkauksille asetetut laatuvaatimukset. Tämän opinnäytetyön tarkoituksena oli tutkia kyseisen materiaalin fyysistä hajoamista teollisessa kompostissa ja verrata tuloksia vaatimuksiin, jotka Euroopan unioni on asettanut pakkausmateriaaleille standardissa *SFS-EN 13432; Pakkaukset. Vaatimukset pakkauksille, jotka ovat hyödynnettävissä kompostoinnin ja biohajoamisen avulla. Testausmenettely ja arviointiperusteet pakkauksen hyväksynnälle.*

Sulapac Oy:n tilaaman tutkimuksen kenttätyöt suoritettiin Kekkilä Oy:n teollisella kompostointilaitoksella Lappeenrannassa Tampereen ammattikorkeakoulun toimesta. Sulapac Oy:n toimittamat puukomposiittinäytepurkit ja muoviset kontrollinäytepurkit pakattiin teräskapseleihin ja haudattiin teolliseen kompostiin 12 viikon ajaksi. Näytteet kuvattiin ja mitattiin ennen kompostointia. Näytteissä tapahtuneita muutoksia tutkittiin kaksi kertaa kompostoinnin aikana ja kompostoinnin päätyttyä. Näytteiden kuivapainot ja fyysisen hajoamisen aste määritettiin kullakin tutkimuskerralla. Kapseleiden sisältö, hajonneine näytteineen, seulottiin kompostoinnin loputtua. Näytteiden haihtuvien kiinteiden aineiden pitoisuus määritettiin.

Tulokset osoittavat näytteiden fyysisen hajoamisen teollisessa kompostissa. 17 päivän kompostoinnin jälkeen 16 näytettä 24:stä oli hajonnut kappaleiksi siten, että kuivapainoa ei pystytty enää määrittämään. 12 viikon kompostoinnin aikana 21 näytettä 24:stä hajosi 1 - 2 mm:n kokoisiksi kappaleiksi. Kuusi muovista kontrollinäytettä pysyi muuttumattomina. Seulontatestin tulokset tukivat oletusta siitä, että näytteiden 90 prosenttinen hajoavuus pienemmiksi kuin 2 mm:n kappaleiksi on mahdollista saavuttaa 12 viikon teollisella kompostoinnilla. Määritys osoitti, että haihtuvien kiinteiden aineiden pitoisuus näytteissä oli yli 50 %.

Tulokset osoittavat tutkittujen puukomposiittipakkausten täyttävän SFS-EN 13432 -standardin vaatimukset fyysisen hajoamisen ja haihtuvien kiinteiden aineiden pitoisuuden osalta. Opinnäytetyön tuloksia voidaan käyttää pohjana mahdolliselle jatkotutkimukselle, jossa pakkausten biohajoavuus ja kompostoitavuus testataan SFS-EN 13432 -standardin edellyttämällä tavalla.

Asiasanat: puukomposiitti, hajoaminen, kompostointi

CONTENTS

TERMS

Biodegradation	Degradation caused by biological activity especially by enzymatic action leading to a significant change of the chemical structure of a material.
Composting	Biological decomposition of organic matter under aerobic conditions (Epstein 2011.)
Compost	Organic soil conditioner obtained by biodegradation of mixture principally consisting various vegetable residues, occasionally with other organic matter and having a limited mineral content.
SFS	Finnish Standards Association.
Degradation	An irreversible process leading to a significant change of the structure of a material, typically characterized by a loss of properties (e.g. integrity, mechanical strength, change of molecular weight or structure) and/or fragmentation.
Disintegration	The physical falling apart into very small fragments of packaging and packaging material.
Total dry solids	Amount of solids obtained by taking a known amount of test material or compost and drying at about 105 °C to constant weight.
Volatile solids	Amount of solids obtained by subtracting the residues of a known amount of test material or compost after incineration at about 550 °C from the total dry solids content of the same sample. The volatile solids content is an indication of the amount of organic matter.
SFS-EN 13432	This standard consists of the English text of the European Standard EN 13432:2000. Packaging. Requirements for packaging recoverable through composting and biodegradation criteria for the final acceptance of packaging.
SFS-EN 13193	This standard consists of the English text of the European Standard EN 13193:2000. Packaging. Packaging and the environment. Terminology.
SFS-EN 14046	This standard consists of the English text of the

	European Standard EN 14046:2003. Packaging. Evaluation of the ultimate aerobic biodegradability of packaging materials under controlled composting conditions. Method by analysis of released carbon dioxide.
CR 13695 - 1:2000	This publication consists of the English text of the CEN Report CR 13695-1:2000 ”Packaging. Requirements for measuring and verifying the four heavy metals and other dangerous substances present in packaging and their release into the environment. Part 1: Requirements for measuring and verifying the four heavy metals present in packaging”

1 INTRODUCTION

1.1 The Study

The aim of the study was to test how wood composite products degrade in industrial composting environment. Different recipes in colours and coatings were tested. It was compared how those properties effect the time of the disintegration. The European Union Directive on Packaging and Packaging Waste (94/62/EC) defines requirements for packaging to be considered recoverable. Five European standards may be used when considered if products placed in markets meet the requirements of the directive. The other aim of this study was to gather the standards which relate packaging materials and to find out what are their requirements for defining a package as biodegradable.

Study of biodegradable wood composite products in an industrial compost was carried out in cooperation with four operators. Sulapac Ltd. was the commissioner of the thesis. The company has been working to find out sustainable biodegradable packaging solutions for the cosmetic industry. They have invented wood composite packaging material that is produced 100% by natural ingredients and could replace plastic packages. Some of those wood composite products were tested in this study. (Sulapac Ltd 2018.)

Kekkilä Ltd. is a manufacturer and marketer of the growing substrates, fertilizers and garden items for consumers, professional gardeners and landscapers. Kekkilä operates an industrial composting facility in Lappeenranta and provided the facility for the testing in industrial composting. (Kekkilä 2017.)

