Tampereen yliopisto

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THE RE-DESIGN OF A SUSTAINABLE MONOCOQUE SHELL FOR ULTRA HIGH FREQUENCY TRANSMITTING RADIOS

Master's Thesis
Materials Science and Environmental Engineering
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ABSTRACT

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In modern days, the impact of human activities to the ecosystem of the world have caused many countries and companies to seek new ways to lower their environmental impact. The rapid industrial development and economic growth combined with the advancements in technology have had their price paid towards the environment in a negative way. Due to this fact, a range of investments, initiatives, and regulations have been made to lower the environmental impact caused by the society.

During the 20th century, the usage of fibre reinforced polymer composites has been increasing due to the developments in composite technologies. With these high-performance materials, we have been able to manufacture objects with a low weight combined with high mechanical performance. My work was to re-design a radome shell structure for the Luxturrim5G project using advanced long-fibre reinforced composites: manufacturing of 'sustainable prototype' for the project, re-design it through the materials selection, and testing novel sustainable composite materials. In the thesis, especially the biodegradation-related properties were under a focus, so the chosen testing method for the novel composite was chosen to be soil burial. The composites used during the work were a type of natural fibre reinforced polymers that used flax fibres as reinforcing element.

The fabrication of the prototype of the sustainable CRU was done by the data received from the Luxturrim5G project and the prototype was showcased during the Luxturrim5Gs project finale, The Impact Day, held in Nokia premises in Karaportti, to demonstrate the usage of alternative composite materials. The NFRPs pose certain problems in their long-term durability in atmospheric conditions. However, these problems can be somewhat avoided with the proper treatments. During the soil burial, degradation can be seen in the composites which would affect the long-term durability. In the work, it was observed that the rosin had positive effect on the biodeterioration.

TIIVISTELMÄ

Jutila, Lauri: Komposiittikuorirakenteen suunnittelu kestävän materiaalivalinnan sekä 5G radioiden vaatimusten rajoitteilla
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Nykyään ihmisen toiminnan vaikutukset maailman ekosysteemiin ovat saaneet maat ja yritykset etsimään uusia tapoja vähentää omia ympäristövaikutuksiaan. Nopea teollisuuden kehittyminen ja talouskasvu yhdistettynä edistyksiin teknologioissa ovat tapahtuneet ympäristömme kustannuksella. Asian vuoksi eri tahot ovat tehneet investointeja, aloitteita ja säädöksiä, jotta yhteiskunnan aiheuttamia negatiivisia ympäristövaikutuksia voitaisiin vähentää.

1900-luvulla kuitulujitettujen muovien käyttö on lisääntynyt komposiittiteknologian kehityksen myötä. Näillä korkean suorituskyvyn materiaaleilla on pystytty valmistamaan keveitä ja mekaanisesti vahvoja kappaleita. Diplomityössäni tarkoituksena oli suunnitella tutkakuvun kuorirakenne uudelleen osana Luxturrim5G projektia käyttäen pitkäkuituisia lujitemuoveja. Työ jakautui kolmeen osaan, jotka olivat “ekologisen prototyypin” valmistus, uudelleen suunnitella tutkakupu kestävän materiaalivalinnan kautta ja valmistaa ja testata uudenlaista kestävää lujitemuovia. Diplomityössä tutkittiin erityisesti materiaalin biohajoavuutta, joten valittu testausmetodi oli maaperään hautaaminen. Työssä käytettiin luonnonkuitulujitettuja muoveja, joissa lujittavana kuituna käytettiin pellavaa.

Kestävän tutkakuvun, eli ‘Camouflage Radome Unit’ rakenteen, prototyypin valmistus tehtiin Luxturim5G-projektin spesifikaation mukaan ja valmis prototyyppi esiteltiin Luxturim5G projektin päätöstapahtumassa, The Impact Day:ssä, Nokian Karaportin yksikössä Espoossa demonstroimaan vaihtoehtoisten materiaalien käyttämistä. Luonnonkuitulujitetuilla muoveilla on ongelmia niiden pitkäaikaiskestävyydessä ulkoilmassa. Nämä ongelmat voidaan kuitenkin välttää sopivilla käsittelyillä. Maaperään hautaamisen aikana komposiiteissa havaittiin biohajoamista, joka vaikuttaa materiaalien pitkäaikaiskestävyyteen. Diplomityössä myös havaittiin, että pihkalla on positiivinen vaikutus biovaurioitumisen ehkäisemiseen.

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PREFACE

This master's thesis was done for the Plastics and Elastomer Technology research group at the Faculty of Engineering and Natural sciences in Tampere University between February 2019 and March 2020. The work was done as part of Luxturrim5G project which is Nokia Bell Labs driven project to develop ecosystem for digital smart city in collaboration with many finish companies and institutes.

First and foremost, I want to thank people working in the research group. Especially, my examiners assistant professor Mikko Kanerva and doctoral researcher Pauli Hakala. Both were great people to work with and gave me great advices for the work itself and for the future of my life.

Finally, I like to express my gratitude to my family for supporting me before the thesis, during the thesis, and after the thesis. Also, big thanks to these three groups: Kahvipytty, The White House, and Team Jutinpaatti. Without you guys, life would be dull.

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ABBREVIATIONS AND TERMS

2x2 Twill	Woven textile pattern
5G	Fifth generation cellular network
CFRP	Carbon fibre reinforced polymer
CRU	Camouflage radome unit
EPS	Expanded polystyrene
FE	Flax Epoxy
FP	Flax PLA
FPR	Flax PLA and Rosin
FRP	Fiber reinforced polymer
GFRP	Glass fiber reinforced polymer
GHE	Greenhouse gas emissions
HRR	Heat release rate
ILSS	Interlaminar shear strength
LCA	Life cycle assessment
Mater-Bi®	Tradename for biobased and biodegradable thermoplastic starch based polymer system produced by Novamont
NFRP	Natural fibre reinforced polymer
NF	Natural fibre
PLA	Polylactic acid
PE	Polyethylene
PET	Polyethylene terephthalate
powerRibs®	Flax supporting reinforcement element produced by Bcomp Ltd.
PP	Polypropylene
PS	Polystyrene
RRR	Reuse, recycle, recover
RtFPR	Rosin treated Flax – PLA and Rosin
RTM	Resin transfer moulding
Super Sap®	Tradename for biobased epoxy resin produced by Entropy Resins
TPS	Thermoplastic starch
UD	Unidirectional
UP/UPE	Unsaturated polyester
VARTM	Vacuum assisted resin transfer moulding

σ	Stress (uniaxial)
A	Area
a	Acceleration
F	Force
g	Gravitational acceleration
m	Mass
T	Thickness
W	Width

1. INTRODUCTION

In modern days, the impact of human activities to the ecosystem of the world have caused many countries and companies to seek new ways to lower their environmental impact. The rapid industrial development and economic growth combined with the advancements in technology have had their price paid towards the environment in a negative way. Due to this fact, a range of investments, initiatives, and regulations have been made to lower the environmental impact caused by the society. In particular, the impact of manufacturing different products has been under the focus. Within sustainable manufacturing, one of the common terms used is 'eco-design', which focuses on the environmental aspects of manufacturing certain product. The principle for the design is to minimize energy and materials used in the production and by saving resources.

During the 20th century, the usage of fibre reinforced polymer composites has been increasing due to the developments in composite technologies. With these high-performance materials, we have been able to manufacture objects with a low weight combined with high mechanical performance. However, traditional composites using glass or carbon fibres as reinforcements and thermoset polymers as matrix have their eco-friendliness reduced due to the petroleum-based matrix and poor recyclability of the products. To lower the environmental impact of composites, different natural fibres as reinforcing elements have been studied and are used at an increasing rate in different industries to lower the environmental impact of products.

My work was to re-design a radome shell structure for the Luxturrim5G project: manufacturing of 'sustainable prototype' for the project, re-design it through the materials selection, and testing novel sustainable composite materials. Luxturrim5G is a Finnish project which aims to develop and pilot technical and business models solutions and concept for 5G infrastructure with a pilot product integrated to smart light poles. Due to the high frequencies used, beyond the ultra-high frequency range (above several GHz), antennas and radios must be installed to a close proximity with each other's. In this project, 5G radios were chosen to be installed to light poles. In the light poles, the radios and antennas are installed to radomes called Camouflage Radome Units (CRU).

The work is divided to five main chapters, Theoretical background, Experimental section, Results, Discussion, and Summary. These topics are then divided to different subchapters. The chapter 2, Theoretical background, is divided to four different subtopics.

First one, chapter 2.1, focuses on sustainable design and how different aspects of sustainability are taken into account during a product design engineering. The second topic in Theoretical background, Sustainable fibre reinforced polymers, handles with fibre reinforced polymers from the sustainability point of view and, after that, deals with the basics of biopolymers and natural fibres. The third topic, Biodegradation in polymeric materials, looks more deeply to biodegradation that may happen in polymers and what can cause it. This topic is divided to three different subchapters, where the first one goes through the biodegradation happening in common pure polymers. The second one, Biodegradation of fibrous composites, goes through possible biodegradation that can happen in fibre reinforced polymers. Especially, the FRPs that use natural fibres as reinforcing elements are studied. The last topic under the chapter Biodegradation in polymeric materials, Prevention of biodeterioration, goes through different methods to prevent an unwanted biodegradation (biodeterioration). The last chapter in Theoretical background, Microwave-based communication technology, goes through the basics of cellular networks and, especially, the possible interactions between materials and a signal as it propagates through a medium.

The chapter 3, Experimental section, is basically divided to two different main topics, which are the manufacturing and testing of the novel sustainable composite material, and the other being the CRU case study. The first chapters go through the materials and methods used to manufacture the specimens during the work. After this, I focus on the testing methods used during the work. The last chapter of Experimental section, Camouflage radome unit, goes though the re-design process of the CRU and the fabrication of the sustainable prototype. The chapter 4, Results, goes though the findings done during the CRU case study, observations made during processing of the materials and, finally, the effects of soil burial on the composites. In the chapter 5, Discussion, I discuss and analyse about the findings done during the CRU case study and the biodegradation of the materials during the soil burial. Additionally, suggestions for future work are given based on the analysis of results. Finally, in chapter 6, Summary, I go through the main findings of the work to conclude the work in a concise way.

2. THEORETICAL BACKGROUND

2.1 Product design engineering

Product design simply aims to design new products. This field involves different kind of aspects of designing new products from its aesthetic to performance. The result of product designing is to conceptalize a product that has, for example, better functionality, more attractive appearance, better physical properties etc. Overall, product design plays important role in company that manufactures certain products to customers or to other companies. Depending on the final user of the product, often certain aspects of the design are emphasized such as making food products for consumers more appealing or increasing the physical properties and overall performance of industrial machines. (Rodgers & Milton, pp. 5-10) Product design is a very broad concept and it overlaps with other branches of designing like industrial design which especially focuses on mass-produced items and makes the possibility of mass production as central point during designing of the product.

Although word design applies especially to the outlook of the product, there is lot of engineering work behind it. To make functional products, materials used in it have to withstand the conditions it will be used in. Also, there might be certain legislations concerning the materials used in the product so any apparently functional material may not be suitable in that certain situation. Another point of view is the possible manufacturing processes to fabricate the product. Complicated shapes and the usage of certain materials in the product may prohibit certain manufacturing methods, which then must be taken into account when creating the concept of the product. Even designing a product that can be processed efficiently and has required physical properties may not be enough, because the products' whole lifecycle must be taken into account in the product design. This includes such categories as degradation of the material and end-of-life disposal. Also, design of the product must be optimized for increased profit. Although functionality of the product is very important thing, other aspects like availability, delivery speed, quality, and price must be taken into account to make the product more appealing for the customers. (Tabala & Roche 2005, 57-60)

In some cases, a product may be re-designed to modify certain properties of the product or plan something to be executed differently compared to the original concept. The re-designing is often referred as product re-engineering or product re-designing and it aims to change certain details in the product to improve certain features while maintaining the functionality of the product. The re-designing of existing products has become an important factor for the modern product development process. The reasons to re-design existing products are, for example, changing of the requirements of the customers, improving quality of the product, reducing its cost, or reducing its environmental impacts. [Smith et al. 2012].

2.1.1 Sustainable design

Nowadays, sustainable development plays an important role in the developing process between different fields of the human society. It is based on developing socially, economically, and environmentally sustainable actions. The sustainable development can be classified to certain framework consisting of different categories, which can be used to describe different strategies, actions etc. to develop sustainable systems. The first category, principles, are fundamental concepts that function as basis for actions and they often refer to simple activities. The next category, approaches, describe tactics combining certain principles to achieve sustainable development. The third category, sub-systems, connect certain approaches together and, the final category, sustainable systems, combine certain sub-systems. The sustainable systems can be considered as the highest level of activities to achieve the sustainable development. Finally, when we have the systems that are used in the sustainable development, the final phase is to create the sustainability policy, which gives the instructions what to do in certain situations. (Glavič & Lukman 2007)

Concerning my work here, one of the more important forms of activity is a sustainable design, which can be considered as an approach according to the sustainable development framework set by Glavič and Lukman (Glavič & Lukman 2007). In the traditional type of product design, the main goals have been the minimizing of costs and maximizing productivity. In the sustainable design, the approach is based on consideration of social, economic, and environmental aspects of the product throughout its lifecycle. In many cases, the sustainable design especially focuses on the environmental impact of the product, which have led to the usage of a term ‘eco-design’ that is based on principles of minimizing energy and material usage and reducing resource consumption (Veshagh et al. 2012). Some of the basic principles to achieve more sustainable design is to reuse, recycle and recover (RRR) materials in their end-of-life stage. The reusing means using certain product for the same purpose without any structural changes. The recycling means structurally changing the material and using it again. The recovery describes the processing of the residue (Seligar 2007, pp. 68-70).

In a normal cradle-to-grave –type design, the materials are allowed to downcycle which means that materials degrade when they are recycled. Another type of design, cradle-to-cradle / closed-loop material cycle (CLMC) –concept aims to combine zero waste and resource-efficient construction. The fundamental characteristics of the closed-loop cycles is the infinity of operational time as the materials are part of some sort of infinite loop. This differs from the cradle-to-grave type of designing as cradle-to-grave type allows downcycling of the materials and the aim is to just reduce the environmental impact of the product. Overall, the above-mentioned types of designs aim to increase the materials eco-efficiency which is a term used to describe the environmental impact of material. (Braungart et al. 2007) One example of the cradle-to-cradle type of material is steel used in construction since it can be recycled many times to make new structures without any major losses in its physical properties. Another example would be timber that can be biodegraded to form nutrients that can be then used as resources for new plants (Sassi 2008).

Often eco-design is linked with the life cycle assessment (LCA). As eco-design aims to develop products that have the lowest possible environmental impact throughout its lifetime, LCA is an environmental accounting and management approach that considers different aspects of the resource usage and environmental releases associated with an industrial system in a cradle-to-grave type of situation. It can be used as tool to evaluate different options and to compare materials, the kinds of energies are used to extract resources, the kinds of recycling methods to be used, and is the final end-of-life disposal of the material etc. to estimate materials environmental sustainability in the long run (EPA). LCA can be applied to study, for example, different materials or different processing methods and the information can be used as part in decision making processes.

As mentioned before, the sustainable development includes also the social and economic factors in addition to the environmental sustainability. Social sustainability takes into account the social factors, like basic needs, equity, access to education, quality of life, social capital, diversity, etc. (Åhman 2013) Economic sustainability refers to the form of sustainability that supports long-term economic growth without having negative impact on the social or environmental factors. This includes, for example, protecting freshwater resources and land areas (Ikerd 2012, pp. 1-3). Sustainable principles, approaches, and systems are combinations of these three different types of sustainability where most of the focus is especially on the environmental aspects of the product (Glavič & Lukman 2007).

Sustainable design can be applied from small objects to bigger constructions like buildings, and often sustainable design is divided to different categories depending on the target. Sustainable architecture is a term that describes the sustainable design of different buildings. In the sustainable architecture, such topics like heating systems, cooling systems, building sites, energy sources, and building materials are analysed. For the maximum eco-efficiency, different possibilities and environmental impacts of different elements have to be analysed. In addition to this, we can have sustainable urban planning, interior design, etc. However, one of the most important types of sustainable development concerning this work is the sustainable manufacturing.

The focus of the sustainable manufacturing is often on the environmental impacts of the manufacturing, but it also includes the social and economic impacts of the manufacturing method. To evaluate the impacts of the manufacturing process, LCA can be used to compare different options. The impact of the manufacturing process can be seen in many areas, like energy consumption, airborne emissions, water consumption, and waste treatment. Energy consumption describes the energy needed during the manufacturing process. Airborne emissions have impact on both social and environmental side of certain manufacturing processes. Different kind of emissions can have negative health effects on the workers but also emissions released during processes can accelerate the climate change. In processes where water is used as part of the manufacturing, for example, to cool or clean the machines, wastewater may be formed which then requires special treatments. The last area is the solid waste created during the process and the ways to utilize waste created during the process. (Haapala et al. 2013)

In applying sustainable design and manufacturing, there are certain advantages that promote the design and disadvantages which can function as barriers for the design. Advantages include compliance with different regulations. There might be certain requirements set for the environmental or social impacts of the manufacturing processes, which could lead to penalties. Also, poor performance in the sustainability can be a risk for the investors that could lead to reduced financial support for the product. Sustainable design can also lead to improved financial performance due to efficient usage of resources and relieve external pressure like lowering dependence of fossil fuels and through that, increase the financial stability within the manufacturing. As sustainable manufacturing improves the social impact of the activity, it may improve company’s reputation and motivate people working in the company. Sustainable manufacturing may also create market opportunities for the industry and competitive advantage over the rival companies. (Veshagh et al. 2012)

Although there are many advantageous aspects for applying sustainable manufacturing to a company’s policy, it also has some disadvantages and barriers. Often the sustainable strategies have high implementation costs as they may require higher investments to acquire the required equipment or procedure. There might also be a lack of customer demand so that the higher costs to produce more sustainable products will not create enough value for the customer when making the buying decision. If alternative technologies are used to manufacture the products, they might cause problems as the technology might be complex or unreliable. Other barriers in the implementation of the sustainable strategies can include lack of in-house knowledge or time constraints as the implementation can be a long process and the support to implement the strategies can decrease over time. (Veshagh et al. 2012)

2.2 Sustainable fibre reinforced polymer composites

Traditionally, the disposal of fibre reinforced polymers has not been a sustainable process and the most common way to handle FRP waste is still landfilling. The second most common way to manage FRP waste is to incinerate it and turn the heat to energy. However, the costs of the incineration are higher compared to landfilling and incineration facilities are more likely to charge more due to the high calorific content, high amount of residue (ash and fibres), and the toxic emissions of traditional FRPs. Only a certain amount of FRPs can be incinerated at a time to avoid emissions overloading the filtering systems in the power plant. Also, the mineral content of the glass fibre reinforced polymers is relatively high so a lot of ash is formed during incineration and it will be required to be dumped to landfills, for instance. However, glass fibre reinforced polymers have been used at cement-kilns as a secondary element to provide both heat and minerals for the process. At the moment, this method is seen as one of the more sustainable ways available to recycle GFRPs. (Yazdanbakhsh & Bank 2014)

The third way to manage FRP waste is recycling. Compared to thermoplastics, thermosets cannot be just reheated until they start to flow which would allow for reshaping, for example. At the moment, there are basically two commercially available ways to ‘recycle’ FRP composites. The first one is to extract valuable fibres from the matrix thermally or chemically, and the second way is to mechanically recycle FPRs – a process that involves breaking down the composite through mechanical processes like milling or shredding. The first method can be quite expensive thus making it suitable only for valuable reinforcing fibres like carbon fibres. Second method, mechanical recycling, provides powder like substance which can be used as filler for other manufacturing methods. (Yazdanbakhsh & Bank 2014) The methods used to extract fibres include pyrolysis-based techniques and solvolysis. Pyrolysis is based on burning the matrix away from the composites and solvolysis is based on using chemicals to solve the matrix away. However, the mechanical properties of the recycled fibres are reduced during any of these recycling methods that makes them unsuitable to replace virgin fibres. This leads materials to be used in any other lower level of technology (or anisotropy) which then, unfortunately, prevents the closed-loop system. (Oliveux et al. 2015)

Traditionally, the raw materials used to manufacture polymers have been extracted from fossil-based sources. Moreover, most of the commercially used plastics have very stable structure so that degradation of the plastics at atmospheric conditions is a slow process. These factors have made it very attractive to research and develop environmentally more sustainable materials, like the research and development of so-called biopolymers. As a term, biopolymer can have many meanings that depend on the field of application, raw materials and biodegradability. The term bio-based means that biomass is used to make the plastic. Biomass in this case means non-fossilized and biodegradable materials extracted from animals, plants and micro-organisms (Kabasci 2013, 2). Another commonly used term to describe properties of polymers is biodegradability. In biodegradation, micro-organisms or the thermos-chemical environment in an organism can decompose the material where in normal degradation plastics are degraded or weathered through abiotic factors like UV-light, heat or mechanical stresses (Fried 2014, 264-274). In Figure 2.1, it is presented a coordinate system covered by European bioplastics -association to categorize plastics based on their biodegradability and source. Overall, biopolymers can be divided into three different groups: polymers that are bio-based but not biodegradable, ones that are fossil-based but biodegradable and the ones that are both bio-based and biodegradable.