Tampere University of Applied Sciences was responsible for providing the equipment, laboratory space, guidance and supervision for the study.
Tampere University of Applied Sciences, Engineering student Petri Sihvonen had the responsibility of practical implementation of the study and delivering the thesis.

1.2 Timeline of the Study

September 2017 Preliminary measurements of the samples in TAMK.
20th of September 2017: Filling samples into the capsules in Lappeenranta.
25th of September 2017: Capsules buried into the tunnel compost in Lappeenranta.
12th of October 2017: The ending of tunnel composting. First observation point of the samples. Capsules were opened and content was examined.
13th of October 2017: Capsules buried into the maturation compost in Lappeenranta.
21st of November 2017: Second observation point. Capsules were opened and content was examined. Capsules were buried again to the maturation compost.
18th of December 2017: End of the composting. Capsules were dug out of the compost.
19th of December 2017: Final examination of the samples in TAMK.
Spring 2018 Reporting of the results.

2 BACKGROUND

2.1 Packaging Regulations

European Union is regulating packaging and has set The Directive on Packaging and Packaging Waste (94/62/EC) that defines requirements for packaging to be considered recoverable. European standards, listed in the table below, amplify these requirements. Standards are used together to support the argument that the packaging is in harmony with the requirements of the directive when it is placed on the market.

TABLE 1. European Standards concerning recoverable packaging.

SFS - EN 13427	Packaging. Requirements for the use of European standards in the field of packaging and packaging waste.
SFS - EN 13428	Packaging. Requirements specific to manufacturing and composition. Prevention by source reduction
SFS - EN 13429	Packaging. Packaging and the environment. Terminology.
SFS- EN 13430	Packaging — Requirements for packaging recoverable by material recycling
SFS- EN 13431	Packaging — Requirements for packaging recoverable in the form of energy recovery, including specification of minimum inferior calorific value.
SFS- EN 13432	Packaging. Requirements for packaging recoverable through composting and biodegradation criteria for the final acceptance of packaging.
CR13695 - 1:2000	Packaging. Requirements for measuring and verifying four heavy metals present in packaging.

2.2 Terminology

As scientists are approaching and studying new environmentally friendly materials, businessmen are already using environmental friendliness as a selling point, sometimes with mixed and blurred terminology. For the public it may be difficult to understand the meaning of terms which are used when the environmental effects of the products are defined. EU directives and standards define criteria for packaging materials need to fulfil for it to be accepted into the market and what kind of words can be used when that material is marketed. Federal Trade Commission (FTC) in the United States has published guidelines on how to use these so called “green” terms in the marketing. In the chapter below, two often misused words, relevant for this study are explained.

Biodegradable:

FTC defines biodegradability as follows: “To claim a product is “biodegradable,” a company should have proof that the product will completely break down and return to the nature within a year. Landfills shut out sunlight, air, and moisture, so even paper and food could take decades to decompose. Most plastics won’t biodegrade even outside of a landfill.” (FTC Green Guides 2012.)

European standards require scientific proof that a product is degrading due to biological activity especially by enzymatic action and that it leads to a significant change in the chemical structure of a material. Products should turn to the carbon dioxide, water and minerals within reasonable time and it needs to be proven using method that standards are given. (SFS-EN 13432 2001.)

Compostable:

FTC defines compostable as follows: “If a product is labelled “compostable," all the materials in it should safely turn into usable compost in a home compost pile. If the product can be composted only at certain places, like a commercial facility, the advertising should say so.” (FTC Green Guides 2012.)

European standards require that compostable product should fully fill the requirements set in the SFS-EN 13432 standard. A compostable product should turn into carbon dioxide, water and minerals within 12 weeks in an aerobic composting facility. Cultivation tests, using compost with and without tested compostable product, need to be conducted and the tests need to prove that the compostable material does not harm plants. (SFS-EN 13432 2001.)

2.3 Composite Materials

A composite is a material which contains two or more materials with different chemical or physical properties. When the materials are combined, the features of the original materials will be changed. Motivation to make composites is to get better or more suitable properties for the material used for a special purpose. Composites are found in a multitude of applications in today world. They are used in building materials (concrete, plywood), car components (glass fiber, tyres), medical devices (thermoplastics, carbon fiber), furniture (chipboard) and in many other places. (Stokke 2014; Finnish Plastics Industries Federation 2018.)

2.3.1 Composite Ingredients

Different ingredients in the composites have their own roles. At least one of the ingredients is called resin or matrix. It acts like a glue and keeps all the other ingredients together. Common raisins are; polyvinyl chloride (PVC), polyethylene (PE), polypropylene (PP), urea formaldehyde, phenol formaldehyde and epoxy. Normally one of the other composite materials is called reinforcement or dispersed phase. That material is fibrous or particulate and enhances the mechanical properties of the composite. Common reinforcement materials are glass fiber, carbon fiber, cellulose and wood. The composite may contain other materials such as colours that give desired properties for the composite. (Oksman Niska & Sain 2008, Stokke 2014.)

“It is increasingly essential that the technology to great composite materials from renewable resources such as wood be understood applied and improved” (Stokke 2014).
In times of global warming and reducing petroleum reserves, conversion of biomass to the polymers and composites, has great potential to reduce use of the fossil fuels. Biomass is growing by sunlight and taking CO_2 from the environment, so it has considerably economic and environmental value. (Wool, Sun 2005.)

Price of the biodegradable natural sources based products is higher than same items made by plastics. That is main reason why bio products are not used more widely. According Sulapac Ltd, production costs or their wood composite material is higher than plastics and that is why price of the single product is 1/3 higher than same product made by plastics. If the volume of the production could be increased to the industrial level, costs per produced unit will decrease. (Sulapac Ltd 2017.)