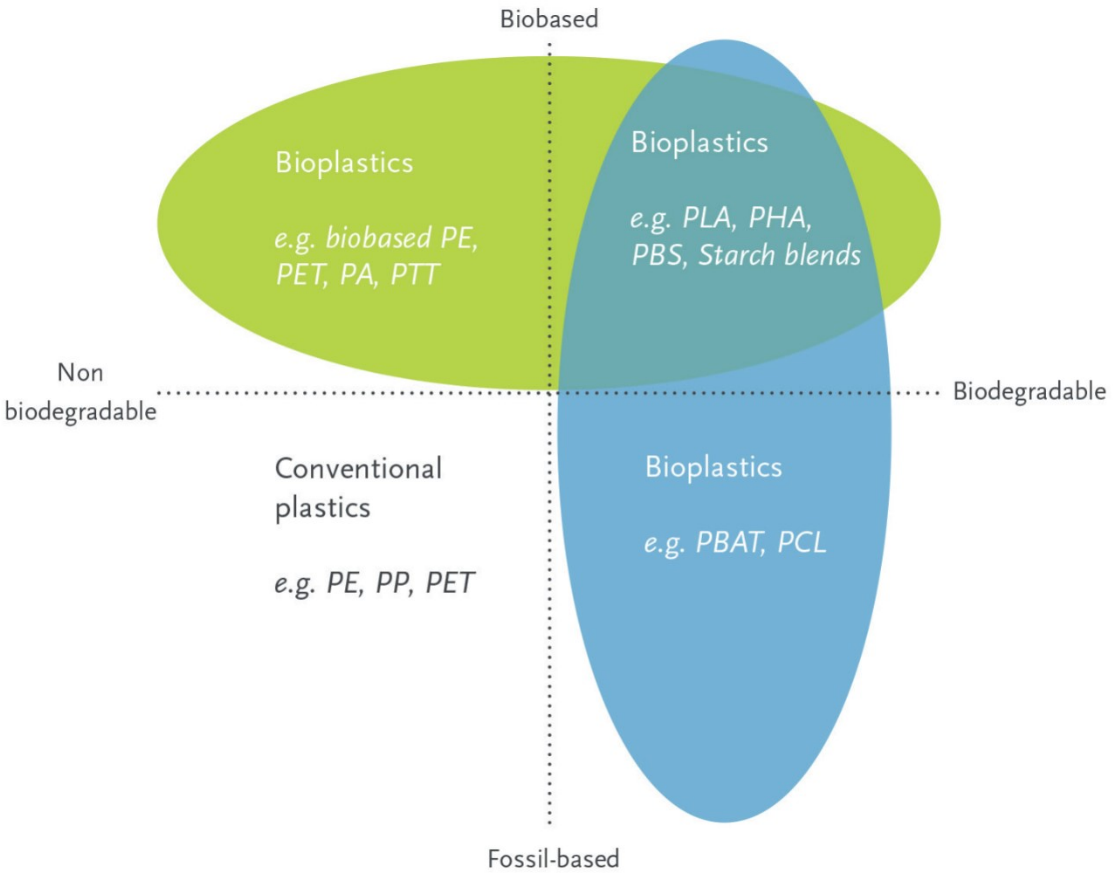

Figure 2.1: *Simple graph describing biopolymers (European bioplastics)*

Fibre reinforced polymers using thermoplastic matrices can be manufactured via several processing methods. The processability of the composites using thermoplastic matrix is also dependant on the form and size of the reinforcing fibres. For discontinuous short or long fibre reinforced composites, the common processing methods include extrusion and injection moulding. However, when continuous fibres are used in components, the amount of processing methods are more limited to basically three common methods: pultrusion, thermoforming, and compression moulding. (Peters 1998, 529) Most common commercially used thermoplastics in continuous-fibre reinforced high-technology polymers are polyetheretherketone (PEEK), polyetherketoneketone (PEKK), polyphenylene sulfide (PPS), and polyetherimide (PEI). In addition to these, polyamide (PA), polypropylene (PP), polyethylene (PE), and polyethylene terephthalate (PET) are being massively used in automotive industry for discontinuous fibre reinforced polymers.

Typically, the fibres used in composites are synthetic glass, aramid or carbon fibres. However, due to the current situation to create more environmentally sustainable products, natural fibres are seeing increasingly larger markets. There are couple of reasons for this, one being availability of sources all over the world though agricultural fibres and wood. The second reason is the good availability of data concerning their processing, structure and properties. The third reason is the ecological concern of non-sustainable materials like the fossil-based rivals, and the fourth being the legislations set for commodity products. (Satyanarayana et al. 2009)

Natural fibres can be divided into three different basis groups depending on the origin of fibre: plant fibres, animal fibres and mineral fibres. These can be divided to different subgroups. (Rowe 2009, 7). Plant fibres generally consist of cellulose, and can be further on categorized based on their origin. The categorization can be done in different ways depending on the part that is used from the plant. According to one source, plant fibres can be divided to seed fibres collected from seeds, leaf fibres collected from leaves, bast fibres collected from the bast of certain plants, fruit fibres collected from fruits, and stalk fibres that are stalks of certain plants including wood fibres. Animal fibres consist mainly of proteins and can be categorized to two main groups: animal hair like wool, feathers, and leather, as well as fibres produced by insects like silk. (Mohanty 2005, 6-8), (Chandramohan 2011), (Rowe 2009, 7) The Classification of natural mineral fibres is heavily dependent on the source of the information. According to some sources, mineral fibres have to occur naturally but occurring to some other sources, minerals in the fibres can be slightly modified and still are treated as natural fibres.

The main components of the outer layers of the plant fibres consist mostly of cellulose, hemicellulose, and lignin (Summerscales et al. 2010). Cellulose is a polysaccharide that consists of 3000 or more glucose units attached together and it is the most abundant naturally occurring organic matter. It is structurally the most important compound in the fibres because it provides the fibres part of their strength. (Ott 1954, 7), (Summerscale et al. 2010) Hemicellulose consist of different kind of polysaccharides that differentiate from cellulose. They surround the cellulose fibres in the plant cells and their basic function is to strengthen the cell walls by interacting with the cellulose and the lignin. Hemicellulose does not function as structural part of the fibre due to its low strength. (Scheller 2010), (Summerscale et al. 2010) Lignin is polyphenolic material created through polymerization of its three main components, i.e. oniferyl alcohol, sinapyl alcohol, and p-coumaryl alcohol. The main function of lignin in the plant is to transport water nutrients and metabolites while giving plant walls their rigidity and acts a binder between wood cells. (Vanholme 2010)

Bast fibres are one subgroup of the plant fibres which can be collected from outer cell layer, phloem, surrounding the stem. Common bast fibre sources are the agricultural plants which are processed further on to extract fibres from the plant’s other parts like xylem and epidermis (Sinclair 2014, 43). The typical production cycle for bast fibres, in this case flax, starts with tillage which prepares the land for the planting of the seeds. The tillage often consists of ploughing the land and spreading certain chemicals like acidity regulators and fertilizers on it. After preparation, the seeds are planted and let grow. The full-grown plants are harvested using a harvester or by pulling and flax seeds are removed (Summerscales 2010). After this, the plants are exposed to certain kind of treatments so that the fibres can be extracted from the stems. This phase, called retting, can be done through different methods that can be divided into different categories depending on the method used: biological, mechanical, physical, and chemical. Biological retting can be further divided into artificial and natural methods. From natural methods, the dew-retting is the most common one. In dew-retting, after harvesting the plants, stalks are left on the field so that the atmospheric conditions combined with bacteria ferment the stem away. In water-retting, stalks are submerged in water that swells the inner cells, bursts outermost layer and increases absorption of moisture and bacteria (Mohanty 2005, 52-55), (Retting 2019). After the retting, fibres have to be extracted from the straws and this action is done in three phases including: breaking, scotching and heckling. In the breaking phase, the flax straws are broken to smaller pieces without harming the fibres. In the scotching phase, the pieces of the straw are scraped off from the fibres. Finally, in the heckling phase, fibres are pulled through ‘hackles’ which comb the fibres. (Sinclair 2014, 44) In table 2.1, the chemical composition and moisture content of some common bast fibres are shown as reported by Siakeng et al. *(Siakeng et al. 2016)*

Table 2.1: *Chemical composition of some bast fibres as reported by Siakeng et al. (Siakeng et al. 2016)*

Fibre type	Flax	Hemp	Jute	Kenaf	Ramie
Cellulose (%)	71	68	61-71	45-57	68.6–76.2
Hemicellulose (%)	18.6–20.6	15	14-20	21.5	13-16
Lignin (%)	2.2	10	12-13	8-13	0.6-0.7
Moisture content (%)	8-12	6.2-12	12.5-13.7	-	7.5-17

Bast fibres are relatively thick and have high tensile strength which make them suitable to be used in coarse textiles like ropes, carpets, geotextiles, and in fibre reinforced composites (Sinclair 2014, 43). In modern days, bast fibre reinforced composites are used in automotive industry, for example. Due to the lower density of the reinforcements, decent weight-normalized mechanical properties, sustainability, recoverability, and price, bast fibres can be used in certain technological parts instead of traditional reinforcements like glass fibres. Due to the low density and other benefits of NFRPs, composite parts made from natural fibres can replace GFRPs in some cases. Also, different legislations require certain percent of cars mass to be recyclable or recoverable which can be seen as advantage for NFRPs when compared to GFRPs (Pandey 2010). Another bast fibre, jute, can be mixed to cement used in construction applications. It has been shown that jute fibres in cement and concrete increase impact resistance and ductility and can function as crack arrestors (Fan 2016, 209-211).

In table 2.2, certain crucial properties of the most common and important bast fibres, flax, hemp, jute, kenaf, and ramie, and the values of e-glass are listed. For natural fibres, mechanical properties are heavily dependent on the processing steps during the production cycle, and the fibres come in a large variety of quality so that the tensile properties between fibres have major differences. In addition to this, fibres appear in bundles so that the number of fibres also affect the final mechanical properties, which is different compared to a single fibre element. (Marrot et al. 2013) There are major differences even in the same type of product produced by different manufacturers. The size of fibres describes the average length of single fibre. When bast fibres are used commercially, fibres appear in bundles held together by gum to make them longer which then can be processed further. (Sinclair 2014, 45-47)

***Table 2.2:** Mechanical properties of common bast fibres and e-glass. The values for the Young's modulus and tensile strength for NFs are for both single fibrils and whole fibers*

Fibre type	E-glass[1]	Flax[2,3,4]	Hemp[3,5,6,7]	Jute[3,8,9]	Kenaf[10,11]	Ramie[3,12,13,14]
Density (g/cm^3)	2,58	1,5	1,5	1,5	1,2	1,5
Young's modulus (GPa)	76-81	12-85	15-90	28-32	39-41	15-128
Tensile strength (MPa)	3450±350	979±360	700±250	560±200	700±200	669±269
Specific modulus (E / Density)	30	32	20	30	33	
Elongation (%)	4,5-4,9	2,4-4,0	0,8-3,0	1,5-2,5	1,1-1,8	1,2-3,8
Average size of a single fibre (mm)	-	25-30	30-40	3-6	20-60	60-250

From the mechanical point of view, glass fibres tend to offer higher mechanical performance compared to bast fibres. However, when the density of the material is used for normalizing the performance, natural fibres can compete with traditional e-glass so that

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[1] Wallenberger & Binigham 2010, 127

[2] Baley 2002

[3] Sinclair 2014, 45-50

[4] Yan et al. 2014

[5] Placet 2009

[6] Marrot et al. 2013

[7] Bunsell 2009, 79

[8] Virk et al. 2009

[9] Tripathy et al. 2000

[10] Ochi 2008

[11] Mahjoub et al. 2014

[12] Mohanty et al. 2000

[13] Lodha & Netravali 2002

[14] Kim & Netravali 2010

we can achieve same stiffness even at a lower weight. Cost vice, natural fibres are very compatible compared to the traditional synthetic fibres like e-glass and carbon fibres. In sliver form, bast fibres are less expensive than synthetic fibres and overall, they are a very abundant source of reinforcing fibres. Finally, an advantage over synthetic fibres is the environmental factor and the damping properties. Natural fibres come from natural sources and they are completely biodegradable materials. These properties can be used to create sustainable composites to meet different legislations and offer greener options for different products compared to traditional fibre reinforced polymers. For these reasons, automotive industry is using more and more NFRPs in different parts of cars.

There are still many disadvantages in the usage of natural fibres which inhibit the usage of natural fibres in different applications. Due to their hydrophilic nature, without any chemical or physical treatments they are unsuitable to be used efficiently with traditional polymer matrices that are hydrophobic. Natural fibres absorb water heavily, which worsens the mechanical properties of the fibres due to swelling of cellulose macromolecules and may decrease the adherence between the fibres and the matrix. Although natural fibres itself are cheap, different fibre treatments and processing techniques may create additional costs to use natural fibres as reinforcing elements in FRPs. These processing steps include, for example, spinning of fibre sliver to create roving and woven fabrics. Also, there are some uncertainty with supply and usage of natural fibres, as the markets are anyway relatively low and the quality of fibres vary depending on the source of the fibres. This way the mechanical properties of these fibres between suppliers aren't homogeneous. It can also be noted that natural fibres have rather low thermal stability which prevents their usage in applications where temperatures over 170 ˚C may occur (Gassan & Bledzki 2001). The low thermal stability may also prohibit the usage of natural fibres with certain thermoplastic polymers which have relatively high processing temperatures.

2.3 Biodegradation in polymeric materials

Degradation of polymers describes a situation where polymers get modified or disintegrate through certain processes under specific environment. The degradation processes can happen when polymers are exposed to high temperatures (thermal degradation), high mechanical stresses (mechanical damage), oxygen and ozone, high frequency electromagnetic radiation (photodegradation), moisture and chemical agents (chemical degradation and leaching). Specific degradation processes may or may not be a desirable property of material. For example, exposure to UV light in atmospheric conditions may lead to chain breakage which then worsens the mechanical properties of the polymer. (Fried 2014, 264-274) In many cases, polymers degrade when they are recycled and processed due to high temperatures and oxidation during (melt) processing (Birley et al. 1992, 477-479).

Degradation in polymers can also occur biologically. In biodegradation, micro-orgasms in the environment breakdown the polymer chain fragments into water, carbon dioxide, biomass and humus. Ultimately, the biomass and the humus are mineralised to different minerals during the process. For any material to be classified as biodegradable material, the biodegradation must happen through certain disposal path, like composting, in certain set of time (Bastioli 2014, 3-4). This type of degradation can be utilized as environmentally friendly method to dispose waste thus lowering the amount of waste with very long decomposition time (at landfills). However, biodegradability can also be a major problem in applications with long operation time because it may lead to premature failure of the material. In these cases, often two terms are used to describe the nature of the biological degradation: biodegradation and biodeterioration. Both describe disintegration of the polymer caused by micro-organisms but biodeterioration as a term emphasizes that the breakage is unwanted. (Janda et al. 2015, 5-7)

End-of-life options for the biodegradable polymers vary from traditional ones used for non-biodegrading polymers to specific ones that can be only applied to biodegradable polymers. The common one is landfilling, which is not recommended for biodegradable polymers due to the release of greenhouse gasses, like methane, which then can not be utilized efficiently. One option, which is used commonly for traditional plastics, is incineration. Basically, in this process material is decomposed at and elevated temperature to smaller compounds with or without oxygen atmosphere and the released heat is turned into energy. Incineration can be seen as a favourable method to dispose bio-based polymers as the ingredients are CO_2 neutral materials. To recycle the polymers, a commonly used recycling method is the thermo-mechanical recycling. It is used for traditional polymers and it can be used also for biopolymers but less successfully due to the stronger degradation of average bioplastic during the heat cycles compared to the traditional plastic. Another recycling method, chemical recycling, could be efficiently utilized with biopolymers, so that with less effort material could be turned back into raw materials. (Endres & Siebert-Raths 2011, 225-232)

Biodegradable polymers can also be disposed through aerobic or anaerobic decomposition process, where the degradation is executed by micro-organisms. The difference between these two is that in the aerobic degradation oxygen is present and in the anaerobic it is not. The lack of oxygen also affects to the micro-organisms living in the environment, which then affects to the potential biodegradation of different materials. The environments for biodegradation can be further down divided into two categories: aquatic and high solids. The biodegradation rate in different environments are influenced by the presence of micro-organisms, availability of oxygen and water, temperature, and chemical agents, like pH and electrolytes. (Bastioli 2014, 5-7)

In aerobic degradation, known as composting, moisture and elevated temperature combined with the microbial activity break the polymer chains to smaller fragments and eventually to carbon dioxide, water, minerals and organic matter like compost and humus (Rudnik 2010, 11-12). The biodegradation can be divided into two phases: in the first one, material is fragmented to smaller pieces through mechanical, chemical, or thermal degradation. The fragmentation can also be executed by biological agents. This leads to increased surface area, which can be attacked by microbes more efficiently. In the second phase, micro-organisms start to digest the product to smaller compounds (Kyrikou & Briassoulis 2007). For effective composting, it is necessary to have suitable temperature, available oxygen concentration, pH, moisture content and correct ratios between elements, mainly carbon and nitrogen. Compost that is formed after processing can be directly used as fertilizer, manufactured topsoil, mulch, and growing medium in agriculture. (Rudnik 2010, 93-96)

Another method used to treat organic waste is anaerobic digestion known as biogasification. In this process, micro-organisms break the organic matter in the absence of atmospheric oxygen resulting in methane and carbon dioxide. The methane created in the process can then incinerated to produce electricity, for example. This end-of-life treatment method has been used to treat organic waste such as sewage sludge, animal waste, agricultural waste and food waste. (Mudhoo 2012, 2-3) Anaerobic degradation has also potential to be a viable method to dispose biodegradable polymers efficiently but it is still in its early stages of development (Endres & Siebert-Raths 2011, 238-239). Both composting and biogasification are commonly used methods to dispose organic waste. The end product of composting, the compost, can be used as efficient fertilizer but the heat formed during composting is hard to collect. On the other hand, biogasification produces biogas containing mostly methane (and CO_2) that can be used to produce energy (Bátori et al. 2018).

For testing biodegradability of plastics, there are several standards that apply different conditions to the exposure. The standard, which is used in testing, is chosen according to the application of that certain plastic and the conditions it is exposed to during the design life cycle. Different kind of exposure conditions used in different testing standards include compost, soil, sewage, and water. Depending on the testing standard, monitored parameters include formation of gasses, like CO_2, weight loss, degraded mechanical properties, changes in the molecular structure, and the oxygen consumption. Respirometer is often used to monitor the formation or consumption of gaseous compounds during the testing. Some commonly used standards to evaluate aerobic biodegradation in composting conditions include ASTM D5338, ASTM D6002, EN 13432, and ISO 14855. For biodegradability in soil, commonly used standards include ASTM D5988 and ISO 17556. For marine exposure conditions, standards like ASTM D6691, ASTM D6692, ISO 15314, and ISO 16221 are used. (Ammala et al. 2011) For anaerobic degradation, standards like ISO 14853, ISO 15985, and ASTM D5511 can be applied to plastics (Bátori et al. 2018).

To analyse materials that have response to bacteria, the disk diffusion test, known also as agar method, can be used. In disk diffusion test, we have a disk covered with agar gel and specimens laying on top of agar. The agar functions as optimal substrate for bacteria to grow and the environment is kept at constant temperature and relative humidity. Specimens are kept in the containers for several weeks, if needed, and after that, agar near specimens is evaluated visually and by using microscopy. If material has inhibiting properties against the micro-organisms, there will be certain area near the specimen, where there is increase in the growth of the bacteria or lack of bacteria.

In the next three chapters I will go through biodegradation of pure polymers, polymeric composites, and finally the prevention of the biodegradation. The first chapter, Biodegradation of pure polymers, deals with biodegradation of pure polymers. The next chapter, Biodegradation of fibrous composites, goes through how biodegradation affects polymer composites and especially the ones that use natural fibres as reinforcements. In the final part, Prevention of biodeterioration, deals with unwanted biodegradation that may happen throughout products' lifecycle and discusses how to prevent it.

2.3.1 Biodegradation of pure polymers

Micro-organisms, that cause most damage to plastics, can be divided into three different groups: bacteria, fungi, and algae. Fungi can be further divided to mushrooms, yeasts and mould. From the three different groups, bacteria degrade plastics by using certain compounds in them as food, which may cause macroscopic staining and pitting. Fungi can degrade the inner structure of different materials, which may lower their mechanical and physical properties. Algae on the other hand does not damage polymers as itself but may collect water and act as medium for fungi (Nichols 2004, 4-6).