Other reason which is limiting usage of the biodegradable products is their properties. In extreme conditions properties of the plastics meet best for the demands. There have been much more research and effort to develop plastics than products from natural origin. This may be changing when awareness of the plastics negative effects to the environment is growing. (Pickering 2008.)

2.3.2 Wood in Composites

Wood is a natural composite that contains lignin, cellulose and hemicelluloses (Oksman 2008). If material contains wood, that is processed and combined with other materials, it belongs to the category of the synthetic composites. The synthetic wood composites are produced to improve properties of wood and use material more efficiently. Wood is used many ways in the composite materials. In the plywood, thin layers of the wood are glued together to make strong plates. Wood can be used as a reinforcement by adding different size of sawdust, wood chips or cellulose fibers to the mass of the composite material. Wood is often cheaper than the synthetic polymers used in the composites, and because of that wood is used also as a filler. (Oksman 2008.)

2.4 Composting

Composting is process where organic matter is degrading to the humic matter, minerals, carbon dioxide and water. The degradation process of the organic compounds in the compost is caused by the microorganisms and by the enzymatic reactions. Hundreds of species of fungi, bacteria and actinobacteria are the primary decomposers. In industrial compost, mesofauna (worms, mites, millipedes) which occurs in the nature and home composts, can be ignored. (Diaz & al 2007.)

Composting process happens in four phases. Limits of the phases are not steep and time of the process varies. Compost composition, humid content, oxygen content and temperature affect the speed and intensity of the composting process. Result of the completed composting process is mature and stable compost. In mature compost temperature is stable, possible pathogens and parasites are dead and most of organic matter is degraded. (Epstein 2011.)

2.4.1 Four Phases of Composting

In the first, Mesophilic phase (called also starting phase) temperature starts to grow because of the microbial activity. Easily degradable sugars and proteins are degraded. Temperature is between 25-40 °C (Diaz 2007).
At the second phase (thermophilic phase) temperature rises and organisms adapted to the higher temperatures replace mesophilic fauna. Decomposition accelerates until temperature reaches 62 °C. At 65 °C, most of the microorganisms dies but temperature can rise to 80 °C due to abiotic exothermic reactions. (Epstein 2011.)

In third, cooling phase (second mesophilic phase) thermophilic organisms have consumed most of their feed and the activity starts to slow down. Temperature drops and mesophilic fauna, that can degrade cellulose and starch, starts to multiply. (Diaz 2007.)

The fourth phase is maturation. Compounds that are not further degradable, like lignin-humus complexes, are formed. Microbial community changes slowly entirely. Activity slows down and the number of the bacteria decreases. (Diaz 2007; Epstein 2011.)

2.4.2 Industrial Composting

Industrial composting sites are large scale composting facilities, where organic waste is treated in controlled environment. Systems available can be grouped into two categories. In the windrow systems, compost material (substrate) is treated in the clamps. In the in-vessel systems all or part of the composting takes place in the reactor. (Diaz 2007.)

In this study, windrow system compost facility was used. Kukkuroinmäen composting site in Lappeenranta is contribution-based tunnel composting facility with capacity of 34 500 tons a year. The facility is handling the home bio-waste of the province, bio-waste from the retail and food industry and sludges from the five municipal wastewater treatment plants. (Etelä-Karjalan Jätehuolto 2018.)

The starting phase and thermophilic phase of the composting process takes place inside of the tin hall where are the concrete sections, called tunnels. The phase in tunnel lasts for 14 to 21 days. At the beginning, the waste is mixed with wood chips and peat as a support. The mass is rotated weekly. After the tunnel phase, the compost is transferred outside to the drained clay field where cooling and maturation phases takes place. Compost is ready to use within six months. Composting takes place under the controlled conditions. Temperature and the amount of oxygen is measured continuously. To prevent emissions to the air, extract air is purified with the acid cleanser and biofilter. The mature compost is used for mould preparation, soil improvement and landscaping. (Etelä-Karjalan Jätehuolto 2018.)

2.5 Compostable Packaging Alternatives

Many biodegradable packaging products have been tested in the compost. Polylactic acid (PLA) is a thermoplastic commonly used in the packaging industry. It can be made from the natural raw materials such as starch, cellulose, soya or sugar. PLA degrades into water, carbon dioxide and minerals best in compost where temperature rises above 70 °C. That temperature is reachable in the industrial composting facility. According to studies PLA deli containers were fully degraded in 30 days, but some residues of PLA bottles was found after 30 days of composting, where maximum temperature rise over 60 ºC. (Kale et al. 2007.)

Properties of the PLA have been tried to improve by adding different natural origin materials. Wood flour PLA composite (PLA-WF) and its degradation in the compost environment have been tested in the Beijing Forestry University. 100 % PLA and PLA-WF containing 50 % PLA and 50% of wood flower (passing 0.149 mm sieve) was studied. Degradation of the samples were followed by loss of the weight of the sample and by electron microscopy. According the results PLA was degraded totally by 5 months and PLA-WF by 4 months in the composting environment. Microscopy analysis shows that wood flour accelerates degradation of the PLA. (Liu 2012.)

Polyhydroxybutyrate (PHB) and poly 3-hydroxybutyrate-co-3-hydroxyvalerate (PHBV) are biodegradable and compostable plastics produced by bacteria. They can be used as packaging material, but their production is expensive. According to studies PHB and PHBV degrades in the compost within 4 to 5 weeks. (Kapanen 2012.)

Study of the Technical Research Centre of Finland (VTT) studied biodegradation time of the biodegradable plastics in the controlled compost environment (Table 2). Biodegradability was measured by measuring emitted amount of the carbon dioxide (CO_2). Biodegradability time varied from 56 days to 150 days. (Kapanen 2012.)