For microbes to digest plastics, there are certain requirements to be met. The first one is the environment of plastic, so that micro-organisms can flourish there and natural elements, like moisture and sunlight, are in a suitable balance. The second factor is a high surface area to mass ratio so that enzymes produced by micro-organisms affect larger areas and in overall more ends of the polymer chains are available for the micro-organisms. The third factor is that the polymer must contain certain chemical groups, like esters (Fried 2014, 277), which can react with the organisms (enzymes). Polymers have to be wettable so that the enzymes and the micro-organisms can degrade the plastic (Kyrikou & Briassoulis 2007).

Many of material properties have an impact on the biodegradability and in different ways. Important factors during the biodegradation of plastics is the surface in general. Factors like surface roughness, water absorption, porosity, hydrophobicity, photochemical activity, and compounds in the surface have an impact in biodegradation. The first-mentioned surface roughness, and porosity, and water absorption eases the adherence of the micro-organisms on the surface which leads to higher colonization rates. Hydrophobicity especially affect the functionality of the enzymes released by the micro-organisms. The enzymes released by the micro-organisms are too large to penetrate to bulk material but they can function in the surface and erode the material away. High molecular weight decreases the rate of process, as less polymer chain ends are available for the micro-organisms (Birley et al. 1992, 486-487). A higher crystallinity leads to lowered degradability, as the oxygen and in general moisture permeability is lower inside crystalline plastics (Kyrikou & Briassoulis 2007) and it has been shown that in biodegradation the amorphous regions degrade first. The pH of the system can affect the growth of micro-organisms and for biodegradation to happen, the conditions must be optimal for that certain micro-organism to act. (Janda et al. 2015, 67-70)

Traditional synthetic thermoplastic polyolefins do not in practice biodegrade or the biodegradation happens over hundreds of years. They pose many aspects, which make them very bioinert. Pure hydrocarbon structure prohibits hydrolysis well, and these polymers are hydrophobic, so enzymes produced by micro-organisms can not react with the polymer chains. Although polyolefins can degrade through oxidative methods, which would then expose the chains to microbes, often antioxidants are added to traditional polyolefin compounds. In addition to the chemical structure of polyolefins, they often have a high molecular weight and high level of crystallinity. A high molecular weight causes less polymer chain ends to be exposed to the micro-organisms and the long chains are unable to enter the cells of the microbes (digestion). A high crystallinity offers low oxygen permability so there is less oxygen available for the degradation to happen, i.e. microbes to live. (Kyrikou & Briassoulis 2007)

For polyolefins, the term oxo-biodegradation can be used to describe their biodegradation process. As mentioned before, for micro-organisms to digest the polymers, the polymer has to be fragmented so that the micro-organisms have access to the ends of the polymer chains, and the enzymes must be able to react with the polymer. This often requires formation of carbonyl groups at the end of the chains that can happen for polyolefins through photodegradation, thermal degradation and with mechanical stresses. In photodegradation, UV light combined with oxygen in the atmosphere breaks down the polymer backbone to smaller fragments, which are then exposed to the micro-organisms. In thermal degradation, this happens at high temperatures. The chain structure of the polyolefins affects also the degradation rate. Higher branching leads to higher degradation rates, as branching increases the permeability of different gaseous elements and a branched structure contains more tertiary carbon atoms, which are necessary points in degradation process. For this reason, LDPE has higher degradation rate compared to LLDPE and HDPE. (Ammala et al. 2011)

Biodegradation of pure PE have been tested in couple of studies by burying the material in soil or to a compost. Otake et al. buried a sheet of polyethylene to soil for 32 years (Otake et al. 1995) and during the time, the sample was partially degraded. According to different studies done to polyethylene, the degradation of 60 µm thin LDPE film would take 300 years. The (bio)degradation rate can be further increased by adding pro-oxidants to the polymer system as shown by Corti et al. (Corti et al. 2010). LLDPE samples were exposed to sunlight, thermoaged, and finally screening tested using fungal strains. Samples containing pro-oxidants had increased amount of carbonyl groups in their structure and showed increased degradation rates during the screening tests. Also, samples that weren’t exposed to micro-organisms showed a decrease in the degradation. Husarova et al. studied the effect of pro-oxidant additive on the biodegradability of pure LDPE and LDPE containing calcium carbonate (Husarova et al. 2010). The rate of the oxidation was decreased when calcium carbonate was present in the system and in the end of oxidation process, average molecular weight was higher compared to the pure polymers. In a compost environment, pure LDPE has reached higher mineralization with a faster pace, but when exposed to soil environment, biodegradation of pure LDPE stopped at 7% where LDPE containing fillers were mineralized 13% for 40 days oxidation and 16% for 80-days oxidation.

The increased biodegradability caused by the pro-oxidants have been observed also on other polyolefins than just polyethylene although they haven’t been so heavily under the focus. Fontanella et al. compared different kind of polypropylenes with a low content of polyethylene and various pro-oxidant Mn/Fe, Co and Mn metal salts. The pro-oxidants, other than Co based metal salts, were observed to increase the biodegradability although the biodegradability rate of PE was still higher (Fontanella et al. 2013). Polystyrene has been observed to biodegrade through formation of carbonyl groups to its structure and the biodegradability of PS was observed when pro-oxidants were added to the system. However, the degradation rate is decreased due to the phenyl groups in the chain (Ojeda et al. 2009). The mechanics of biodegradability of PE, PP, and PS happen through oxo-biodegradation but it has been shown that PE is able to go through hydro-biodegradation when catalysed with transition metals and if certain type of micro-organisms are present (Bonhomme et al. 2003).

Traditional polyolefins have also been mixed with biodegradable materials, like thermoplastic starch, to study its effect on the biodegradability. Ruiz et al. blended polyethylene with starch with different ratios and then composted the blends for 125 days. Up to certain point, blended starch had positive effect on biodegradability which may have been caused by fragmentation of polyethylene to smaller particles when starch degrades away which then makes polyethylene more vulnerable to the micro-organisms. (Ruiz et al. 2011) Studies executed by Li et al. showed an increased biodegradability when thermoplastic starch was blended with LDPE so that about 25% of blend was mineralized (Li et al. 2011). Valle´s-Lluch et al. studied the morphological properties of PE blended with Mater-Bi®, a starch-based blend. It was found that Mater-Bi® could hinder the uniform growth of crystallites in the polymer during processing (Valle´s-Lluch et al. 2001). Other studies made for polyolefins have shown that the blending of biodegradable materials does not affect the degradation of the polyolefin or so that only the additive degrades (Ramis et al. 2004), (Pang et al. 2013)

Polyethylene terephthalate is one of the most used polymers in the world in addition to PE, PP, PS, and PVC. Although PET is petroleum-derived synthetic polymer like previously mentioned PE, PP, and PS, it is part of polyester family where the other three are polyolefins. It differs from the other three for having ester group in its repeating unit, which are more susceptible to biodegradation. However, the high ratio of terephthalate units in the polymer chains reduce the hydrolysability of the ester links. The hydrolysation of the ester links has been major topic on the area of biodegradability of PET to find enzymes that are able to attack the polymer chains of PET and to date, there have been numerous findings on the enzymes able to attack and degrade PET (Taniguchi et al. 2019). Most remarkable discovery was done by Yoshida et al. who were able to find and isolate near PET bottle factory species of bacteria which could hydrolyse PET through two types of enzymes (Yoshida et al. 2016).

Thermoset plastics are not registered as biodegradable plastics, but they are still able to go through certain amount of biodegradation when exposed certain species of micro-organisms. Wang et al. exposed epoxy resin coating to sterile seawater and seawater containing *Pseudomonas putida* bacteria. It was found that the seawater containing micro-organisms decreased the corrosion resistance of the epoxy resin compared to pure seawater (Wang et al. 2016). Also, Eliaz et al. observed microbial degradation of an epoxy resin by a set of microbes (Eliaz et al. 2018). In future, it might be possible to biodegrade cured epoxy resin but it would be a challenging task. Thermoset polyesters have ester groups in their polymer chains but the polymers itself are still very resistant against hydrolytic degradation. However, some studies have been able to identify microbial degradation of polyesters (Ying et al. 2005).

Polymers mentioned above are non-biodegradable materials. In modern times, when concerns about plastic pollution are rising, there has been development of biodegradable polymers like thermoplastic starch, polycaprolate, and polylactic acid. Biodegradable polymers go also through the two-step process, where first the polymer disintegrate and the backbones are fragmented by hydrolysis or enzymes into smaller polymer chains. This is followed by the second phase, where chains enter the cells of the micro-organisms that digest the polymer chains to produce water, carbon dioxide, and biomass. Generally, biodegradable plastics chemical structure contains certain bonds that are easily breakable through hydrolysis or enzymes. The fragmentation in the first step is most of the time caused by hydrolysing so that water breaks the polymer chains. For this reason, most of the biodegradable plastics contain ester, lactose, or ketone groups which are sensitive to water. (Bastioli 2014, 174-178)

Starch is a natural material produced by vegetables and plants during photosynthesis for their food reserve. After cellulose, it’s one of the most abundant biomass materials in the nature that can be found in stalks, crop seeds, and plant roots. Main sources for starch are, in order from the most to least in the production rates, corn, wheat, potatoes, and cassava. It can be used to create starch-based plastics with almost 100% utilization rate, or it can be used as a source of smaller compounds for a polymer synthesis with about 45% utilization rate. Starch is a form of polysaccharide than consist of amylose and amylopectin macromolecules. In its pure form, starch ca not be used to make plastic products due to melting range of the crystallites formed by its compounds which have their degradation temperature below melting range. For this reason, the starch must be modified chemically and/or certain additives must be added to the system to create the starch-based plastics. (Bastioli 2014, 266-270)

Starch in its pure form have been used as filler in synthetic polymers like PE to enhance its degradability in the soil so that degradation of the starch leads to porosity and disintegration of the polyethylene. Modifying natural starch to thermoplastic form is carried out through mechanical or thermal mechanisms where the amount of water in the system has a major impact on the material. The main goal is to destroy the crystallinity in the starch molecules in aqueous solution through gelatinisation, like in water, and by heating the system. (Bastioli 2014, 270-272) If the concentration of the plasticizer is correct, the end product is a thermoplastic starch. In this form, the starch is malleable using heat as instead of having starch-starch contacts in the system, small plasticizers penetrate between the molecules which leads to starch-plasticizer contacts. During aging of the TPS, plasticizers exit from the matrix to the surface of the polymer that leads to retrogradation, which is recrystallization of the amylose and amylopectin. (Nafchi et al. 2013) In TPS, the number of amorphous regions is about 90% and the crystallinity is around 10%. As TPS goes through retrogradation, its mechanical properties change significantly. Higher crystallinity increases the ultimate tensile stress but decreases materials elongation and makes it more brittle. The retrogation is often considered as an unwanted process as it changes quality, acceptability, and shelf-life of the products made from the TPS. (Zhang & Rempel 2012)

Like many of the biodegradable plastics, TPS goes through the two-step degradation process. In the first phase, TPS is fragmented through hydrolysis in the presence of amylases, one type of enzymes produced by bacteria. Compared to some other biodegradable materials, starch-based materials can go through significant biodegradation even in a home composter (Czaja-Jagielska & Melski 2013) and it’s able to degrade anaerobically compared to other biopolymers, like PLA or PCL (Mohee et al. 2008), (Massardier-Nageotte et al 2006). One of the aspects in using biodegradable materials is the ecotoxicity of the compounds when material biodegrades. For thermoplastic starch containing plastics, such as Mater-Bi®, the ecotoxicity has been tested for in soil containing several types of bacteria. It has been found, that compounds released when Mater-Bi® degraded were not harmful to any of the bacteria in the soil. (Sforzini et al. 2016)

One of the most common biopolymer in commercial usage and one which has potential to replace some more common petrochemical based polymers, like PE, PP, PS and PET, is polylactid acid. It is used today in both packaging and medical applications at growing rate and could be used in future to produce fully compostable biocomposites. PLA has relatively good mechanical properties and its tensile strength and Young’s modulus are higher than polyethylenes or polypropylenes. However, due to the low impact resistance, low toughness and low heat distortion temperature of PLA, applications in the engineering field are somewhat limited. (Piemonte 2012, 2-4).

PLA has been confirmed to biodegrade in soil and compost environments though hydrolysis. Biodegradation of PLA starts with hydrolysis so that the chains are broken to smaller segments and the smaller chains can be then consumed by the micro-organisms. There have been many studies on the effect of bacteria and enzymes to the degradation rate in the first phase, but it’s not still clear how the micro-organisms affect the depolymerisation step. (Karamlioglu et al. 2013) It has been reported by Hakkarainen et al. that PLA degraded to fine powder in a biotic environment whereas in an abiotic environment fragmentation was not observed. The degradation rate is also affected by the crystalline and isomeric structure of PLA so that PLA containing L-isomer lactide units had higher degradation time compared to D-isomer. (Hakkarainen) Although PLA can be classified as biodegradable material, the biodegradation rate in a normal house compost or soil is very slow. Also, in ambient temperature PLA is not very vulnerable to microbial attacks and only certain bacteria and enzymes are able to breakdown PLA. (Tokiwa & Calabia 2006)

2.3.2 Biodegradation of fibrous composites

This chapter mainly focuses on the biodegradation of FRPs that use natural fibres as reinforcing elements. Traditional synthetic fibre reinforced composites, like carbon-epoxy composite can’t be degraded by micro-organisms. However, micro-organisms can cause some biodeterioration in the material, for example by disrupting bonding between fibres and the resin (Wagner et al. 1996). For this reason, this chapter mainly focuses on the biodegradability of composites using natural fibres as reinforcing elements which has been under focus and many studies have been published where researchers were using different natural fibres with different matrix materials. As mentioned in chapter 2.2, natural fibres come from different origins which affects the chemical composition of that certain type of fibre. Plant-based fibres, like flax and hemp, mainly consist of cellulose where animal-based fibres are proteins. This then affects to the degradation mechanisms involved during biodegradation.

Plant-based fibres consist mainly of cellulose, hemicellulose, and lignin, which are complex systems of polysaccharides. These polysaccharides are susceptible to different micro-organisms, which can form a biofilm on top of the fibres in the presence of moisture, heat, and dirt. After formation, they start to degrade the cellulosic fibres. During the degradation, micro-organisms spread enzymes which catalyse the depolymerisation reactions. It has been observed that there is a difference in the mechanisms between biodegradation caused by fungi and the degradation caused by the bacteria. Bacteria degrades material away from the surface where fungi penetrate inside the cellulosic fibres through cracks and starts to degrade it inside out. The cellulosic fibres degrade in soil very fast as demonstrated by Simončič. Untreated cotton fibres were buried in the soil for 12 days which led to breakage of the fabrics to small fragments and even after six days, superficial cracks could be observed. For this reason, fibres are also treated to prevent biodegradation. This can be done in several ways like adding antimicrobial additives to the system which kill the bacteria or treating surfaces of the fibres to be water and oil repellent so that the micro-organisms can’t form biofilms on the surface of the material. (Simončič 2010, 2-6, 18) Due to the chemical composition of the natural fibres, the high water absorption causes swelling of the fibres which leads to higher absorption of water and oxidative agents to the fibres which then further on increases the swelling and disintegration of the cellulose and hemicellulose macromolecules. (Saha et al. 2015)

Although biodegradation rate of plain cellulosic fibres can be very high, in composite materials we have also the matrix covering the fibres which also affects to the biodegradability. As demonstrated by Chattopadhyay et al., natural fibre reinforced maleic anhydride grafted PP demonstrated only 5 to 15% fibre degradation during 90 days of soil incubation where the degradation of the reference samples from cellulose reached 85% degradation. This could be explained by the lack of free hydroxyl groups in the cellulosic structures due to the formation of the ester linkages with the maleic anhydride which then prevents attacks of different microbes. (Chattopadhyay et al. 2011) In another study, anaerobic degradation of flax fibre PE and flax fibre PLA composite was studied. Although the presence of the flax fibres should increase the degradation rate of the polymers through fragmentation because more material is exposed to the environment (Siakeng et al. 2019), such observations were not made during the study conducted by Iwańczuk et al. One reason could be that the polymer matrix creates covering layer on top of the fibres thus preventing the anaerobic digestion (Iwańczuk et al. 2015). Further on, biodegradation of flax fibre PLA composites can be enhanced with certain additives as demonstrated by Kumar et al., who added amphiphilic compounds to the composite. These hydrophilic compounds were observed to have higher biodegradation rates compared to neat flax fibre PLA samples as the other additive, dicumyl peroxide, delayed the biodegradation. (Kumar et al. 2010) This study, for example, demonstrates how the biodegradation of the composites can be adjusted to meet the lifetime of certain products while keeping the end-of-life disposal method the same.

Compared to neat polymers, like PLA, natural fibres have been observed to increase the biodegradation rate. The studies include soil burial testing and different kind of accelerated weathering tests. (Siakeng et al. 2019) Also, the architecture of the fibres has influence on the biodegradation as indicated by study conducted by Bayerl et al. In this study, pure PLAs biodegradation under normal composting conditions was compared to flax-PLA composites with different kind of flax architecture and weight content. In normal composting conditions, pure PLA does not degrade very quickly so there was no remarkable mass change during the eight-week testing period. Instead by increasing fibre weight content, the relative mass loss increased. Also, the architecture of the fibres seems to have influence on the degradation. After the eight weeks, UD samples had the highest mass loss compared to short fibre and long fibre samples. This indicates, that micro-organisms and water can use the fibres as channels throughout the samples which then accelerates degradation of the fibres and potentially the degradation of the PLA too. (Bayerl 2014)

Biodegradation of natural fibre composites is major concern when designing NFRPs for many industries. Effect of micro-organisms has been studied for different bast fibres used with different kind of matrices. In the study conducted by Crafword et al., nonwoven flax and hemp mats were used as reinforcements for PP matrices. Only mats and both types of composites were exposed to three different kind of environments: no-water and no-fungi, only water, and water and fungi for 7 and 28 days. After 28 days, maximum mass reduction observed for the pure flax mats mass was 28,58% in only-water conditions where the maximum mass loss for hemp mat was 15,4% in water and fungi -conditions. These tests indicate that there are indigenous fungal species inside the fibres which have been deposited there during stages of processing, for example dew-retting and the different chemical composition between natural fibres may affect to the biodegradation rate. (Crawford et al. 2017)

2.3.3 Prevention of biodeterioration

Biodegradation can be utilized as sustainable method to dispose certain materials, but it can also be a major problem in some applications. Unwanted biodegradation, biodeterioration, leads to reduction in materials mechanical properties, which may lead to premature failure of that certain part. This causes additional designing for biodegradable materials to be used in constructions or in civil engineering. It is often necessary to define materials biostability which describes materials resistance to biological factors, which may cause damage to the material. Biostability can be further divided to different subgroups like materials resistance to bacteria, fungi, termite, moth, and rodent damage. (Pekhtasheva et al. 2012, 93)

The biodeterioration of natural fibres can be a major problem especially in construction, paper, textile, and automotive industries due to the damage caused by the micro-organisms in certain conditions like high humidity and high temperature. For natural fibres, the biodeterioration is mostly caused by fungi, most commonly mildew, which then weakens the materials and causes certain unwanted odours to be released. Optimal conditions for the fungi are somewhere between 70-90% relative humidity, temperature of 24-30 °C, and about pH of 6. In some cases, micro-organisms can break fibres made from polypropylene, polyethylene and polyamide (Kozowski 2012, 532-534). In addition to fungi, some bacteria can damage the natural fibres, for example Clostridium and Cytophaga (Papaspyrides 2013, 823). In additional to the conditions that material is used in, the applied retting method during manufacturing and different treatments affect to the final biostability of the fibres (Pekhtasheva et al. 2012, 131).

Biostabilization of materials can be achieved through adding biocides to the material. Biocides are basically chemical compounds designed to prevent the biodegradation from specific source (Falkiewicz-Dulik et al. 2015, 59-60). Biocides can be further divided to four different main groups depending on application: disinfectants and general biocidal products, preservatives, pest control, and other biocidal products which then can be divided further to different subgroups depending on the target of the biocide (Directive 98/8/EC 1998).

Ideal biocides should efficiently prevent the activity of most of the micro-organisms present in the environment, have physical stability, won’t cause health issues, have low price, and be compatible with the fibres. Biocides can be applied during many phases of the fabrication process. It is possible to treat the fibres with the biocides or solution containing biocides, or to add biocides to the materials during manufacturing (Pekhtasheva et al. 2012, 148-149). Also, it has been shown that the matrix and the interface between the fibres and matrix affect the biodeterioration. With treating the matrix polymer so that it forms chemical links between polymer and the fibres, these chemical links can prevent some of the microbial attacks by reducing the chemical compounds susceptible to the microbial attacks. Also, the architecture of fibre structure has major effect on the degradability as the matrix polymer may prevent the micro-organisms from accessing the fibres. (Bayerl et al. 2014) (Chattopadhyay et al. 2011)

Different kinds of chemical and physical treatments have been made to natural fibre composites to study their effects on the biodegradability. For many of the studies, main goal has been improving the adhesion between the fibres and the matrix which plays important role in the biodegradation and has major impact on the mechanical properties of the composite. The aim of the treatments has been to decrease the hydrophilic nature of the fibres thus increasing the adhesion between the polymer matrix and the reinforcing fibres. Many of the chemical treatments done to the fibres aim to remove or moderate the hydroxyl groups in the natural fibres through esterification, which then increases moisture resistance of the fibres and increases the adhesion. From the physical treatments, plasma and corona have been used successfully to increase the adhesion between the fibres and the matrix. (Siakeng et al. 2019) (Saha et al. 2016) Esterification using acetic anhydride has been observed to improve the water absorption properties and making the natural fibres very resilient to biodegradation. Esterification treatments have also been done using benzoyl chloride and fatty acids and the results have been practically the same. However, these different methods use hazardous chemicals and are expensive. Another method is to use silane treatment, where the silane is hydrolysed and bonded to hydroxyl groups of cellulose molecules to increase the hydrophobicity. Silane treatments have been observed to increase the tensile strength of the composite and increase hydrophobicity of the fibres, but the treatment method is expensive and reduces the flexibility and Young's modulus of the fibres. Treatments can be done by applying certain coating on top of the fibres which would improve the mechanical properties and degradation resistance of material. However, these methods can use hazardous chemicals, be expensive and may require long reaction times. (Saha et al. 2016) Chemical modification of natural fibres can be seen as more favourable treatment method as the coatings may leach out to the nature and harm the environment.

Nowadays, several treatments methods have been studied which are based on non-hazardous chemicals and bio-based ingredients. Saha et al. studied transesterification-based treatment method using plant-based vegetable-oil and phenolic resin. The treatment decreased water absorption, increased tensile strength, and the treated fibred retain their tensile properties much more efficiently compared to untreated fibres. (Saha et al. 2012) Fiore et al. used sodium bicarbonate, commonly known as baking soda, solution to threat sisal fibres and studied its effect on single fibres and sisal – epoxy composite. Sodium bicarbonate with a long treatment time increased the adhesion between fibres and matrix and decreased water absorption of the fibres. (Fiore et al. 2016) Santos et al. found sodium bicarbonate treatment to decrease tensile strength of coir fibres but increase elastic modulus. For the coir epoxy and coir polyester composites, treatments did not increase the materials mechanical strength but increased the tensile and flexural modules. (Santos et al. 2019)

2.4 Microwave-based communication technology

In modern society, the transferring of data has a huge impact on our everyday life. Basically, all our communications nowadays happen through the transmission of data through telecommunications network which utilize, for example, electrical cables or electromagnetic radiation. In the past 20 years, especially the wireless technologies have had huge growth in their usage. Wireless telecommunication technologies use electromagnetic radiation as data transferring element instead of copper cabling or optical fibre cabling. These wireless data transmission technologies offer versatility compared to cabling solutions because electromagnetic radiation as transferring element doesn't need special medium, like electrically conductive or optically transparent, between the starting point and the end. For these reasons, wireless technologies are used to transfer data to moving objects whereas cabling can be used to provide faster data transfer speeds to stationary objects.

Mobile networks, or so-called cellular networks, use both wired and wireless communication technologies. A network consists of cells that are connected to the stationary backbone network. These cells then contain a transceiver which is a component consisting of a radio transmitter and a receiver. These cells then use electromagnetic radiation to communicate with mobile systems by transferring and receiving data. When the first cellular network technology was commercially launched in 1991, so called 2G or Global System Mobile, the data transferring speed was up to 64 kbit/s. However, due to advancements in signal processing and data transferring technologies, the speed of the networks has gone up from maximum of 64 kbit/s to up to 1 Gbit/s. (Gawas 2015) In addition to technological advancements in signal processing and data transferring technologies, higher electromagnetic frequencies are used to transfer the data and in upcoming 5th generation cellular network, so called New Radio, the maximum used frequencies can go up from the maximum of 2 GHz used in 4th generation networks to up to frequencies above 50 GHz in a 5G network. (Vora 2015)

Electromagnetic radiation can propagate with different frequencies, which then can have different properties. Radiation with the lowest frequencies are called radio waves and their frequencies go up to 300GHz. These radio waves can be further down divided into different categories depending on the range on the frequency, like very high frequency (VHF), ultra-high frequency (UHF) etc. Basic applications of radio wave frequencies (RF) are different kinds of telecommunication technologies and data transferring, like mobile networks or TV and radio broadcasting. The frequencies used above radio waves are called infrared radiation which covers frequencies between 300Ghz and 430 THz. Above this is the visible light between 430 and 790 Thz. Above visible light is the ultraviolet (UV) radiation covering frequencies from 790 THz to 30 PHz. The last two types of radiation are X-ray and gamma radiation. The frequency of the X-rays is commonly between 30 PHz and 30 EHz and gamma radiation above this, although the main difference between these two radiations is the source. X-rays are formed by actions of electrons and gamma radiation originates close to the nucleus of an atom. Compared to other types of electromagnetic radiation, higher energy levels of X-rays and gamma radiation can ionize atoms which makes them very dangerous especially for living organisms. (Electromagnetic radiation 2017)

As electromagnetic waves progress between different points, the power density is reduced through different factors. The reduction of power density is called attenuation and the reduction happening between the starting point of electromagnetic radiation and the end point is called path loss or path attenuation. Some of the basic mechanisms that attenuate the signal are reflection, diffraction, multiple diffraction, scattering, and absorption. For this work, the most important factors are the mechanisms are based on the properties of materials themselves and not so much on signal propagation routes between different points, for example between structures in a city. When electromagnetic waves propagate through materials, the loss of energy when waves propagate through the medium is called dielectric loss which describes materials capability to attenuate the signal. The electrical loss is often reported using two factors: dielectric constant and loss tangent.

Dielectric constant, or relative permittivity, describes permittivity of certain material to the permittivity of vacuum. Permittivity is describing the ratio of the electrical displacement in a medium to the electric field producing it and the unit of permittivity is Faradays per meter. Dielectric constant, or relatively permittivity, is dimensionless number describing the relation between vacuum and certain material. (Dielectric constant 2020) In dielectric material, external electric field causes polarization of atoms and depending on the electronegativity and chemical bonds between atoms in the material, dielectric constant varies between materials and basically, the lower the number is, the better the insulation capabilities are against electrical current. When materials properties to attenuate RF signals are evaluated, another value commonly used with dielectric constant is loss tangent. Loss tangent is a dimensionless number that describes dissipation of the electrical energy due to different physical processes like electrical conduction and dielectric relaxation. Loss factor is the tangent of a vector between lossless part of the material compared to the lossy part, so when electrical field passes through a dielectric material, loss tangent can be used to calculate the dielectric loss factor caused in the material based on the dielectric constant. (Sebastian 2008, 12-15)

In materials, the dielectric properties are based on the chemical structure and materials response on the varying electrical and magnetic field. When electromagnetic waves propagate through material, varying electrical field polarizes and moves charges within the atoms and molecules which then causes the permittivity of the material. Some basic mechanisms that are present in the material are dipolar relaxation, ionic relaxation, atomic polarization, electronic polarization, and ionic conduction. Dielectric relaxation describes the lag of the moving of the charges in the atoms or molecules as electromagnetic radiation passes through the material. As electromagnetic radiation can be described as varying electrical field, it moves the dipoles and charges inside atoms depending on the phase of the radiation on that certain moment. When the frequency is low enough compared to the lag of the dipoles and atoms, they can relax before the changing of the electrical phase which leads to higher dielectric constant. The relaxation phenomena then affect the attenuation so that the moving of the dipoles can attenuate the signal. (Blythe 1979, 38-41)

Dielectric constants and loss tangents of some basic materials have been collected in Table 2.3. In the table, there is each material, dielectric constant, loss factor, and the frequency where the dielectric properties were measured. For some of the materials, like HDPE, there is not that much variation between different studies. However, dielectric properties of epoxies seem to vary a lot depending on the type of epoxy that has been used and whether it is cured or not. The studies chosen to represent the values for dielectric properties have been chosen based on the frequency used in the measurements, so that it would be close to the frequencies that could be used in the 5G network. The dielectric constant is dependent also on the frequency of the radiation. The dielectric constant is also affected by the temperature of the material as it affects to the dielectric relaxation properties especially for the polymers, which go through change in the dielectric properties at the glass transition temperature.

Table 2.3: *Dielectric properties of some common materials*

Material	Dielectric constant	Loss tangent	Frequency for the measured values
High-density Polyethylene	2.37	0.0001	11.3 GHz
Polypropylene	2.26	0.0001	9.4 GHz
Polystyrene	2.55	0.0003	9 GHz
Polytetrafluoroethylene	2.04-2.08	0.0008	11.5 GHz
Poly(lactic acid)	2.57	0.0095	30 GHz
Epoxy	3.6	0.04	8-12 GHz
Unsaturated Polyester resin	2.95	0.007	8-12 GHz
E-Glass	6.06	0.004	8-12 GHz
E-Glass reinforced polyester	4.15	0.015	8-12 GHz
E-Glass reinforced epoxy	4.4	0.016	8-12 GHz

One application, that is especially relevant to the topic of this thesis, is the materials used in radomes. A radome, shortened from radar dome, is a structure used to cover and protect antennas from the environment like wind, water, etc. They are used in many kinds of applications from boats to missile guiding systems, so basically in applications where usage of antennas is necessary. To increase the performance of the radar, the materials must have a low attenuation in general that is basically based on the dielectric constant and loss tangent in the operational range of the antenna. The requirements for certain application are good enough mechanical properties, low water absorption, stable mechanical and electrical properties in the environment – i.e. independent of the temperature, and also the environmental resistance. The walls of the radomes can be monolithic, so that there is only one solid layer, or layered structures, like sandwich panels. The basic materials, used for example aircraft, are glass reinforced polyesters and different honeycomb structures. In many applications, the radome material is also chosen to be functional over certain bandwidth so that it could prevent coupling from other sources of electromagnetic radiation and so improve the performance of the radar. (Kozakoff 2010, 1-5, 55-62)

3. EXPERIMENTAL SECTION

The experimental work of the thesis is divided into two major tasks: Camouflage Radome Unit (CRU), and the testing and the analysing of the composites fabricated during the work. In Figure 3.1, the division of the work into two topics is roughly presented, and also what the content of both topics is. The first topic, Camouflage Radome Unit (CRU), focuses on the CRU as a product, creating the re-design for it, and studying it as an industrial product. The second topic, Novel composite material, focuses on the development, processing and testing of sustainable novel composite materials that could function as candidate materials for the CRU re-design.

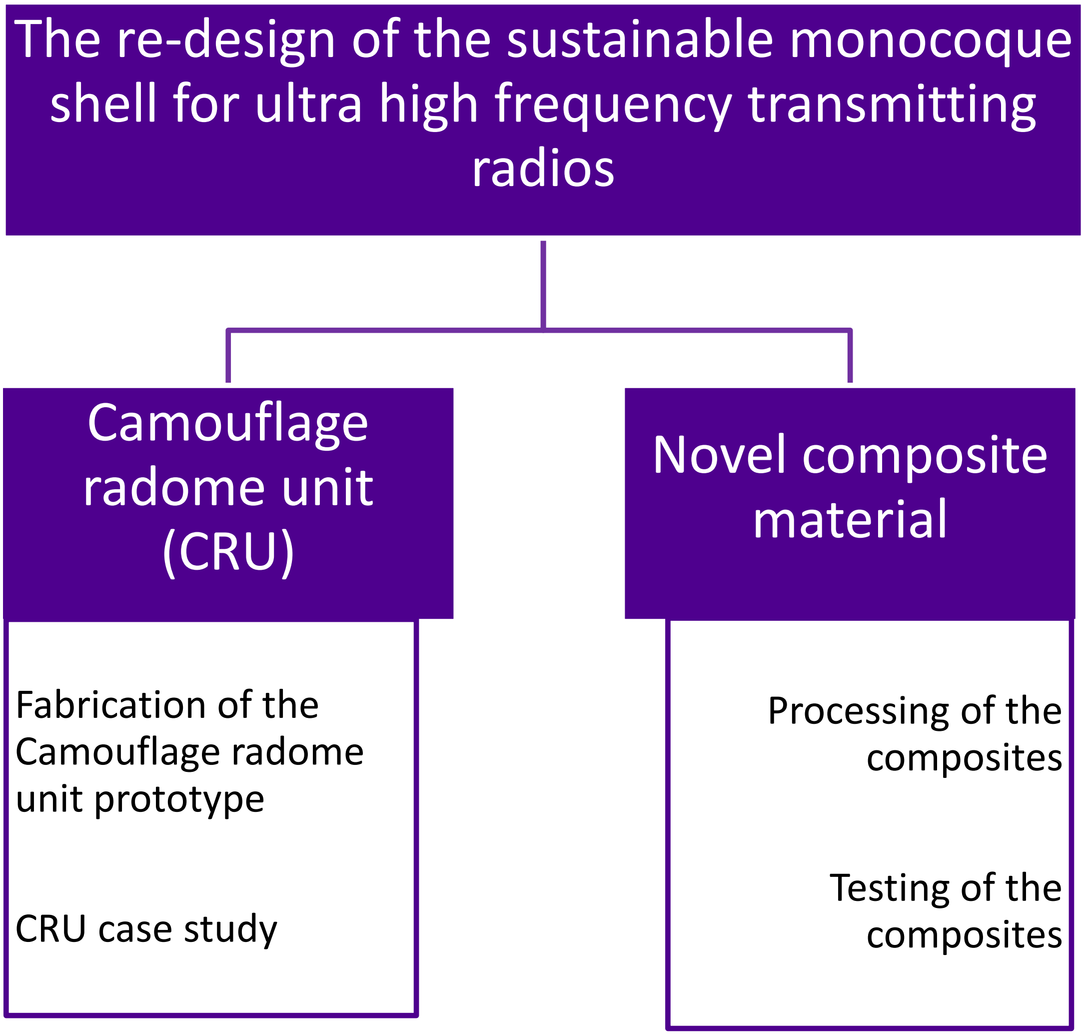

Figure 3.1: *The division of the experimental work of the thesis in different topics*

In this chapter, I go first through the materials applied during the work and, after that, the processing and testing conditions used for the composite materials. After going through the different processing methods and the parameters, I focus on the Camouflage Radome Unit (CRU) which acts as a benchmark case for a composite. The topics for the CRU are re-design, material selection and fabrication of the prototype of the composite Camouflage Radome Unit.

3.1 Materials

In this thesis, the aim of the work is to analyse, manufacture and study sustainable composites. For this reason, flax fibres were originally chosen to be used as the reinforcing element. This material was chosen due to the environmentally friendly nature, good mechanical properties compared to other natural fibers, and the good availability in Finland. The chosen type of reinforcement was a 200 g/m^2 2x2 twill fabric of flax, a woven fabric, produced by Biotex. In additional to this, powerRibs® supporting flax fiber element produced by Bcomp was used to fabricate laminates that were showcased in the Luxturrim5G project's finale. Figure 3.2 represents the flax fibre products' visual appearance. The woven flax fabrics were used as reinforcing element in the composites whereas powerRibs® was used only as demonstrative material for the fabrication tasks.

Figure 3.2: *Woven flax fibre fabric and a piece of Power Ribs® -fabric used in the thesis*

In the fibrous composites, two types of plastics were used as matrix materials: a thermoset epoxy and polylactid acid. The epoxy resin was called SuperSap® produced by Entropy Resins. SuperSap® is a partly bio-based epoxy resin that comes in different forms depending on the usage. For this work, two types of SuperSap® epoxy resins were used. The first one was Super Sap® CLR Epoxy resin with Super Sap® CLS Hardener and Super Sap® INH, which were used to produce flax epoxy laminates. Super Sap® INH was used to decrease the viscosity of the resin so that it could be used in vacuum infusion processing. Another type of SuperSap® epoxy resin that was used during the work was SuperSap® ONE epoxy resin combined with SuperSap® ONS hardener, which were used in the hand lay-up process. SuperSap® was chosen due to the biobased content of up to 40%, and the good availability for different purposes, mainly vacuum infusion and hand lay-up, which were necessary processing methods for this thesis. The applied polylactic acid was Ingeo Biopolymer 2003D produced by NatureWorks. It is a grade of PLA designed to be used with conventional extrusion equipment. PLA was chosen to be used as another matrix material due to the biodegradability of the material, good mechanical properties, thermoplastic nature, and availability of PLA.

Figure 3.3: *Pine gum rosin produced by Forchem*

Another thermoplastic polymer that was chosen to be studied during the work was a commercial blend Mater-Bi®, a thermoplastic starch-based system, manufactured by Novamont. Its function for the thesis was the different effect on the antibacterial rosin-flax composites and the biodegradability in the soil environment.

The last material component used during the work was pine gum rosin produced by Forchem. The rosin was used as a natural additive to study its effects on the antibacterial properties and effects on the interfacial properties of the fibre reinforced composites that use thermoplastic polymer as a matrix material. Rosin was chosen to be studied as an additive due to the promising antibacterial and antifungal results in the current literature and as found in the previous studies by Tampere University. Rosin, being natural material, and having excellent antibacterial properties, affects decomposition of pine heartwood, for example. The rosin particles were compounded with PLA and also rosin-based treatments were done to the flax fibre fabrics by immersing them in a 10 m-% rosin (and alcohol) solution for 24h and after that drying the fabrics in room temperature.

3.2 Thermoplastic specimen preparation methods

In this chapter, I go through the different manufacturing methods used during the work. Due to the nature of the thermoset and thermoplastic materials, the fabrication methods, depending on the sample, were several different. For the epoxy resin, vacuum assisted resin transfer moulding and hand lay-up combined with vacuum bagging were used. For the thermoplastics, compounding, injection moulding, and hot pressing were used in material and specimen preparation.

3.2.1 Compounding extrusion

Compounding extrusion was used to compound pine gum rosin with the PLA and Mater-Bi® so that the compound would contain 10 m-% of the rosin. For PLA, 200 °C was used as the compounding extrusion temperature as PLA had melting range between 150 and 180 °C. For Mater-Bi®, the temperature used during extrusion was 160 °C. Also, pure materials (no rosin) were extruded once to compensate the mechanical losses caused by the extrusion cycle. The extruder was Brabender 25E (twin-screw extruder). After the extrusion, the materials were chopped to granulates in a hacker that they could be used in the injection moulding and hot pressing stages of preparation.

During the compounding extrusion, there may have been possible degradation of the rosin as the degradation (in terms of mass loss) happens between 220 and 250 °C (Kanerva et al. 2019). Also, there has possibly occurred some degradation of PLA, but it was compensated from the results point of view by exposing pure PLA to one extrusion cycle. The rosin addition caused some problems during the extrusion due to the melting and gas formation range that is below the mass loss initiation temperature. This caused some feeding problems in the hopper so that there were extra procedures to get even and well dissolved compound mass. During the compounding, the hopper was practically empty throughout the extrusion so that the material was fed to the extruder only to fill the extrusion screw below hopper and not more than that. The hacking of Mater-Bi® to get a granulate form caused some problems during the hacking due to the rubber-like behaviour of Mater-Bi® and Mater-Bi® compounded with rosin as they easily fouled to the hacking blades.

3.2.2 Injection moulding

Injection moulding was used to fabricate tensile test specimens for PLA and Mater-Bi® test series, and their counterparts containing 10 m-% of rosin. Table 3.1 presents the most critical processing parameters used during the fabrication. The parameters were mostly defined experimentally, by a trial and error manner with several initial processing experiments. The injection moulding machine used during the processing was Roboshot α-C30 (single-screw).

Table 3.1: *The processing parameters used to fabricate the tensile specimen*

Material system	Extrusion temperature (°C)	Holding pressure (bar)	Holding time (s)	Cooling time (s)
PLA	220	700	10	30
PLA + Rosin	200	400	30	30
Mater-Bi®	160	250	10	25
Mater-Bi® + Rosin	160	190	15	30

3.3 Fabrication of composite laminates

The lay-up for test laminates was selected to be quasi-isotropic and total layers used in the laminates consisted of four fabrics so that the final thickness was close to 1,7 mm. Figure 3.4 illustrates the lay-up used with both Super Sap® and PLA matrix. The 45/-45 fabrics were placed on the top and bottom of the fabric stack because it is desired to have the same orientation for both top and bottom fabrics in laminates to minimize warpage due coupling. Also, having the 45/-45 fabrics on the top and bottom of the laminate gives more accurate results during flexural testing because midplane shear can significantly delaminate the structure.