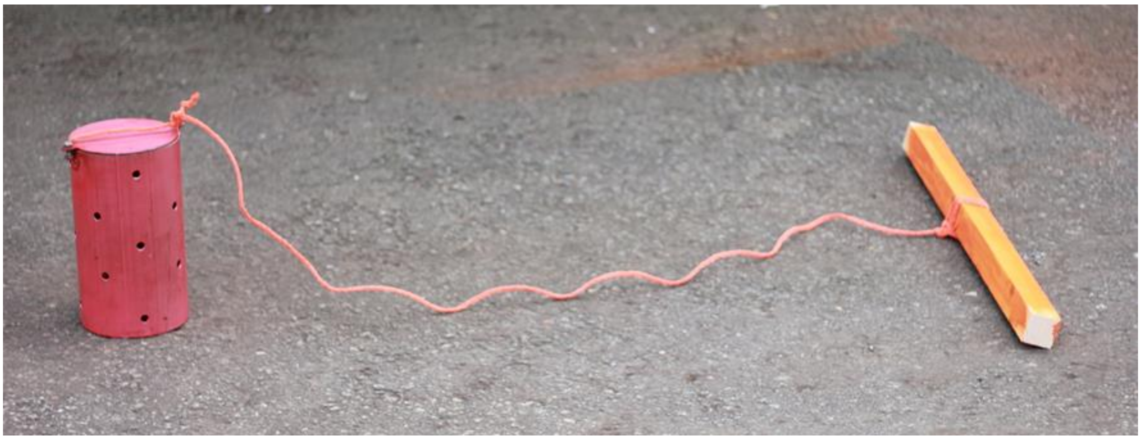
TABLE 2. Biodegradation times of biodegradable plastics (Kapanen 2012, modified)

Sample	Biodegradation (%) CO2 of ThCO2	Time (d)
Experimental product composed of starch,poly(caprolactone) and poly(urethane)	90	56
Lactic acid oligomer, 4% BD	85	112
HMDI -based poly(ester-urethane)	87	112
HMDI -based poly(ester-urethane)	95	112
BDI -based poly(ester-urethane)	91	70
PEA Poly(ester-amide)	100	112
PLLA Polylactide (POLLAIT, Fortum, Finland)	92	150

3 MATERIALS AND METHODS

3.1 Capsules inside the compost

The capsules used in the testing were cylinders made of stainless steel. Height of a capsule was 30 cm and diameter 16,5 cm. A capsule had 20 drilled holes, each a diameter of 12 mm, to ensure humidity and gas exchange. A lid of the capsule was made of stainless steel and it was attached to the cylinder with two nuts. To make location of the capsules more visible in the compost piles, the outsides of the capsules were painted pink and a rope with a marking stick was attached. The rope was made of polyethylene with a 6 mm diameter. Colour if the rope was orange and it the material was UV durable. As A marking stick, wooden beam size of 5 times 5 cm and length of 80 cm, was used. The stick was painted by orange spray paint to increase visibility. During the composting some of the ropes and sticks went broken and missing. A missing stick were replaced by a plastic ploughing sign stick. The ploughing sign was made of orange plastic and its length was 150 cm and diameter 19 mm.



PICTURE 1. Capsule, rope and marking stick used in the study (Petri Sihvonen 2017.)

3.2 Samples

Wood composite packaging products, later called samples, were provided by Sulapac Ltd. Samples were shape of the cans that are commonly used in the cosmetic packing. Diameter of the can was 7 cm and height of the edge 3 cm. Samples were made by moulding machine and because of that technique, they had 5 cm long and 0,5 cm thick spike on the middle of the backside of the sample. That spike was removed before the samples were used. The spikes were stored and used later for the volatile solids analysis.

Eight different recipes of the wood composite material were tested and three (3) samples of each type was used. For the control six (6) plastic cans commonly used in cosmetic industry were used. Plastic cans were approximately same shape and size than wood composite cans. A total of 24 wood composite packaging products and six (6) plastic samples were loaded into 12 capsules with the raw material of the industrial compost. Each capsule had two or three samples. Four of the capsules had loggers which were measuring temperature during the test.

PICTURE 2. Three examples of the samples (Petri Sihvonen 2017)

PICTURE 3. A plastic jar, used as control sample in the study (Petri Sihvonen 2017)

3.3 Composting

Practical work of the study was conducted in the Kekkilä Ltd. industrial composting facility in Lappeenranta. The compost manufactured is mixture of the municipal waste water treatment plant sludge and household bio-waste. Untreated sorted waste wood is used as a support material and source of the carbon. 12 capsules containing samples were buried at the depth of 1-1,5 meter into the compost pile with a bucket loader by workers of the industrial composting facility. In the first composting phase capsules were buried into the two different compost piles which were in the tin hall in the separated sectors called tunnels. During tunnel composting phase piles were turned and mixed one time so that all compost material from a tunnel was moved from one tunnel to the other tunnel. On this study tunnel composting time was 17 days.

On the second composting phase compost material war dumped on one pile to the outside maturation field. All the capsules were buried into the same compost pile at the depth of 1-1,5 meter.

3.3.1 Conditions in the Compost

Composting conditions were regulated during tunnel phase. Temperature and oxygen were controlled by adjusting the flow of the air blown thought the compost pile. Temperature was measured from the supply air channel and from the extract air channel. Four temperature loggers were located into the capsules and they measured temperature inside of the capsule in the compost pile. Oxygen was measured from the extract air channel. Temperature of the air in the supply air channel varied from 0 °C to 20 °C, (depending of the weather and the amount of the recycled air in the channel.) Air temperature in the extract channel varied between 22 °C and 59 °C. (Appendix 2,3.)

Amount of the oxygen in the air was lowest at 13,9 % and average was 20 %. To keep mass of the compost moist, 60 m^3 water was supplied to the piles (Kaipia 2017). On the maturation field compost laid without extra water or air supply.

3.4 Measurements

Preliminary measurements of the samples were done in the environment laboratory of TAMK. The samples were dried in a drying oven for 2 hours in 105 °C and their masses were measured using a laboratory scale (Pag Oerlikon Ag 250-9320). Individual pictures were taken from each sample by Canon EOS 550 and mobile phone camera of Samsung Galaxy Xcover 4.

The first two measurements were conducted during a test period from 12th of October and 21th of November. The capsules were dug up from compost pile, opened and the contents were observed. If the sample had not broken into pieces it was washed, dried in the oven for two hours in 105 °C and the dry mass measured with a laboratory scale. A picture was taken of each capsule’s content and every sample. After the inspections and the measurements, the contents were put back into the capsules and the capsules were buried into the compost pile. At the end of the test, a final examination was done in the environment laboratory of TAMK.

Volatile solids content was measured from the removed spikes of the wood composite samples. Four spikes were used. 1 g of the wood composite material was dried in the drying oven 2 hours in 105 °C and the dry mass was measured. Then samples were put to the 550 °C oven for 15 minutes. After cooling in the desiccator, weight of the residue was measured and compared with the dry weight.

Sieving test was done by using soil sample sieving set. 4 mm and 2 mm sieves were used. Contents of the capsules were dried three months, in the room temperature, on the open boxes. Each sample was placed to the sieve and mechanical shaker was used three (3) minutes to shake the sieve set. Three sieving divisions were separated and mass was measured. Percentage shares of the divisions were calculated.

4 RESULTS

4.1 Degradation

According to SFS-EN 13193 standard, "*degradation is an irreversible process leading to a significant change of the structure of a material, typically characterized by a loss of properties (e.g. integrity, mechanical strength, change of molecular weight or structure) and /or fragmentation*". Degradation of the samples was followed by measuring their dry mass and integrity. Table 1 shows changes in the dry mass of the samples on four measuring points. The first measurement on 20th of September was done before the samples were buried into the compost. The second measurement point was 12th of October when tunnel composting period finished. 21th of November was the third measuring point in the middle of the maturing composting period and the fourth and final measurements were done on 18th of December, when the composting ended.

On 12th of October the measuring point show that 16 samples out of 24 sample were mixed in compost substrate so that measuring of the dry mass was impossible. Only three samples out of 24 kept their shape to the third and fourth measuring points so that measuring of the dry mass was possible. The plastic jars which were used as a control, remained unchanged through the whole study period.

TABLE 3. Dry masses of the samples. A minus (–) sign indicates the loss of integrity of the sample, where the dry mass could not be measured.

Sample ID	Initial mass/g	Dry mass/g 20th of September	Dry mass/g 12th of October	Dry mass/g 21th of November	Dry mass/g 18th of December
1N200917PS	47,108	46,731	-	-	-
2N200917PS	47,199	46,788	-	-	-
3N200917PS	47,062	46,702	-	-	-
1D200917PS	47,574	47,188	-	-	-
2D200917PS	47,481	47,111	-	-	-
3D200917PS	47,451	47,085	46,602	-	-
1E200917PS	47,272	46,852	-	-	-
2E200917PS	47,21	46,801	-	-	-
3E200917PS	47,176	46,756	-	-	-
1F200917PS	46,287	46,061	46,766	45,579	42,513
2F200917PS	46,323	46,108	46,803	45,042	44,807
3F200917PS	46,785	46,559	47,577	45,316	44,524
1W200917PS	47,502	47,135	-	-	-
2W200917PS	47,386	47,007	-	-	-
3W200917PS	48,251	47,532	-	-	-
1C200917PS	47,84	47,523	47,482	-	-
2C200917PS	47,894	47,537	44,72	-	-
3C200917PS	47,892	47,532	-	-	-
1G200917PS	47,698	47,357	41,95	-	-
2G200917PS	47,64	47,252	31,841	-	-
3G200917PS	47,709	47,309	-	-	-
1B200917PS	47,273	46,843	-	-	-
2B200917PS	47,21	46,836	-	-	-
3B200917PS	47,83	47,413	-	-	-

4.2 Disintegration

Physical changes of the samples were observed three times during the study. According to SFS-EN 13432 standard, "*disintegration is the physical falling apart into very small fragments of packaging and packaging material*". In the beginning of the study, the wood composite packaging product samples were solid cans with smooth surface (Picture 1). During the disintegration process, the sample swelled, lost original form and eventually broke into 1 to 2 mm pieces.

The samples were inside of the capsules buried into the industrial compost material. After a sample reached the level of disintegration where it had broken into 1-2 mm size particles, further observation could not be done because the material from the samples and the industrial compost material could not be distinguished and separated from one another. First time samples were observed on 12th of October two and half weeks from the start of the test. Disintegration could already be detected in several samples. Seven samples out of 24 were disintegrated to the 1-2 mm size of particles. On 21st of November, eight weeks from the start of the test, 19 samples out of 24 had reached above mentioned level of the disintegration. When the last observation was done at the end of the testing period only three samples out of the 24 had not disintegrated to the 1-2 mm size particles. Six (6) control samples were not disintegrated and kept their shape and integrity during composting (table 4, picture 5,6,7).

TABLE 4. Disintegration of the samples during composting. Sign X indicates that sample was observed disintegrated to the 1-2 mm size particles on that observation date.