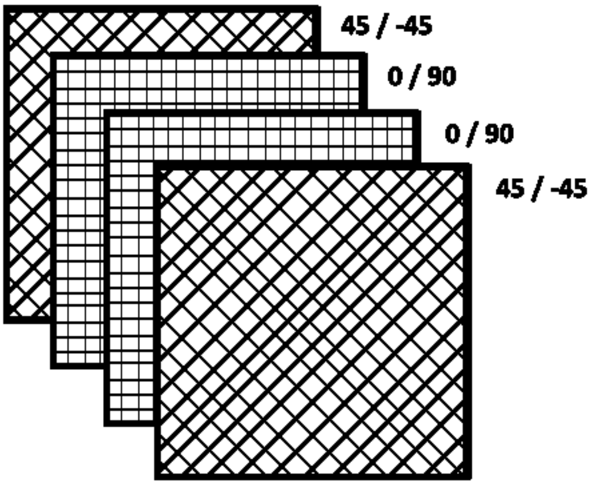

Figure 3.4: *Quasi-isotropic lay-up for composite laminates of this thesis. Each layer is 2x2 twill fabric piece.*

The coupons used for the mechanical testing were cut off laminates fabricated either with a vacuum assisted transfer moulding method or a hot pressing method depending on the matrix material. In this chapter, I first go through the practice of the VARTM-process, and the equipment used in the VARTM process. After this, I focus on the fabrication of the flax epoxy laminates using VARTM and, finally, the hot pressing of the flax PLA laminates. For the work, total of two laminates of each type of composite material were manufactured.

3.3.1 Fabrication of the trial laminates

Before fabrication of CRU or quasi-isotropic laminates used for actual material testing, a couple of trial laminates were fabricated using the vacuum assisted resin transfer moulding to learn the fabrication process and examine used materials behaviour and optimal fabrication setup. Dimensions of the first laminate were 30x30 centimetres and used materials were Biotex flax 200g/m^2 2x2 twill mat and Super Sap® Clear Laminating epoxy. Other components used in the mould were two pieces of peel ply for top and bottom sides, two pieces of net bleeder, releasing agent, sealing tape and vacuum bagging plastic.

For the first test laminate, a stack of ten 30x30 cm plies were cut off the woven flax fibre fabric using sharp scissors. Flax fibre plies and the scissors are presented in Figure 3.5. According to the manufacturer, the thickness of one ply should be between 0,25 and 0,4 mm so in average the total thickness of the laminate should be somewhere around 3,0 to 3,5 millimetres. Flax fibre plies were dried in an air-circulating oven before placing them on the mould to remove excess humidity. The dehydration temperature was 100 °C and the dwell time was about one hour and fifteen minutes.

Figure 3.5: *A stack of 30x30cm plies of the flax fibre fabric.*

In Figure 3.6, the mould before placing of the different plies is presented. First, sealant tape was placed on the glass base to form the frames for the mould. The sealant tape was added to the system before applying releasing agents because the releasing agent may worsen the sticking of the tape and that might lead to unwanted leaking (air) after the vacuum has been applied. After the mould's frames were build using sealant tape, the mould was cleaned using acetone and cloth. After this, the base was wiped with a clean cloth and a layer of releasing agent was applied on top of the glass. After this, excess releasing agent was removed with dry cloth, base cleaned with acetone and new layer of release agent was applied.

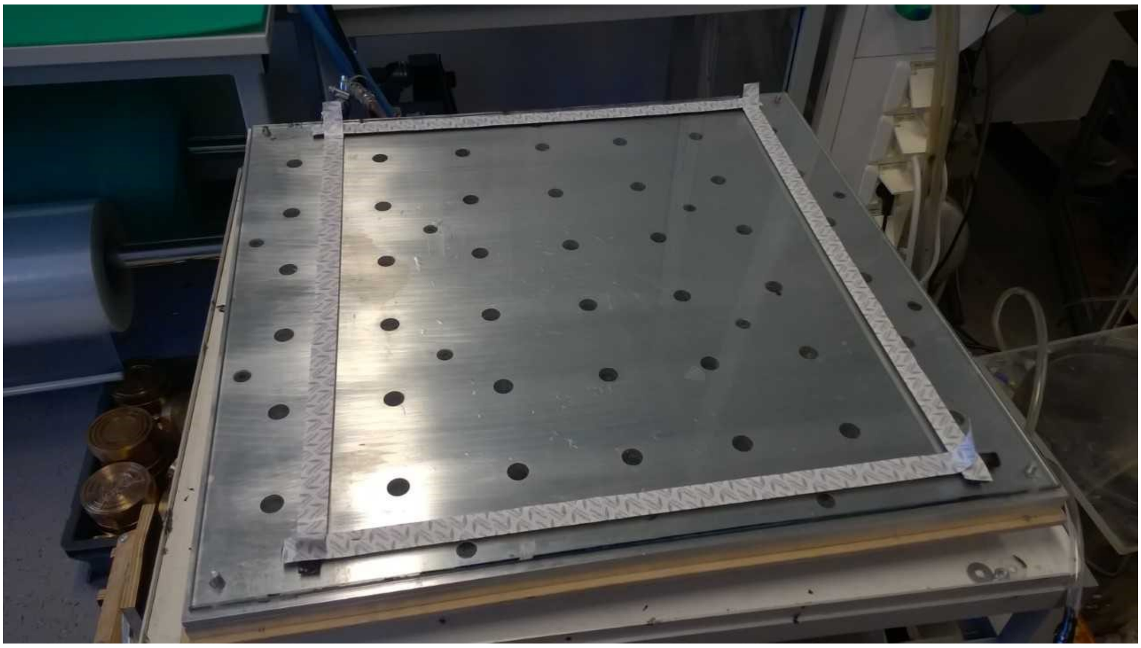

Figure 3.6: *The stage of the mould fabrication process before the placing of the different plies.*

After the frames were build using sealant tape and release agent layers were applied on top of the base, different plies were stacked on the mould. In Figure 4.3, the mould is presented, where different plies have been applied. For the trial laminate, one layer of bleeder, about the size of the fabric pieces, was placed on top of the glass. On top of the bleeder, one layer of peel ply, bigger than the fabrics, was placed. After this, flax fibre pieces were taken out of the oven, weighted and placed on top of the peel ply layer so that the orientation throughout the laminate was [0° / 90^0]. On top of the flax fibres one layer of peel ply was placed and on top of that one layer of bleeder. Additional to these, to the sucking end of the mould, a piece of peel ply and a layer of cotton was placed to ease the flow.

After placing all the different layers, housings were placed in the mould, sealed up with a sealant tape and a vacuum bag was put on the sealant tape frames. After this, a vacuum pump was used to pump air out of the system and to see any leakage in the system. The next step was to mix the resin using Super Sap® epoxy with two kind of hardeners: slow and another one hardener system to lower the viscosity for the infusion process. After mixing the resin, it was infused to mould. In Figure 3.7, the mould after impregnation and cured for 20 hours at the room temperature is presented. For the curing in the mould at room temperature, a steel plate and a total weight of 12 kg were placed on the laminate (glass surface).

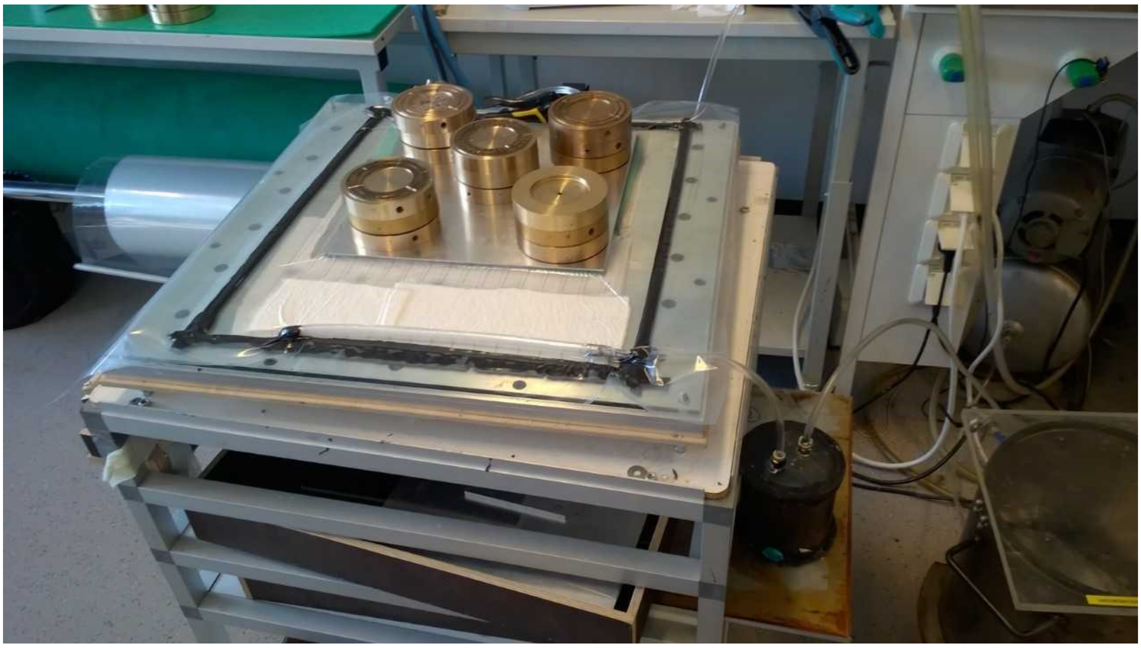

Figure 3.7: *Mould during curing of epoxy-flax trial laminates and additional weights on the mould setup.*

After 20 hours since the initial starting point of impregnation of fibres, the mould was disassembled and the laminate was taken out. After curing at room temperature for 20 hours, the laminate was post-cured in 80 °C for three hours. The trial laminate number 1 had many problems induced yet these could be utilized to enhance the fabrication process in the future. The main flaw was the amount of infused resin. The trial lamination was not impregnated throughout and one corner of the laminate did not have epoxy in it. The second one, the thickness of the laminate was not correct. The aim was to make it about 3,0 mm thick but, in the end, the thickness of the laminate was in average 4,75 mm. In addition to these, the laminate had quite much voids and air bubbles at the surface and the laminate had some warpage. The trial laminate number 1 is presented in Figure 3.8. By using the information got during the fabrication of the trial laminate 1, another better trial laminate was fabricated.

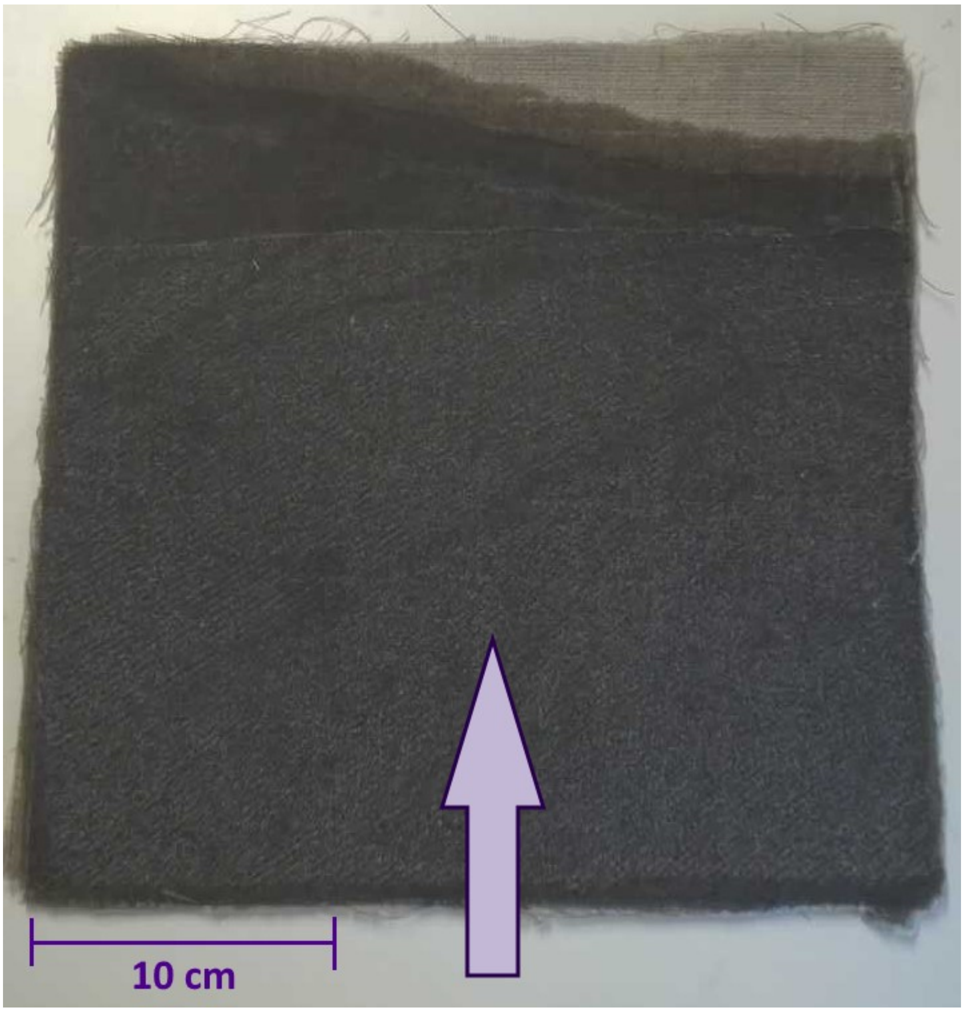

Figure 3.8: *The trial laminate number 1 with a dry corner (left upper corner) due to non-optimal infusion parameters. The arrow in the figure presents the direction of the flow.*

The trial laminate number 2 was fabricated based on the observations made during fabrication of the first trial laminate. For the next laminate, the amount of flax fibre layers was decreased from 10 to 6 to achieve a close to optimal thickness. Another change was to use more resin to achieve full impregnation. Also, few of smaller adjustments were made to the setting up of the mould. In Figure 3.9, the trial laminate number 2 is presented. In this laminate, the average thickness was calculated to be around 2.75 mm, all the reinforcement is impregnated, the laminate was not curved and there were less voids in the surface. The laminate was cured at room temperature for about 20 hours and then it was post-cured at 80 °C for 2 hours. For the last trial laminate, number 3, seven plies were used, and the size of the fabric pieces was decreased to 25x25 cm and small adjustments were made to the setting up of the mould.

Figure 3.9: *The trial laminate number 2 with fully impregnated layers of flax and close to optimum thickness for testing purposes.*

In Table 3.2, processing parameters and measured values from the trial laminates are marked. The first three rows are the amount of Super Sap® components used in the both trial laminates. The first one, CLR Epoxy, is the epoxy component of the infused resin. The CLS hardener is slow hardener for the resin and INH is the modified hardener used to lower the viscosity of the resin system for infusion. The amount of flax fibres describes the number of plies used to make the laminate and weight is the total weight of all the flax plies (i.e. the stack). All the laminates were cured at equal temperatures, but for the trial laminates number 2 and 3, the post-curing time was set to be shorter due to the thickness compared to the trial laminate number 1.

Table 3.2: *Processing parameters of the trial laminates*

Trial laminate	Number 1	Number 2	Number 3
Super Sap® CLR Epoxy	309 g	350 g	350 g
Super Sap® CLS Hardener	59 g	66,5 g	66,5 g
Super Sap® INH	59 g	66,5 g	66,5 g
Amount of flax fibre plies	10	6	7
Size of the plies	30x30 cm	30x30 cm	25x25 cm
Weight of flax fibre plies	159,6g	95,5 g	78,3 g
Curing	20 h, room temperature	20 h, room temperature	20 h, room temperature
Post-curing	3 h, 80 °C	2 h, 80 °C	3h, 80 °C

The fabrication of the trial laminates gave me important information about the process itself and about what kind of factors must be taken into account for the fabrication of composite samples. These factors include sizes of different plies, amount of flax fibres and epoxy resin, and the optimal settings for the mould. Also, the final thickness (post-cure) for each laminate was used to determinate the final number of plies used to fabricate successful test laminates. According to the calculations and the observations obtained based on the trial laminates, the actual test laminates are to be made with four layers of flax fabrics where orientation of the fibre bundles, from top to bottom, resembles the sequence [45/-45/0/90,0/90,45/-45]. This system of preparation should give laminates that have the final thickness of approximately 1,7 mm.

3.3.2 VARTM of Flax-epoxy laminates

The observations based on the fabrication of the trial laminates were used as the basis of information to predict the final thickness of laminates and to study limitations for the fabrication process. The size of the test laminates made from Super Sap® epoxy resin and flax were 25x30 cm. This certain size was chosen due to planar size of the tensile test specimen, which was 1x25 cm, and to make it small enough so that vacuum infusion could be used more easily. The system of the VARTM was finally one layer of bleeder on the bottom, on top of that a layer of peel ply, then four layers of flax fabrics in a quasi-isotropic lay-up, then one layer of peel ply, and on top of the pile a layer of bleeder. In addition to these, one slice of cotton was placed in the other end of the mould to absorb external resin. Before stacking up the layers, the mould was cleaned, and a cleaning agent was applied. The flax fabrics were dried in an oven for 1h at 80 °C and, after that, for 1h at 100 °C to remove moisture. The curing time at an atmospheric temperature under vacuum was 20h and, after that, laminates were post-cured at 80 °C for 2h.

3.3.3 Hot pressing of flax PLA coupons

The flax and PLA laminates were in size 20x27 cm. In this case, the dimensions of the test laminates were smaller due to the equipment used in the hot pressing of the laminates. First, the flax fabrics were dried in an oven overnight at 80 °C and pure PLA in a special drier at 90 °C for 2h. The hot pressing was done by first pressing 28x23,5x0,1 cm PLA sheet at 180 °C, 100 bar, for 10 minutes, and between two steel plates and metallic frame between the plates. After pressing the PLA sheets, one flax fabric and a PLA sheet was placed between the steel plates and pressed at 180 °C for six minutes. When total of four flax PLA sheets were pressed, all the four layers were laid-up as illustrated in Figure 4.7 and pressed for eight minutes at 180 °C. Parameters chosen for the hot pressing were determined based on observations during training with the hot press and by a literature survey. The final dimensions for the first test laminate was 15x27 cm. A smaller size compared to the original size of fabrics was caused by slipping of the flax layers during pressing, so that part of the laminates had to be cut off. The second laminate was produced with the same parameters, but this time less slipping occurred, so the final test laminate was close to 20x27 cm.

For the hot pressing of flax and PLA compounded with rosin, some adjustments had to be made to the process. Due to the melting range of rosin being approximately between 50 and 70 °C, the temperature used in the hot pressing was lower. Also, PLA rosin compound was dried overnight at 45 °C due to the low softening point of rosin. During hot pressing, the procedure was the same as was used with flax PLA laminates, but temperature was set to 160 °C and less time was used during pressing. Also, the rosin treated flax and PLA and rosin laminates were processed with the same parameters.

Table 3.3 presents the processing parameters used to fabricate the composite laminates by the hot press. The difference in the chosen processing temperature was caused by the decreased melting temperature when rosin was compounded with PLA. The pressure during the hot pressing was selected to be only 10 bars as higher pressures were observed to break down the flax fibre fabrics. Lower pressing times were found recommended especially with pure PLA as flax fibres had been observed to start to degrade above 170 °C.

Table 3.3: *Processing parameters used during the hot pressing of composite laminates.*

Thermoplastic laminates	Flax PLA	Flax PLA+rosin	Rosin treated flax PLA+rosin
Processing temperature (°C)	180	160	160
Pressure (bar)	10	10	10
One-layer pressing time	6 minutes	5 minutes 30 seconds	5 minutes 30 seconds
Layer combination pressing time	8 minutes	8 minutes	8 minutes

Between the laminates, there was a difference in the final thickness and the overall quality of the coupons. Flax PLA laminates were observed to be about 2,7 mm thick whereas laminates containing rosin were observed to be much thinner, about 1,7 mm in average. Also, the laminates containing rosin were observed to have larger (higher level) porosity. The higher the rosin content, the lower the quality of the laminates was observed to be. The tensile and flexural testing specimens were cut from the laminates using a band saw and a circular saw. Finally, both types of specimens were grinded to the final dimensions using sandpaper and a grinding machine depending on the specimen type (to achieve best surface finish).

3.4 Test methods

In this chapter, I go through the test methods that were used during the experimental work of developing and analysing materials. In the thesis, especially the biodegradation-related properties were in the focus, so the soil burial testing played a major role. The mechanical testing activities consisted of tensile tests done for composites and pure polymeric materials, and short-beam three-point bending done only to the reinforced composites.

3.4.1 Soil burial testing

The soil burial testing of the selected materials was carried out in a compost bin, in detail by using a Pikakompostori 220, manufactured by Biolan. The specimens were buried in soil for 14 days (batch 1) and 55 days (batch 2) and the effect of soil burial was studied by visually inspecting the materials and performing the mechanical testing (Section 3.4.2). The compost was located in a room with essentially constant environment. The composting was started 4. October 2020 and the first batch was taken out 18. October, and the final batch was taken out on 29. November. In Table 3.4, all the different materials and types of specimens that were included in the compost are collected and described.

Table 3.4: *Specimens prepared and reserved for the soil burial test*

Material system	Types of the specimen per patch
Pure Mater-Bi®	10 tensile test specimens
Mater-Bi® with 10 m-% of rosin	10 tensile test specimens
Pure PLA	10 tensile test specimens
PLA with 10 m-% rosin	5 tensile test specimens
Flax epoxy composite	5 tensile test specimens 5 Interlaminar shear strength specimens
Flax PLA composite	5 tensile test specimens 5 Interlaminar shear strength specimens
Flax PLA + rosin composite	5 tensile test specimens 5 Interlaminar shear strength specimens
Rosin treated flax PLA + rosin composite	5 tensile test specimens 5 Interlaminar shear strength specimens

In Figures 3.10 and 3.11, the setup for the soil burial testing is presented. Due to the high number of specimens and two batches from which the first batch was removed from the compost after two weeks, the filling of the compost bin had to be planned so that the specimens wouldn’t get lost inside but would be evenly treated. For this purpose, tubes and wrappers were made of stainless steel wire mesh to help with the removal of the specimens out of the compost bin. Inside the compost bin, the specimens were also divided with a fence made of the stainless steel wire mesh so that, on the other side of the compost, were the specimen containing rosin and, on the other side, the specimens without any rosin. The division ensured that any water-dissolved part from rosin (specimens) would not affect the specimens without rosin (possibly in the direction of drainage inside the bin). The mesh anyway allowed for even moisture distribution and growth of microbe colonies.

Figure 3.10: *The compost bin used for the soil burial testing of polymer and composite specimens*

Figure 3.11: *The compost bin from the inside showing the stainless steel wire mesh separator tubes and the wall*

For the soil burial testing, the ends of the composite tensile testing specimens were covered with electrical tape to protect them from degradation in the tab zone This way, they would be expected to break from the middle (gauge section) during the tensile testing. For the ILSS specimen, wrappers, for each set of five specimens, were made from the stainless steel wire mesh and closed with stainless steel, acid resistant screws. A wrapper containing a set of specimens is represented in Figure 3.12. Due to the possible degradation of the specimens to the point where they couldn't be identified and distinguished from each other, certain operations were done to the specimens and wrappers. In the case of the ILSS specimens, the wrappers were marked by the number of screws. Figure 3.13 presents blueprints of the differences between the wrappers. Although the shapes of the wrappers were same, the positioning and amount of screws were varying between the wrappers containing certain types of specimens. In the case of the tensile specimens, cut-marks were done to the specimen ends (gripping area) to make sure that they could be identified and distinguished from each other.

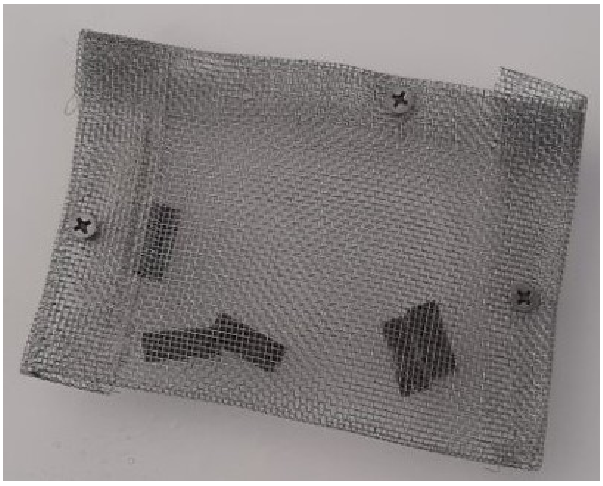

Figure 3.12: *A wrapper for the ILSS specimens during soil burial testing*

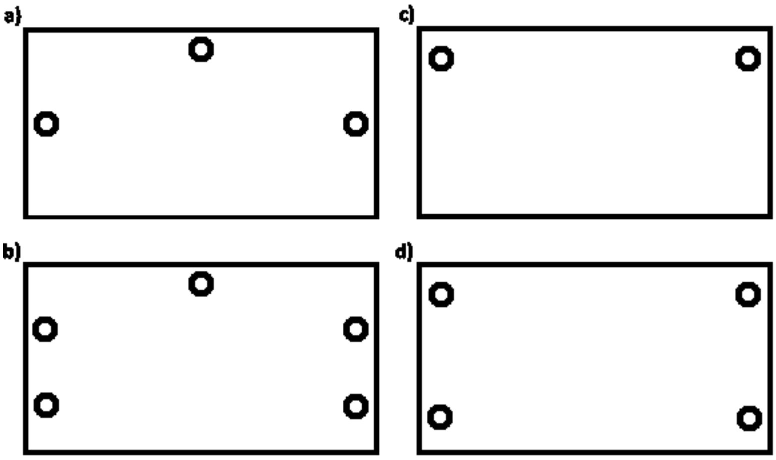

Figure 3.13: *The positioning of the screws in the ILSS wrappers. a) flax epoxy, b) flax PLA, c) flax PLA+rosin, and d) rosin treated flax PLA+rosin series.*

The soil inside the compost bin consisted of partly garden peat, manufactured by Biolan, hummus and leaves collected from the nearby forest, and biological activator. In table 3.5, the components of the medium, that was used to fill the compost, is collected and shown what was the composition of each of the ingredients. Material collected from the nature, mainly hummus, leaves, and branches, were used as source for different types of micro-organisms present in this region of the world (Scandinavian mixed forest). Garden peat functioned as basic living environment for the present bacteria and fungi. The soil from the nature containing branches and leaves acted as food for the bacteria, and also provided local micro-organism strain to be acting in the compost bin. Throughout the composting, activator solution (activator and lukewarm water) was added to the compost to sustain and accelerate the bacterial activity, keep the moisture content at appropriate level, and to add extra heat to the compost. In the beginning, a bag of another solid activator (0.6kg) was mixed with the peat and the soil.

Table 3.5: *Composition of the medium in the compost bin*

Material	Composition
Garden peat, Biolan	Compost mixture (chicken manure, bark chips and peat). Nutrients: Nitrogen (15 mg/l), phosphorus (100mg/l), and potassium (500mg/l)
Compost activator (solution), Neko	Bacteria: Bacillius strains and Pseudomans strains. Fungus: Aspergillus and Trichoderma. Nutrients, salts, and emulgators
Compost activator (solid), Multicraft	Wheat bran, spelt husk, micro-organisms, water, sunflower oil, sugarcane molasses, and coralline algae calcium
Soil collected from the nature	Humus, branches, and leaves

In Figures 3.14 and 3.15, the mixture of garden peat, and the soil collected from the nature that was used to fill the compost is shown. Total of 200 litres of garden peat produced by Biolan and 50 litres of soil collected from the nature was used to fill the compost bin. After filling the compost, total of 0.75 dl of compost activator (Neko) mixed with warm water (≈37 °C) was added to the system. This solution was mixed (poured) to the compost frequently during the process. Figure 3.12 represents the total amount of medium put inside the compost bin and Figure 3.13 presents single spade full of the compost, which was then poured to the compost bin.

Figure 3.14: *Total amount of compost that was mixed well and used to fill the compost bin.*

Figure 3.15: *Close-up picture from the compost that was used to fill the compost bin*

The positioning of the specimen was done so that different specimens (from different series) were equally spread to the compost bin. This was done to compensate for the different conditions that may be present in the different parts of the compost, for example higher temperatures or moisture content in the bottom part. The filling of the compost was started by first filling about a 20 cm layer of compost on the bottom of the compost bin. After this, the first set of specimens were put in. The specimens that would be then extracted two weeks later were put into the steel mesh tubes. After filling about ¼ of specimens from each series, another layer of compost was poured in so that the specimens were totally covered, and after that, another set of specimens were placed inside. In Figure 3.16 the compost bin after it has been filled to the 2/3 level is presented. During the filling, specimens containing rosin had been put into the right side of the mesh wall in the middle. This was done due to possible leaching of rosin components to the system, which would then contaminate the compost bin (or specimens without rosin). By placing the mesh, pure polymer systems were be kept separated from the ones containing rosin. In Figure 3.17, one of the steel tubes is shown when compost bin has been partly filled and a bunch of specimens have been placed in.

Figure 3.16: *Compost bin being almost full during the loading process of medium and specimens.*

Figure 3.17: *One of the stainless steel mesh tubes after placing in a set of specimens.*

After the specimens had been put in, the compost bin was fully filled with compost and compost activator with warm water was poured in. During composting, the temperature of the system was monitored constantly, and now and then more compost activator with warm water was added to the system. After two weeks, the first set of specimens were extracted from the compost bin. After the extracting, the specimens were cleaned and put inside an oven to dehydrate the specimens. In Figure 3.18, one of the two steel tubes after extracting from the compost is shown. In Figure 3.19, the composite tensile specimens after composting are shown; from left to right flax epoxy, flax PLA, flax PLA+rosin, and rosin treated flax PLA+rosin. In Figure 3.20, the polymer tensile specimen after composting are shown. On the left side of the figure are the pure ones and on the right side the ones containing rosin. The pure Mater-Bi® specimens are clearly (visually observed) colonized by bacteria that makes them look stained. However, colonization was not observed in the Mater-Bi® specimens containing rosin. Only some black stains, probably some sort of fungi, was observed on the surface of both types of the specimens. In PLA specimens, there weren't any visual changes observed.

Figure 3.18: *One of the steel tubes, containing the compost medium and specimens at the time of extraction from compost.*

Figure 3.19: *The composite tensile specimens after the composting. From left to right: flax epoxy, flax PLA, flax PLA+rosin, and rosin treated flax PLA+rosin.*

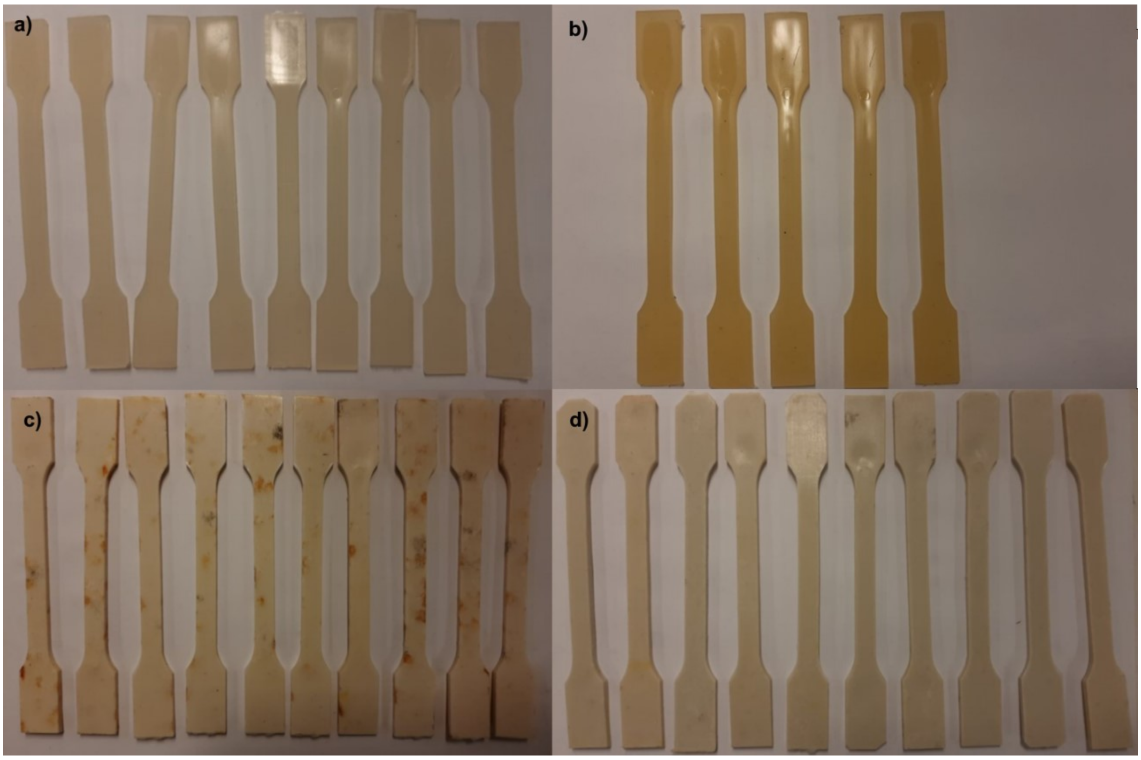

Figure 3.20: *The polymer tensile test specimen. a) PLA, b) PLA + rosin, c) Mater-Bi®, and d) Mater-Bi® + rosin*

After cleaning the specimens from medium, they were dried in an oven for 72 hours in 25 °C and the mass was measured every 24h to make sure that the composites would be completely dry. After the drying, the colour of the composite specimens was much lighter compared to the original colour. In Figure 3.21, the surfaces of the composite with flax and PLA+rosin before and after the soil burial testing are presented. The colour of the composite specimens was dark brownish when taken out from the compost. This would indicate, that the water absorbed by the natural fibres would damage the composite in some way, probably in the fibre-matric interface.

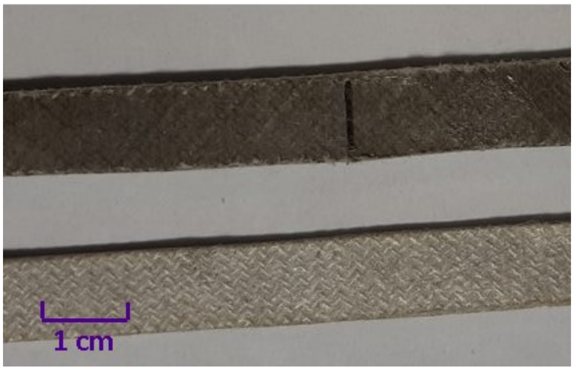

Figure 3.21: *Comparison of flax PLA+rosin specimens before composting (upper specimen) and after composting (lower specimen)*

During the composting, the temperature outside and inside the compost was monitored. For the monitoring, Testo 175H1 data logger was used to collect data from the outside of the compost and Testo 175T2 was used to monitor the temperature in the bottom part of the compost and near the floor outside the compost. In addition to the loggers, data from the compost bin's own thermometer was monitored. Figure 3.22 presents the monitored temperature throughout the test (Testo data loggers). The purple line is the data from the bottom (inside) part of the compost, the blue line is data collected from outside the compost bin near the floor, and the light purple line is the data collected from outside of the compost from top of the compost bin.

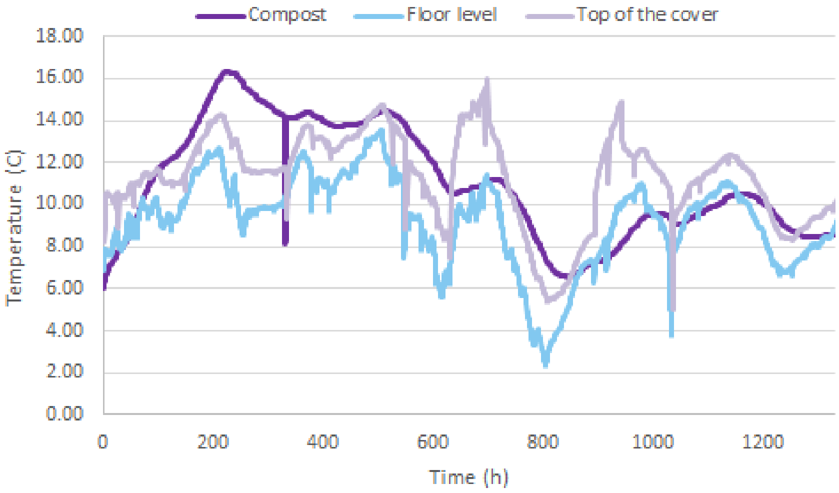

Figure 3.22: *The temperature data collected from the compost bin and its environment*

From the graph it can be seen that the compost was active for the first four weeks. After that, it more or less followed the temperature of the environment outside the compost. This could be seen as point, where the bacterial activity stopped due to a lack of food in the compost bin. The sharp point in the temperature of the compost is the point where the first batch of specimens was extracted from the compost bin.

3.4.2 Mechanical testing

The mechanical testing in this thesis consisted of three types of testing methods:

- tensile testing for the polymer and composite specimens,
- short-beam strength three-point bending for the composite specimens, and
- traditional three-point bending for the pure SuperSap® specimens.

To fabricate the specimens, the coupons were cut to down using band saw and/or circular saw. Band saw was used with thermoplastic composites, where circular saw with special blade was used for flax – epoxy coupons. To achieve the final dimensions for the specimens, grinders were used. For the small ILSS specimen, 8x20mm, grinding and polishing machine was used. For the tensile test specimen, 1x250mm, that were too long to be grinded with the machine, the grinding to the final dimensions was done manually. Before testing, specimens were kept in constant air humidity and temperature for a week before testing as the humidity in the natural fibres affects greatly on the mechanical properties of the material. The temperature was about 23°C and air humidity was about 50%.

Tensile testing was done with Instron 5967 tensile tester and according to the standard ISO 572 for composite specimens, except that exceptionally narrow (10 mm) specimen was used for the quasi-isotropic lay-up in order to allow for fast degradation in compost. ASTM D 638 was applied for the isotropic plastic specimen. For the composites, the coupons were cut down to slices so that the width of a single specimen was 10,0 mm and the total length was 250 mm (gauge length 150 mm). The plastic specimens were manufactured with injection moulding to the standard ‘dogbone’ shape. Only for the first batch of the composite samples (reference series in ambient conditions), tabs were adhesively attached to the gripping ends of the specimens. Figure 3.23 presents the setup used in the tensile testing. For the extensometer, a gauge length of approximately 50 mm was used. In the Figure 3.23, the specimen in the jaws a Mater-Bi® specimen that has been aged in the compost bin for two weeks. For each of the specimens, the external extensiometer is used for the accurate measurement of the strain for the determination of the Young’s modulus. The extensiometer was detached from the specimens after the first 2.0 mm of displacement (extension), which made the testing faster for the Mater-Bi®-based specimens. For the composites and the PLA specimens, the extensiometer was kept attached until the end of the test (for the second batch).

For the composite specimens, lines were marked for the positions of the jaws and the extensiometer so that the data could be used in future for the modelling of flax fibre reinforced composites. The extension speed with the extensiometer was 2.0 mm/min. After extension of 2.0 mm, the tensile testing control program was set to stop, the extensiometer was detached, and the tensile test was continued with an increased speed of 20.0 mm/min until the breakage. For the calculations, extension and force data were recorded throughout the test.

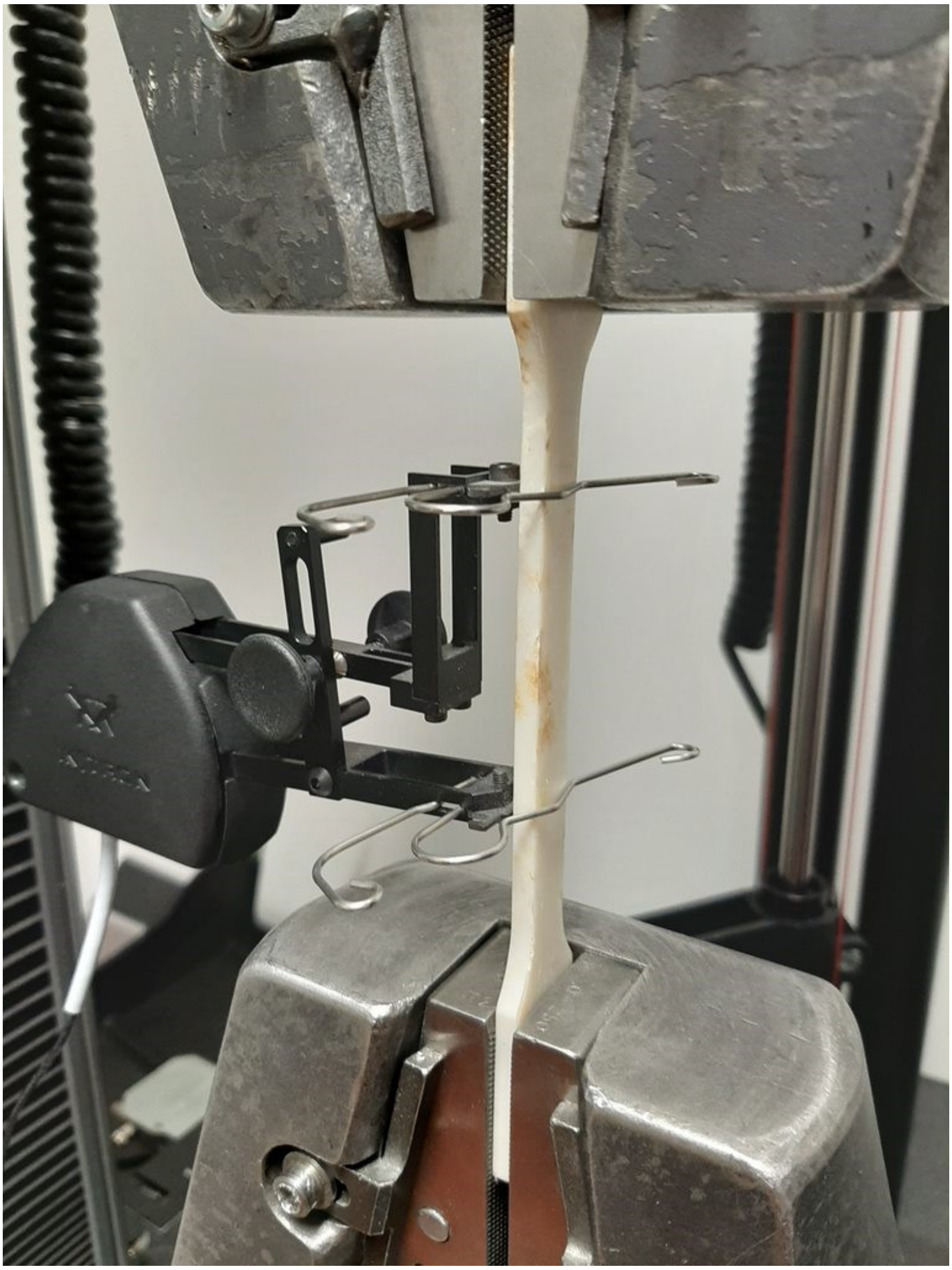

Figure 3.23: *Close-up image about the tensile testing system. The specimen in the figure is made of Mater-Bi® and is in the state of after the soil burial testing.*

The short-beam three-point bending test were carried out for the composite samples according to the ASTM D 2344 standard and the testing method in general is used to test the interlaminate strength of fibre reinforced plastics. For the testing, specimens were cut off from the coupon laminates and then grinded to the final dimensions of 8 x 20 mm. Before testing, the thickness for each of the specimens were measured, and this value determined the span length (pin-to-pin) in the three-point bending fixture. For the flax epoxy specimens, the span was 9.0 mm and for the flax thermoplastics, the span was 11.0 mm. The testing was done with the Instron 5967 electrical testing machine. In this setup, we had three support points (pins), and during the testing the force and the extension was recorded. The crosshead displacement rate in the bending tests was 1.0 mm/min.