Sample ID	Disintegrated to the 1-2mm size particles on 12th of October	Disintegrated to the 1-2mm size particles on 21th of November	Disintegrated to the 1-2mm size particles on 18th of December	Not Disintegrated
1N200917PS	X			
2N200917PS	X			
3N200917PS	X			
1D200917PS	X			
2D200917PS	X			
3D200917PS		X		
1E200917PS	X			
2E200917PS	X			
3E200917PS		X		
1F200917PS				X
2F200917PS				X
3F200917PS				X
1W200917PS		X		
2W200917PS		X		
3W200917PS		X		
1C200917PS			X	
2C200917PS			X	
3C200917PS		X		
1G200917PS			X	
2G200917PS			X	
3G200917PS			X	
1B200917PS		X		
2B200917PS		X		
3B200917PS		X		
Plastic1M				X
Plastic2M				X
Plastic3M				X
Plastic1K				X
Plastic2K				X
Plastic3K				X

PICTURE 4. Disintegration of the sample No:2C200917PS. On the Left, original sample on 20th of September, before the starting of the test. In the middle, sample 12th of October, after 17 days composting in industrial composting facility. On the right, picture on 21th of November, after 17 days composting in industrial composting facility and 39 days in maturation field. Testing lasted 56 days in total on the composting field. (Petri Sihvonen 2018)

PICTURE 5. Reference sample PlasticM1 on the left. Sample 1G200917PS on the right. Pictures on 20th of September, before the starting of the testing. (Petri Sihvonen 2017)

PICTURE 6. Reference sample PlasticM1 on the left. Sample 1G200917PS on the right. Picture on 12th of October, after 17 days composting in industrial composting facility. (Petri Sihvonen 2017)

PICTURE 7. Reference sample PlasticM1 on the left. Sample 1G200917PS on the right. Picture on 21th of November, after 17 days composting in industrial composting facility and 39 days in maturation field. Testing lasted 56 days in total on the composting field. (Petri Sihvonen 2017)

4.3 Volatile Solids

SFS-EN 13432 standard requires that volatile solids content of the packaging material, which is evaluated, should be more than 50 %. To find out if studied wood composite material meets the requirements of the standards, loss of ignition test was done. The sample of the scientific test was small, only four samples, but still it gives an indication of the magnitude of the volatile solids content in the studied wood composite material. In all four samples volatile solids content was significantly higher than 50%.

TABLE 5. Volatile solid contents (Petri Sihvonen 2017)

Sample id	Mass of the sample / g	Volatile solids content / g	Volatile solids content / %
1B200917PS	0,966	0,94	97,3
1F200917PS	0,823	0,82	99,6
1W200917PS	0,806	0,704	87,3
3F200917PS	0,722	0,698	96,7

4.4 Sieving Test

The samples and the compost material (bio-waste, sludge and support wood) were inside of the capsules and they were mixed well. According to the results most of the capsule content particles did not pass the 4 mm sieve (Table 6.) Support material of the compost was coarse wood and it contains stones as well. Compost substrate material was heterogenous, that may have affected the results.

There was some evidence that disintegrated samples increased the amount of the smallest < 2 mm fracture sieved. Those capsules which had three samples per capsule had also largest percentage of the smallest particles in the receiver.

TABLE 6. Percent distribution of capsule content particles according to the size of the particles.

Capsule Content Mass / g	Particles % per fraction > 4 mm	Particles % per fraction 2 - 4 mm %	Particles % per fraction < 2 mm %	Samples in the Capsule
1166	86	4	10	1
767	82	5	13	1
1076	88	3	9	1
1184	88	3	9	1
785	87	3	10	1
922	82	3	15	3
1205	80	7	14	3
1006	81	3	16	3
894	80	4	16	3
1008	80	6	15	3

Visual observation gave indications that 1-2 mm wood ship particles, which were visible on the receiver, had originated from sample jars. Also fracture size 2 mm - 4 mm had visible particles which were formed from the sawdust chips stuck together. It seems that some of these particles were degraded from the samples (Picture 8).

PICTURE 8. Sieving residue; on the left 4 mm sieve, on the middle 2 mm sieve, on the right receiver.

5 DISCUSSION AND CONCLUSIONS

5.1 The aims of the study were achieved

The first aim of the study was to test how wood composite products degrade in industrial composting environment. The greatest challenge in the implementation of the study was the long distance between composting facility and Tampere. Co-operation with the staff of the composting facility worked well and room for the handle the samples was sufficient. Implementation of the study began one month later than was planned, still the desired result was achieved. According to the results, the studied material disintegrated in the industrial composting environment on the time frame given in the standards concerning compostable material. Sieving test supports the assumption that 90 percent disintegration to the smaller than 2 mm size pieces, may be reachable in 12 weeks in an industrial composting environment. Volatile solid content tested from the samples was above the limits of the standards thus filling the standard requirements.

The second aim of this study was to gather the standards, which relate packaging materials and to find out what are their requirements for defining a package as biodegradable. To find and read through plenty of standards was time consuming work. Standards are listed in the chapter 2.1. All listed standards are normative references of the SFS-EN 13432 standard. To get the final acceptance the compostable biodegradable material should pass all steps and requirements written in SFS-EN 13432 standard, which sets precise criteria for packaging recovery through composting and its final acceptance. Standard Chapter 4. point 4.3.2. concerns materials of natural origin. It is acceptable only if all the materials and constituents in the package are chemically unmodified. If so, the package shall be accepted as being biodegradable without testing. In addition to that it needs to be chemically characterized. That includes information on, and identification of the constituents of the packaging materials. Presence of hazardous substances, organic carbon content, total dry solids and volatile solids need to be determined. Packaging needs to pass the test of the disintegration, tested in the pilot scale or full-scale aerobic composting facility. After 12 weeks test period in the compost 90 % of the packaging material tested should pass through a 2 mm sieve. The last test which the packaging must pass, is the quality of the compost test. In said test, pure compost and compost with added packaging material, are compared by physical-chemical parameters and by a cultivation test.

Difference in the growth of the biomass in the cultivation test should not be more than 10%. Packaging material added to the compost should not cause negative effect to the quality of the compost. (SFS-EN 13432.)