3.5 Camouflage Radome Unit

The re-design for the Camouflage Radome Unit (CRU) is expexted to follow the basic requirements and specification defined and set by the Luxturrim5G project [www.luxturrim5g.com]. The electrical devices to be mounted inside are the same as in the current version of the CRU, which is presented in Figures 3.24 and 3.25. Figure 3.24 is a projection taken from the top side of the CRU and Figure 3.25 is a projection from the front side. These figures are captured from the 3D CAD model of the current CRU design. In Figure 3.25, the two octagon shaped profiles are the signal windows necessary for the 5G radios and the round profile in the middle is the cut-out for a speaker

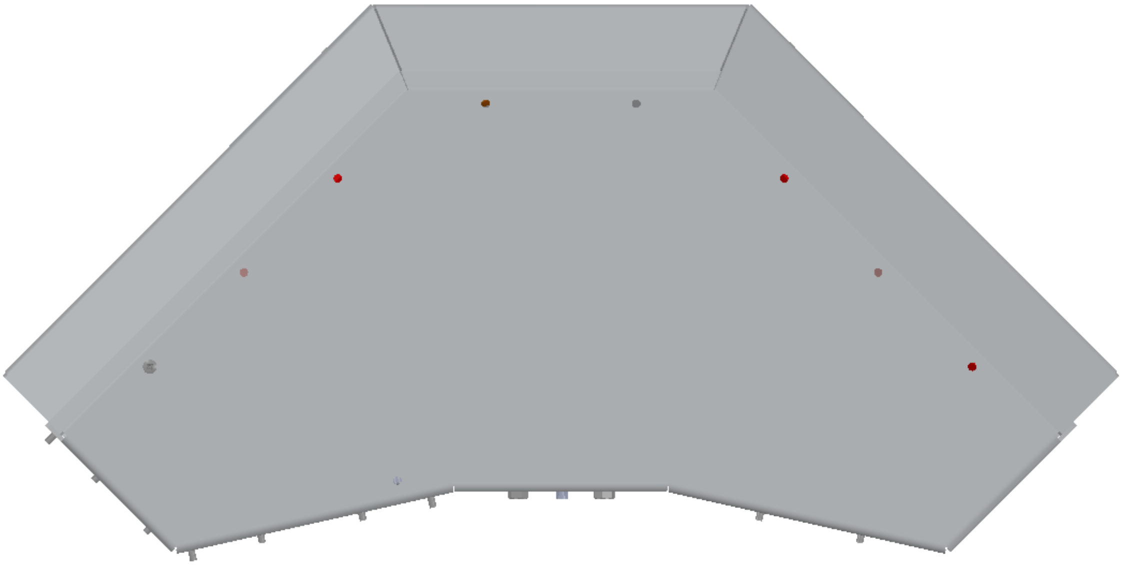

Figure 3.24: *Top view of the CRU – the original reference design.*

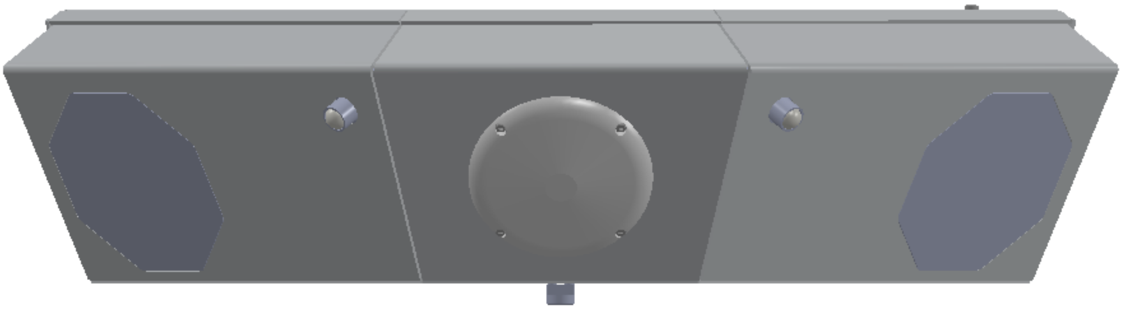

Figure 3.25: *Front view of the CRU – the original reference design.*

The position of the CRU in the Luxturrim5G smart light pole can be seen in Figure 3.26, which presents the head of a prototype of the light pole installed at the Nokia (Bell Labs) campus in Espoo. As seen in the figure, an older version ('version I') of the CRU is installed in this pole and below it is the light sources so that they wouldn't interfere with the signal to and from the radios. The radios, i.e. CRUs in this case, have to be placed every ≈50 m to provide good network and signal strength due to the short range of the ultra-high frequency signals.

Figure 3.26: *The position of the camouflage radome unit (CRU) in the light pole.*

In the next two chapters, I go through my work concerning the Camouflage Radome Unit (CRU). The first chapter, The re-design of the CRU unit through material selection, deals with the materials that could be used in the monocoque of a new CRU. The chapter covers the usage of natural fibres in such shell-structure applications. The second chapter, The fabrication of the CRU prototype, covers descriptions of the different phases and preparations during the manufacturing of the prototype I designed.

3.5.1 The re-design of the CRU unit through material selection

For the material selection, sustainability, mechanical properties, long-term durability, and safety are the key design factors for the candidate materials. In the project, the main goal is to develop and pilot technical solutions and concepts for 5G infrastructure and its platform based on smart light poles. My work aims to find alternative materials that could be used in the CRU instead of steel (in the current original design) which would meet the requirements but also act as a more sustainable solution for the final CRU version.

In modern days, various companies have started to research and develop new materials to replace conventional ones, which would allow more climate-friendly materials to be used in different applications. Especially for this master's thesis, the sustainability of the chosen material is to be observed. It's desired, that used material would have relatively low environmental impact compared to traditional ones (such as steels). For the re-design of the radome, material selection is revolved around using certain kind of fibre reinforced polymer.

For the reinforcements in FRPs, traditionally most common fibres have been glass, carbon and aramid fibres. However, the sustainability is a major factor concerning the re-design of the 5G component, so natural fibres have been studied as a substitute for the more traditional fibres. The factors affecting to the conclusion about the material selection are mechanical properties of the fibres, the form of the fibres, the compatibility with possible matrix materials, and the environmental impact of using natural fibres as a substitute for glass fibre in the composite. Overall, depending on the source of the natural fibres, they can be divided into three different main groups depending on the source: vegetable, animal and mineral (Rowe 2009, 7). For the sustainable CRU, commercially available vegetable fibres, more in detail certain bast fibres, are chosen as the reinforcing element.

The most common bast fibres used in industrial applications are flax, hemp, jute, kenaf and ramie. In Chapter 2.4, the basics of each bast fibre type were observed and which is used as a basis information when choosing the reinforcing element for the system in this thesis. From the mechanical point of view, flax fibres seem to have relatively high Young modulus and excellent tensile strength compared to other common bast fibres. For commercial availability, there are couple of companies in Finland that import natural fibers to Finland's composite markets.

The next step is to choose the form of the fibres used in the radome. The fibres used in fibre reinforced composites can be basically divided into two different groups: discontinuous and continuous fibres. The splitting to these groups is based on the fibre length compared to diameter (ratio), called as aspect ratio. For continuous fibres, this ratio is much larger in value compared to short fibres. The length of the fibres also determines, how they can be aligned to the composite structures, as continuous fibres are aligned to certain controlled direction – different to short fibres for a randomly oriented mat. Discontinuous fibres are generally easier to process and include lower costs, but composites made off them have generally worse mechanical properties. Due to the loss of mechanical properties and the rather simple geometry of the anticipated new CRU, continuous fibres were used instead of discontinuous reinforcements. Continuous fibre reinforcements can further be divided into unidirectional fibres, woven fibre fabrics, and roving fibers. From these alternatives, woven fabrics were chosen here to be used in the new CRU and the textile pattern style selected was a 2x2 twill fabric (easy to handle).

The components (devices) inside the CRU weigh about 5 kg and the temperature range of their operation is from 0 to 40 °C. The selected material should have high enough strength and stiffness to withstand these loads but also to function properly through the given temperature range. The weight of the components combined with the temperature range shouldn't be a limiting requirement to most of engineering materials (composites). However, due to the position of the CRU in a pole, materials should withstand the environmental factors like long-term humidity, freezing temperatures (below 0°C), wind (sway of pole) and direct sunlight. In addition to these, the machinability of the material selected should be good enough in order to let install the components inside the monocoque shell and to allow for processing technologies (fabrication of the shell).

The first concerning factor of the natural fibres taken into account was that hydrophilic fibres tend to have poor adhesion between hydrophobic petrochemical polymers. Different (surface) treatments have been applied to natural fibres by manufacturers to increase the adhesion between the matrix and the natural fibres. These different methods applied to fibres can be divided into physical treatments and chemical treatments. The physical methods are used to modify the topography, surface physico-chemistry, and/or functionalize the surface of the natural fibres. The physical treatments include methods like plasma treatments, where plasma is used to modify topography and physico-chemistry of the surface and/or creating functional groups (e.g. hydroxyl). In another method, irradiation, gamma radiation or electron beam is used to modify the fibres. The chemical treatments aim to functionalize surface of the natural fibres through certain ways such as esterification, treating with isocyanates, or treating with organosilanes. The processes used to do the chemical treatments can be divided to two different categories: wet and dry treatments. The wet treatments can be done in a liquid phase, vapour phase or using supercritical fluids. In the treatments done in the liquid phase, the fibres are immersed in a bath containing a solvent and the agent (reagent). In the vapour phase, the coupling agents are vaporized and spread on the fibres. The dry treatment processes include reactive extrusion, dry-blending, and dry powder impregnation. In the reactive extrusion, the coupling agents are added in the compounding phase during extrusion of the composite. In the dry-blending, pre-mixes containing fibres, polymer and additives are manufactured. In the dry powder impregnation, a thermoplastic polymer powder is first spread on fibres, then it is melted and finally solidified. (Le Moigne et al. 2018, 43-62)

Different treatments have been done and studied for natural fibres. Wu et al. fabricated hot-pressed flax-PP composites using two different kind of treatments: vinyltrimethoxy silane (VTMO) and maleic anhydride treated fibres (MAPP). The used flax was in the form of a yarn and two kind of polypropylenes were used, alpha and beta forms. Compared to the untreated system, MAPP increased both tensile and flexural properties, whereas VTMO had a higher stiffness compared to the untreated and MAPP-treated systems yet had the lowest tensile strength. VTMO increased the flexural properties the most that was caused by VTMO creating crosslinks between fibres and PP. (Wu et al. 2016) In turn, John & Anandjiwala used zein, a protein found in corn, to modify unwoven flax mats used with polypropylene. Zein was observed to enhance interfacial bonding between PP and flax, which led to a 14% increase in tensile strength and almost two times higher flexural strength for composite specimens containing 30% of flax fibres. (John & Anandjiwala 2009).

The materials used in the CRU structure have to withstand atmospheric conditions like ultraviolet radiation and humidity. The UV stability of plain fibres has been studied by a couple of researchers. Bi et al. conducted a study to test UV resistance of different bast fibres and compared the results to synthetic PET fibres. The results suggest that gum in the fibres functioned as anti-photoaging agent due to the gum absorbing UV radiation. Hemp had the best UV protection values from the measured bast fibres. Instead, flax and kenaf had best antiphotoaging properties and were able to retain about 60% of their initial strength. Bi et al. (Bi et al. 2019) studied the effect of environmental conditions on untreated and treated flax fibres by studying fibres behaviour at different levels of relative air humidity. In every case, the treated fibres absorbed less water from the air compared to the untreated fibres and the time consumed before the saturation was higher in every case except for a very high humidity (93% RH). The mechanical properties of untreated fibres had less variations compared to the treated fibres for different magnitudes of relative humidity. The highest tensile strengths for both fibres were observed at a 66% relative humidity level and a decrease was observed after this point for both fibre types, especially for treated fibres. (Stamboulis et al. 2001)

Instead of observing the effect of the environment on pure fibres, matrix and reinforcement systems must be analysed as a whole. Many researches have been made to study natural fibres used with certain matrix. In many cases, the hydrophilic nature of the fibres plays a major obstacle for the natural fibre reinforced polymers. This has been demonstrated in many studies, like in the study conducted by Khanlou et al. who exposed flax PLA composites to a very humid environments, water, and freezing temperatures. When totally saturated in distilled water, the tensile strength dropped about 53% whereas the dropping for the composites saturated with water (in 75% air humidity and 45 °C) was about 25%. The water uptake as saturation percentages were 22 in water and 2.6 in air. (Mohammed Khanlou et al. 2018) The same kind of degradation have been observed by many scientists to happen for thermoplastic and thermoset matrices (Mohammed Khanlou et al. 2018), (Scida et al. 2013). When PLA was used as matrix polymers, chain scission could be observed when the material was immersed in water (Duigou et al. 2011). Hygrothermal degradation in some degree can also be observed in composites when using synthetic fibres, but the degradation has not been reported to be as severe compared to NFRPs (Rocha et al. 2017),(Gibhardt et al. 2019). The water uptake can be reduced with certain surface-treatments (Alix et al. 2009), (Chen et al. 2011). When specimens were cyclically frozen and thawed, the tensile strength for the specimen dropped after 10 cycles, which could be caused by the 1,5% moisture content in the composites (Mohammed Khanlou et al. 2018). The UV aging has been proven to play a major role in the degradation of fibre reinforced polymers. It has been observed in many studies that the UV exposure caused degradation also for the natural fibre composites. In future applications, UV stabilizers should be used (Beg & Pickering 2008). However, when flax fibres were added to a hybrid PP composite containing flax and glass fibres, they were observed to prevent the UV degradation of PP compared to neat glass fibre reinforced PP composites (Ghasemzadeh-Barvarz et al. 2015). As mentioned before, the gum in natural fibres absorb the UV radiation thus protecting the fibres themselves and the matrix polymer.

In addition to the desired mechanical properties, the flammability of the material must be considered when new materials are used. In modern days, fire protection plays an important role in civil engineering applications to lower the risk of a major fire hazard.

The fire engineering can be further divided into two types of fire engineering: fire protection engineering and fire safety engineering. The purpose of the fire safety engineering is to create a design which provides protection for people and buildings so that the likelihood of injuries or death would be as low as possible. Different factors are, for example, sprinkler systems, firefighting possibilities, fire spread control and escape routes. (Smith et al. 2010, 1-2, 16) Fire protection engineering on the other hand deals with structural, mechanical, electrical and chemical part of the fire control. From the structural point of view, construction materials should have high enough strength (at elevated temperatures) and fire resistance to prevent collapse of frames and support the weight of a sprinkler system. From mechanical point of view, water flow through the sprinkler system must be calculated and air flow through the smoke control systems. In my case of the 5G era, the most important point of view for the object is the chemical standpoint and different kinds of hazards caused by the material, for example, possible source of ignition, fumes through burning etc. (Lataille 2002, 1-2) From fire engineering point of view, a CRU should have excellent flame retardancy and low degree of flammability. Because CRUs have to be placed almost every 50 meters in the city, they can spread the fire more quickly if the structure has a high degree of flammability. Also, destruction of the radios and antennas inside the CRU might cause network problems which then might lead to increased communication problems.

The burning process composes of five fundamental phases: heating, decomposition, ignition, and propagation. To increase the fire retardancy of a material, applied treatments and/or additives should prevent or slow down one of these five fundamental phases. (Kim 2018) Fire retardants as additives can be applied during manufacturing process or as a finishing coating for the part. On the other hand, applying fire retardants to fibre reinforced composites tend to have a negative impact on the mechanical properties of the composite (Elsabbagh et al. 2018, 128-131). If fire retardants are applied to the composite, one way is to impregnate natural fibers with fire retardant compounds before manufacturing, or the fire retardants can be applied during a manufacturing process. Other methods to decrease flammability is to insulate the composite from the heat, use fire retardant nanoparticles, or to apply a fire-retardant coating. (Kozlowski & Wladyka-Przybylak 2008)

In the case where NFRPs would be used, we would have two materials with a poor flame resistance: natural fibres and hydrocarbon rich polymer. Many of the bulk polymers have autoignition temperatures between 350-490 °C and flash ignition between 300-400 °C if enough oxygen is present and are able to sustain a fire once ignited. In the case of thermoplastics, materials often start to flow even before ignition which then may lead to the failure of the structure in question. (Horrocks 2001, 3-7) As mentioned in Chapter 2, bast fibres are lignocellulosic materials which are composed mainly of cellulose, hemicellulose, lignin, and other components in small quantities. These textiles, which are mainly composed of cellulose, pose a high fire hazard as being highly flammable (Killinc 2013, 45-46).

Flammability and fire retardancy of composites made from natural fibres have been studied by many researchers due to it being a critical factor in many applications. Standard for safety of Flammability of Plastic Materials for Parts in Devices and Appliances, UL 94, is commonly used to define fire retardancy of material by testing the fire spread rate horizontally or vertically in the specimen. Another commonly used testing device in measurements is cone calorimeter (Kozlowski & Wladyka-Przybylak 2008),(Kim 2018). Operational principle is to heat 100 x 100 mm specimen with certain power until it ignites or can be ignited with a spark. During the burning of the specimen, the fumes go to an exhaust duct where composition of the gases and the flow rate is measured. Device can be used to calculate heat release rate (HRR), mass loss rate (MLR), ignitability, smoke and soot, and possible toxic gases. The information got from the device can be used to compare the test materials, obtain thermophysical constants, as data for models and calculations, and for a regulatory compliance. (Janssens 2015, 911-912)(Babrauskas 2015, 952-953)

Sain et al. studied flame retardancy and mechanical properties of natural fibre PP composites with only magnesium hydroxide and small amounts of boric acid and zinc borate. For sawdust – PP composite, best flame retardancy was found when only magnesium hydroxide was mixed to the composite. Compared to pure composites, 25% of magnesium hydroxide in the mixture was able to decrease fire spread rate by 50% for both of the composites. The reduction of mechanical properties caused by magnesium hydroxide were from 11% in flexural modulus and 17% in tensile strength. For rice husk – PP composites, the respective values were 6% and 7%. (Sain et al. 2004) El-sabbagh et al. tested the flammability and mechanical properties of a flax-PP composite with different weight percentages of PP, flax and $Mg(OH)_2$ flame retardant. The weight fractions for the flax fibres were 30 and 50%, fire retardant ratios were 0, 20 and 30% and the rest of the weight was PP. LOI values for composites without additives were below 21, which means that they support flame progression in atmospheric conditions. However, values above 21 until certain point won't extinguish the flame but only slow it down. The best results were observed for the composite containing 30% flax when 30% $Mg(OH)_2$ was added. Horizontal burning ratio was reduced for 52%, stiffness value improved by 45% but strength decreased by 10%. For composites containing 50% flax, horizontal burning ratios were reduced for 71% and 93% using different amount of the flame retardants. From the results, it can be seen that by using more fibres in the system reduced the horizontal burning speed. (El-sabbagh et al. 2013) El-Sabbagh et al. have later on continued to optimize the amount of flame retardant that could be used for the flax PP composites. It was found, that category V-0 fire retardancy could be achieved using 40 weight percent of FR agents instead of predicted 67% without losing that much mechanical properties. (El-sabbagh et al. 2016)

Compared to glass fibres, natural fibres are highly flammable as mentioned above which can make NFRPs unsuitable to be used in certain applications without any treatments or additives. (Kim 2018) Chai et al. compared flammability by measuring HRR and MLR of glass fibre and flax fibre reinforced epoxy using different kind of fibre architecture: UD and two types of weave. The flax fibre composites ignited earlier, released more heat overall, and took more time to extinguish. Where natural fibres are highly flammable, glass fibres provide protection to the polymer matrix as a heat sink and physical barrier. (Chai et al. 2012)

Flammability of fully biodegradable composites have been studied by couple of researchers. Bocz et al. studied the fire resistance of a compounded PLA and thermoplastic starch (TPS) that was reinforced with chopped flax fibres using a multifunctional additive system. To increase the fire retardancy, some of the glycerol used as plasticizer for TPS was changed to glycerol phosphate. In addition to this, flax fibres were treated with phosphorous silane, and ammonium polyphostape was added to the system. Both glycerol phosphate and phosphorous silane had a positive impact on the flame retardancy by decreasing peak HRR values and increasing LOI values. However, only by adding ammonium polyphostapes at a rate of 10 weight percent increased LOI values dramatically and made the material self-extinguishing. (Bocz et al. 2014) Furthermore, Khalili et al. studied flammability, moisture absorption, and mechanical properties of an UD flax fabric reinforced PLA filled with nano-hydroxyapatite to increase the fire retardancy. Increasing the amount of the fire retardants had positive effect on the flame retardancy but negative effect on the mechanical properties. Also, an increase in the amount of nano-hydroxyapatite caused a higher moisture absorption which would indicate bad adhesion between the fillers and the rest of the composite. If such a case, compromises should be done between the fire retardancy and mechanical/moisture absorption properties. (Khalili et al. 2019)

The final criteria for choosing the reinforcement is the sustainability of material. In this work, I have chosen to compare composites manufactured of flax fibres to ones made from glass fibres. Glass fibres have larger tensile strength and less variation between different glass fibres (filaments) where the quality of flax fibres may vary a lot. However, the density of flax fibres is less compared to glass fibres and when this is taken into account in a comparison, glass and flax fibres have about the same (normalized) stiffness to density ratio (Summerscales 2010). Natural fibres are completely bio-based and biodegradable whereas glass fibres are synthesized from certain ceramics, mainly silicon dioxide, and glass fibres do not biodegrade (is a mineral system). From this point of view, flax fibres can be seen a superior option over synthesized fibres like a glass fibre. Still, the embodied energy through processing for both types of the fibres must be taken into account to determine the sustainability. Although flax fibres are biodegradable, when used, for example, with polyester or epoxy matrix, biodegradability doesn't give material any extra value due to a non-biodegradable matrix. Different LCAs have been reported to evaluate the impact of manufacturing natural fibres and the usage in composites.