If the natural origin material is chemically modified the biodegradability test need to be done according to SFS-EN 14046 standard. *Packaging. Evaluation of the ultimate aerobic biodegradability of packaging materials under controlled composting conditions. Method by analysis of released carbon dioxide.*

5.2 Uncertainties of the study

According to discussion with operator of the industrial composting facility Vesa Kaipia and based to the temperature data from loggers, this study had some uncertainties, which may have influenced the results but had not decisive significance to the results. Temperature, humidity and oxygen conditions were not equal on all parts of the industrial compost pile. Top of the pile was cooler and the humidity inside of the pile was higher. Compost pile was sprayed with water to ensure sufficient humidity levels, but the capsule and position of it in the pile may have prevented water flow inside the capsule. Secondly, the capsule may contain increased humidity inside of the capsule by preventing airflow, which is blown through the compost pile to ensure sufficient oxygen level.

Samples were dug up from the compost two times during the composting period. When capsules were out of the compost pile temperature of the capsule, samples and content degreased temporarily to 0 - 5 °C, which was ambient air temperature (Appendix 1). Temporarily degreased temperature may have delayed the degeneration process at some extent.

5.3 Development suggestions

Even though the study was implemented successful as planned, there could be room for improvement. In the industrial composting site heavy machinery was used when compost pile was turned over and when compost material was moved from tunnel composting pile to a maturation pile. Heavy machinery was used to dig up the sample cylinders from the compost pile. That caused the breaking of two cylinders and half of the marking sticks. One cylinder was bent badly, when the capsules were dug up on 21st of November 2017 for the second observation. The content of the capsule was uncompromised, but due to damage the capsule could not be put back into the compost, since there was no extra capsule available. At the end of composting, the lids of two capsules were broken, when capsules were dug out of the compost. Capsule number six (6) was totally empty and all its content was lost. Capsule number nine (9) was open but half of the content was uncompromised. To minimize loss of the contents and samples, capsules should be redesigned. To ensure gas change between the samples and the compost material, the number of ventilation holes in the capsule could be increased. The material of the capsules should be pondered upon. More careful machinery operation would minimize damage to the capsules. When it comes to the finding capsules from the compost piles, the wooden marking sticks worked better than the plastic ones. Essential is that the rope between the mark and the capsule is strong enough and the connection to the capsule is solid. Possibilities to use a digital tracking device or a microchip inside of the capsules may be worth to investigate.

Temperature of the tunnel composting pile is measured from the exhaust and intake air channels. When temperature data from the loggers, used in this study, was compared with the data from the composting facility, they have differences. Loggers show that the inside temperature of the compost pile grows to 72 °C in the tunnel composting time. Exhaust air measurements show maximum temperature of the exhaust air to be 59 °C (Appendix 2,3). Target values are 57 °C (hygienisation limit > 55 °C) for 72 hours and after that 49 – 52 °C (Kaipia 2017). Inside of the maturation pile loggers show that maximum temperature was 78 °C and three of four loggers showed temperatures over 70 °C for more than 10 days (Appendix 1.) According to sources, most of the microorganisms will perish at 65 °C, and composting slows down if temperature is above 62 °C. Ammonia emissions to the air rise significantly if the temperature will raise above 70 °C. (Diaz 2008; Epstein 2011.) There might be several reasons why the loggers and sensors show different temperature. Steel capsule may store heat inside of the capsule and walls may prevent flow of air. Location in the compost pile may affect to the temperature. To get more accuracy to the temperature measurements, a few loggers could be put into the compost pile without capsules to get reference data.

Results of this study show that the wood composite material invented by Sulapac Ltd. goes through significant changes in the industrial composting facility. Samples meet the

SFS-EN 13432 standard requirements for disintegration and volatile solid content. The results together with the listed standards gives a starting point for further studies which could be biodegradability and compostability, life cycle analysis, carbon footprint, hazardous substance analyses, quality of the compost tests and degeneration test in the soil.

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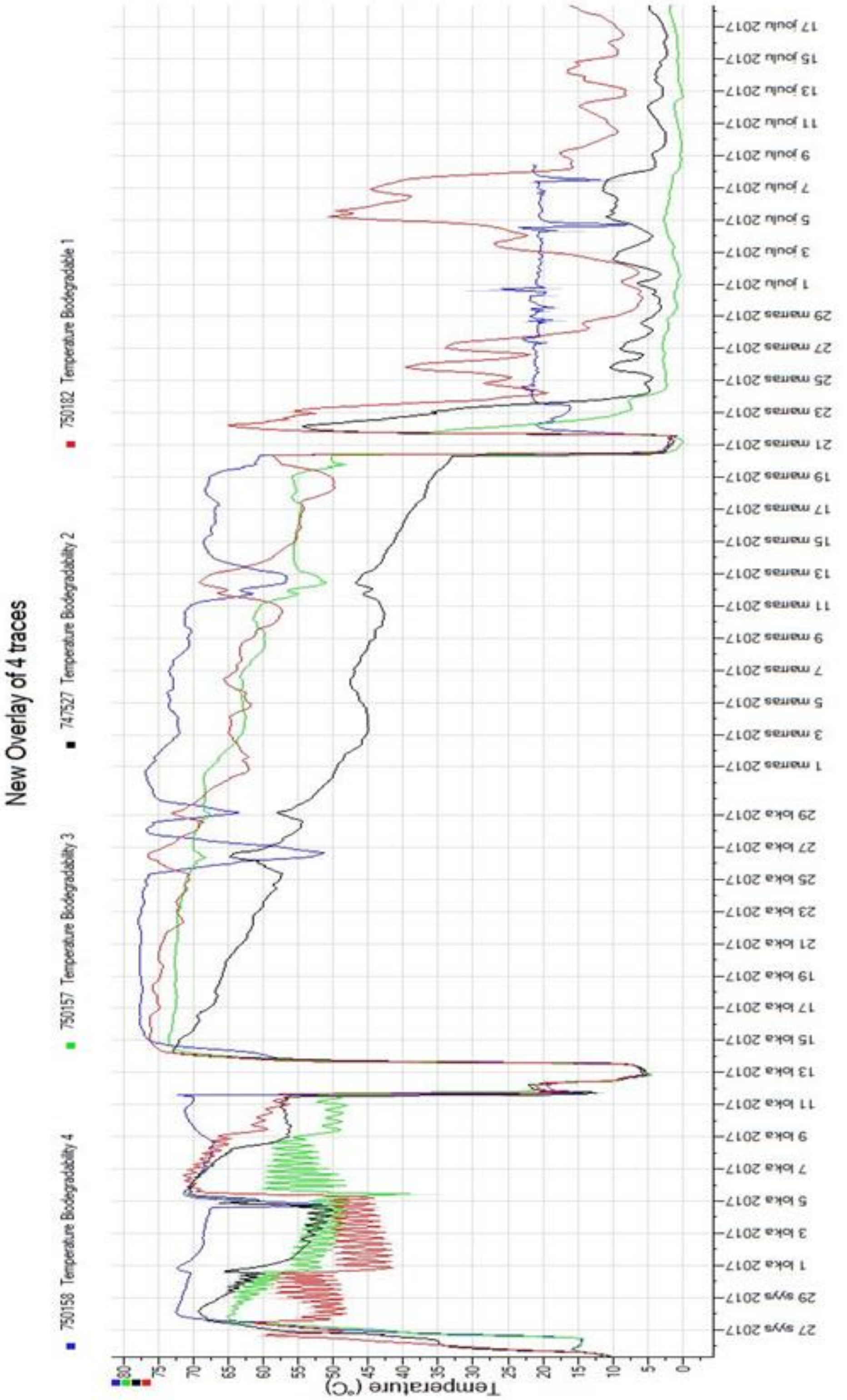
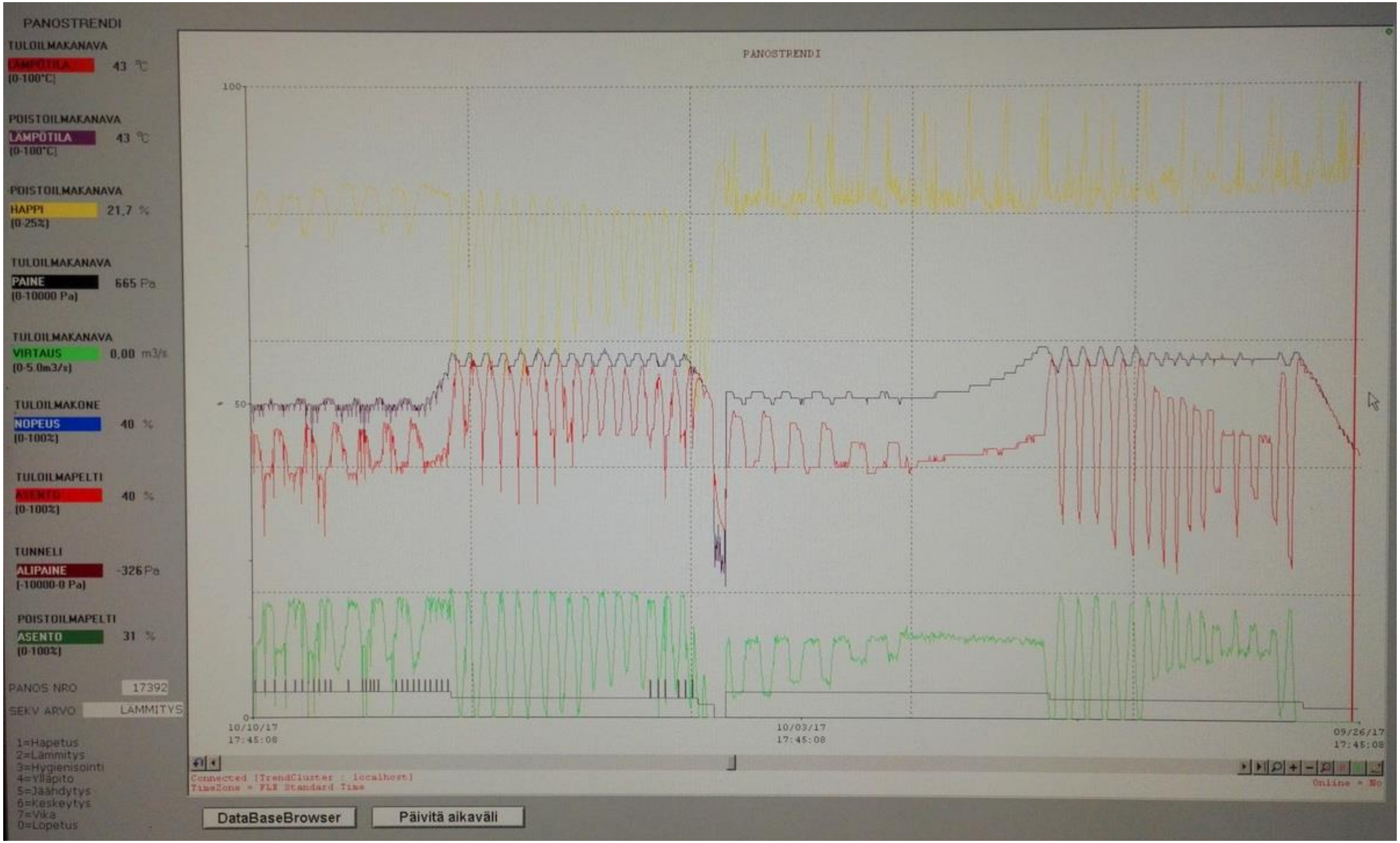
APPENDICES

Appendix 1. Temperature measured by loggers from inside of the compost pile.



Appendix 2. Temperatures measured from Exhaust air channel (purple) and intake air channel (red) during tunnel composting. Pile 1. (Kaipia 2017.)



Appendix 3. Temperatures measured from exhaust air channel (purple) and intake air channel (red) during tunnel composting. Pile 2. (Kaipia 2017.)