The embodied energy assessment during a processing of flax fibres have been conducted by couple of researchers. Nilmini et al. conducted a life cycle assessment to evaluate the energy used to produce flax fibre reinforcements and then compared the results to that of glass fibres. Total energy used for agricultural operations were between 7,5 and 9,6 GJ per one tonne of yarn. When pesticides and fertilisers are used, the amount of energy consumed was increased by 41,79 GJ per one tonne of a yarn. This is increased to 59 GJ per one tonne of sliver and up to 86 GJ per tonne of a yarn after spinning of the sliver. A comparison to glass fibreis needed; glass fibre mat requires 55 GJ per tonne compared to 59 of the flax. The production of continuous glass fibre (for yarn, bundle) requires 26 GJ where the responding value for flax is 86 GJ. (Dissanayake et al. 2009) However, the analysis by Dissanayake et al. did not take into account the by-products created during the cultivation. In another study conducted by Le Duigou et al., the usage of the by-products was counted as usable resource and made the analysis based on the production of hackled fibres that does not include the spinning phase. In this LCA conducted, the production of hackled flax fibres was seen as environmentally friendlier compared to the production of glass fibres and the only aspect, where the production of glass fibres was environmentally more sustainable, was the land occupation and europhication through the usage of fertilizers and pesticides. The embodied energy to produce hackled flax fibres is mainly dominated by fertilizers and pesticides but other than that the production of hackled flax fibres doesn't include any energy intensive steps. (Le Duigou et al. 2011) Yelin et al. used consequential life cycle assessment (CLCA) to evaluate and compare environmental impact of flax-PP mat fabricated from both French and Chinese fibres to glass-PP mat. It was observed, that flax production in China has in overall higher environmental impact compared to glass fibres even when the end-of-life incineration is taken into account. On the other hand, if flax fibres would be manufactured in France, the environmental impact would decrease with an exception of land usage and freshwater ecotoxicity. (Deng & Tian 2015) From the results of the LCAs done about flax fibre production, the environmental impact seems to be dependent on the origin country of the flax product, the form of the flax used in the composites, and the utilization of the by-products.

For more in-depth type of a re-design, it is also advisable to re-design the shape of the CRU to make the part visually more attractive for consumers, but also to keep the design practical. In the re-design, almost every component excluding signal windows would be installed on a baseplate, whereas in the previous (original) design the components were installed on a horizontally located bottom plane. In this thesis, I approached the design from a different point of view by suggesting the baseplate to be vertical as an alternative. This way the width of the design would be smaller compared to previous designs yet the radome will be taller (in the pole main axis direction). In this case, the minimum width is set by some of the space needs of larger components (typically radios) inside the CRU. The components inside the CRU can be also installed vertically compared to the present design, where they are horizontal. In Figure 3.27, the new proposed shape is presented. The aim was to make it more round-shaped but still a bit angular to make the look more technical. The radome is divided to two distinct parts, the baseplate and the ‘cover’.

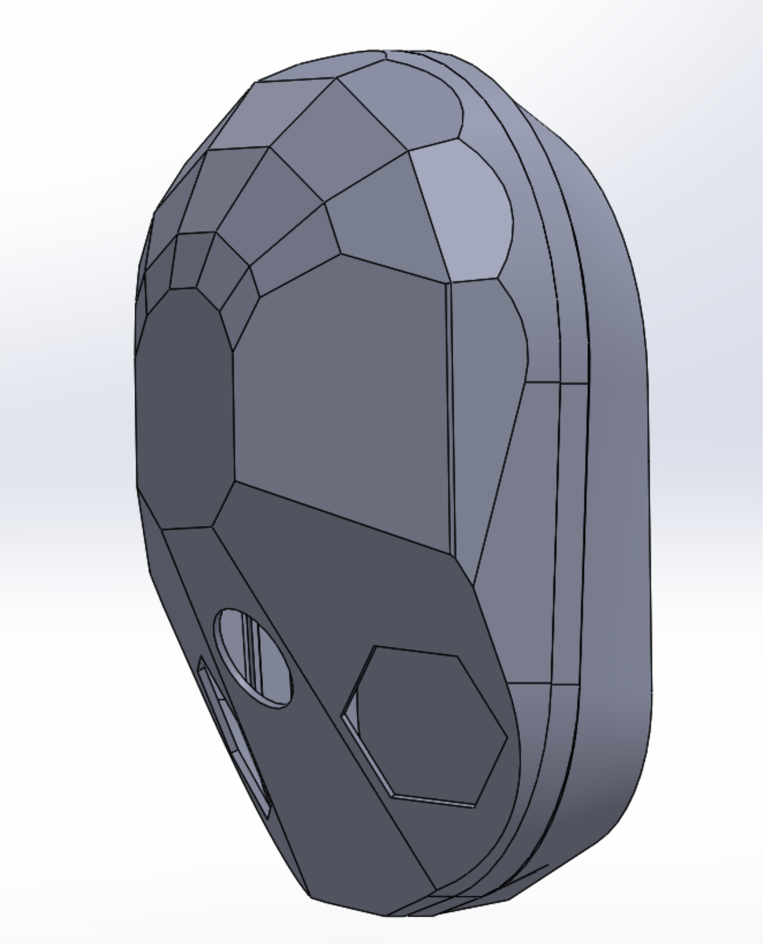

Figure 3.27: *The new proposed shape for the CRU (re-design geometry)*

In Figure 3.28 the baseplate for the new proposed shape is presented. The big square-shaped hole in the middle is the point where the CRU is attached to the pole. The devices inside the CRU are installed in to racks which are installed near the hole. Using the new proposed shape, I have made simplified calculations which would give the minimum wall thickness for the baseplate to support the load due to the devices.

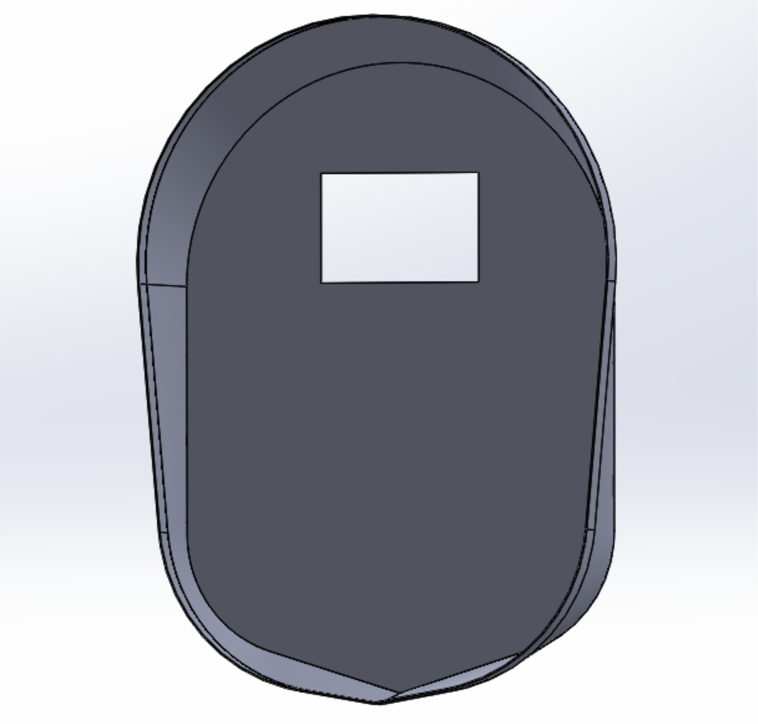

Figure 3.28: *The baseplate for the new proposed shape*

To estimate the minimum required wall thickness, the width of the baseplate in the middle is calculated. In this case, the width is about 500 mm. The estimated (i.e. quasi-static assumption and no stress concentrations considered) limits calculations for the wall thickness are determined by the tensile strength of the component (wall material). For more in-depth type of calculations, we would create a numerical model, add screws to the model etc. and determine where the highest stress concentrations are present (i.e. ‘hot spots’). For the conceptual design here, we use two formulas. The first formula (1) is used to determine the level of force (F) using acceleration (a) and mass (m) of the object (devices inside the CRU in this thesis). The second formula (2) is used to calculate average laminate stress (σ) by dividing the subjected force by the cross-section area (of the baseplate) (A) where it is focused on

$$F = ma \tag{1}$$

$$\sigma = \frac{F}{A} \tag{2}$$

By using these two formulas, we can make an equation to solve what would be the required thickness for the wall. In the equation (1), acceleration is equal to the gravitational acceleration ($g = 9{,}81\frac{m}{s^2}$). The area in Formula (2) can be divided to two different factors, which are the width (*W*) and thickness (*T*) of the baseplate back wall. From these two formulas, we can create Formula (3) to solve the required wall thickness. In addition, the safety factor for the wall thickness is determined to be $SF = 2$.

$$T \geq 2 \times \frac{mg}{\sigma_{u,critical}W} \qquad (3)$$

For the calculations in this thesis, $m = 5\ kg$ and $W\ =\ 500\ mm$. The stress allowable $\sigma_{u,exp} = 56{,}1\ MPa$ is determined in the section: Results. Hence, by substituting the values to Formula (3), we can calculate the necessary wall thickness for the flax epoxy that has been exposed to soil burial being about 3,5 μm. Typically, in this type of application, the wall thickness are even several millimetres. From this result, it can be determined that the mechanical requirements for the part are not critical with the material studied in this thesis. For more in-depth type of calculations, modelling should be done to solve out the criticality of possible stress concentrations.

3.5.2 The fabrication of composite CRU prototype

A prototype of the sustainable CRU was manufactured for the Luxturrim5G project's finale, The Impact Day. As mentioned in the previous chapter, the reinforcement selected for the new CRU was flax (200 g/m^2 2x2 twill) fabric produced by Biotex and the polymer used in the composite was Super Sap® epoxy resin produced by Entropy Resins. Manufacturing process to produce the prototype was a hand lay-up combined with vacuum bagging. The CRU consists of two different parts: a hull and a top cover. Also, for the project finale, two laminate demos were fabricated to demonstrate the usage of other alternative composite materials. In Figure 3.27 the laminate demos are presented. The left-side laminate in the figure contains woven flax fibre fabrics (200 g/m^2 2x2 twill, Biotex), Super Sap® epoxy (Entropy Resins), and powerRibs® flax supporting reinforcement element produced by Bcomp Ltd. The right-side laminate in the figure contains flax fibre fabrics (200 g/m^2 2x2 twill, Biotex) with PLA matrix. However, the main focus of the thesis for the CRU was todevelop a process and produce a sustainable prototype of the CRU. The other (demo) laminates were used only as supporting demonstrations of alternative materials and their manufacture.

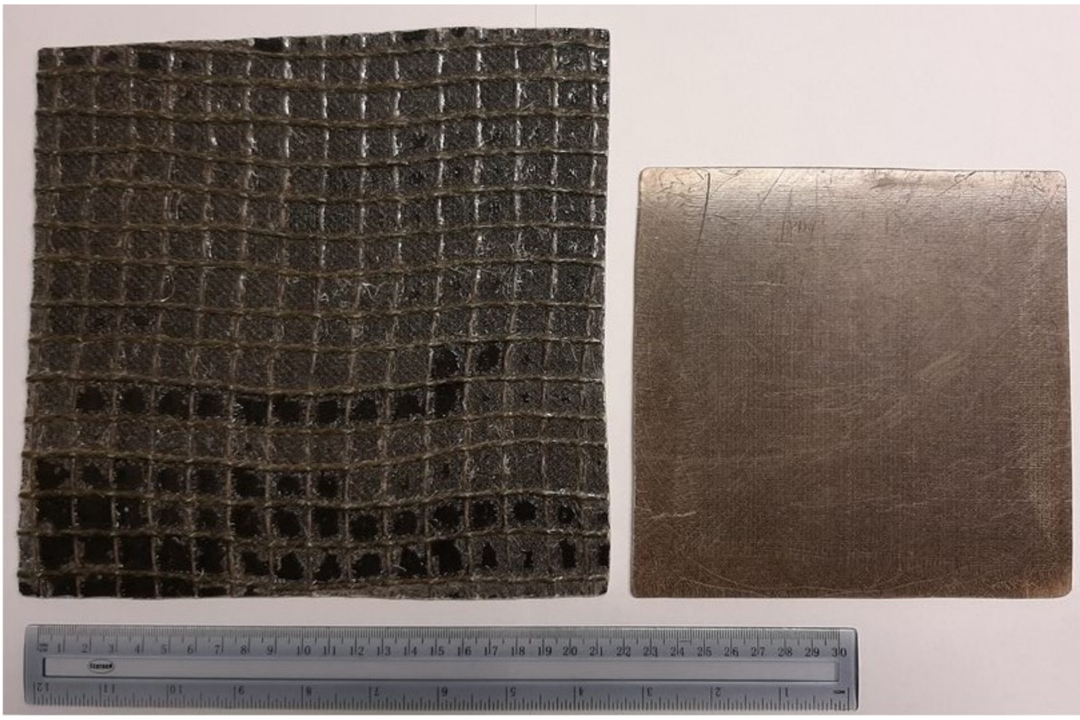

Figure 3.27: *Two demonstration laminates of the alternative composite materials for %G applications. Left-side laminate is flax fibre reinforced epoxy combined with flax fibre supporting reinforcement element. The right-side laminate is flax fibre reinforced polylactic acid.*

To fabricate the sustainable prototype for the Luxturrim5G project finale, the Impact day, a mould was needed for the hand lay-up process. In this case, a male mould was designed and manufactured. The moulds of the hull and the top cover for the hand lay-up were prepared of a block of EPS 'high density foam' using hot-wire foam cutter, handsaw, pocket rasp, and sandpaper. The hot-wire cutter and handsaw were used to achieve rough outlines for the moulds (step 1). After this, a pocket rasp (step 2) and sandpaper (step 3) were used to round the corners and to give the moulds smooth surface. Figure 3.28 presents the finished mould for the hull and Figure 3.29 presents the mould for the top cover. Small variations in the dimensions compared to the original (CAD model) are not critical due to the prototype being shown as a demonstration of composite materials and their usability.

Figure 3.28: *Fabricated EPS mould for the hull in this thesis for a CRU prototype.*

Figure 3.29: *Fabricated EPS mould for the top cover in this thesis for a CRU prototype.*

The hull and top cover laminates were made of a stack off four layers of flax fibre fabrics and one layer of peel ply to give the prototype an appropriate surface finish (in mass production this is typical for paints). For the hand lay-up lamination process, the EPS moulds were covered with aluminium foil to prevent the epoxy resin from diffusing and sticking to the mould surface. For the hull, the lamination was done by first placing one layer of fabric on top of the mould and then pouring (572g) mixed epoxy resin on top of the fabrics. After the pouring of resin, brushes were used to spread the epoxy resin over the fabrics. When the first fabric was impregnated with the mixed epoxy resin, the second fabric layer was positioned on top of the mould before pouring another dose of the epoxy resin. In Figure 3.30 a picture taken during the hand lay-up is presented. In the figure, the first flax fabric layer has been placed on top of the mould and the epoxy resin has been poured on top of it. On the left, it is shown how the folding of the corners was made to achieve desired shape.

Figure 3.30: *Fabrication of the CRU hull in this thesis for a CRU prototype.*

After applying all the fabrics given in the lay-up design and epoxy resin, the parts were vacuum bagged. Due to the large height of the hull and the lack of an advanced vacuum bagging equipment, the vacuum bagging caused some problems during the manufacturing. The vacuum bag was prepared from a plastic sheet that was first folded to form a bag and then sealant tape was used to fasten the edges. For the shallow top cover, the vacuum bagging was clearly easier to do due to a plate-like shape of the top cover. For the hull and the top cover, vacuum bagging (dwell time) lasted overnight before removing the cured composite parts from the vacuum bags. After the removal, the peel plies were removed from the parts. In Figures 3.31 and 3.32 the hull and the top cover of the new CRU are presented before the finishing treatments.

Figure 3.31: *CRU hull before finishing treatments in this thesis for a CRU prototype.*

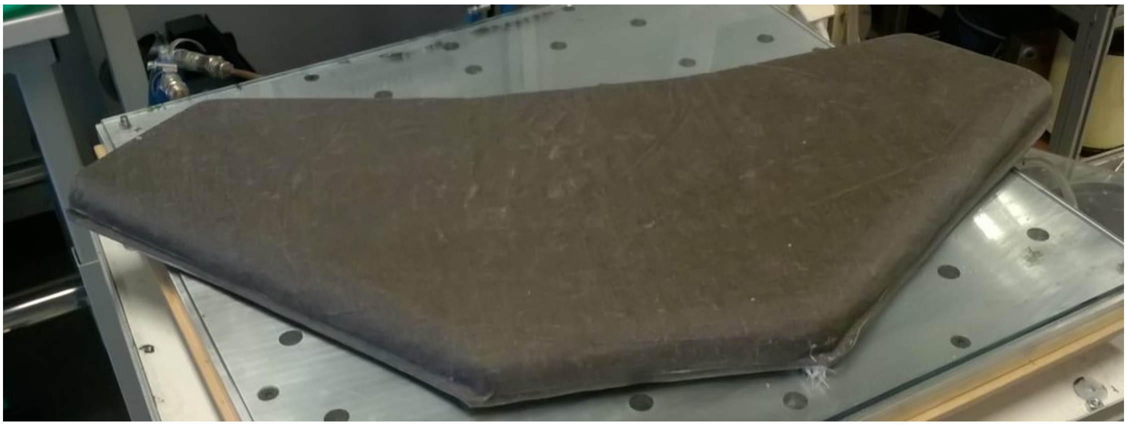

Figure 3.32: *CRU top cover before finishing treatments after vacuum bagging and before the finishing treatments.*

For the finishing treatments, an angler grinder and a jigsaw were used. The angler grinder was used to trim non-essential fillets and edges away from the parts. After removing the edges and the fillets, the angler grinder was used to smooth the surfaces of the parts. Also, some unwanted details were grinded away from the prototypes.

The cut outs for the signal windows and the speaker, the angler grinder was used to make lines for jigsaw, which was then used to cut the pieces off. Finally, the EPS block was carved out from the hull using various tools. For the top cover, the mould was removed off easily. For the final treatment, acryl lacquer was sprayed on top of the parts. Figure 3.33 presents the hull of the prototype and Figure 3.34 presents the top cover. In Figure 3.33, the white stripes in the hull are caused by the problems to fold the fabrics finely during the hand lay-up of the layers. When the angler grinder was used to smooth the surfaces, material had to be removed from the upper region of the CRU that caused the visual ‘white’ stripes. Due to the simpler shape of the top cover, the surface quality of the part was better.

Figure 3.33: *The CRU hull after finishing treatments to smooth outer surfaces.*

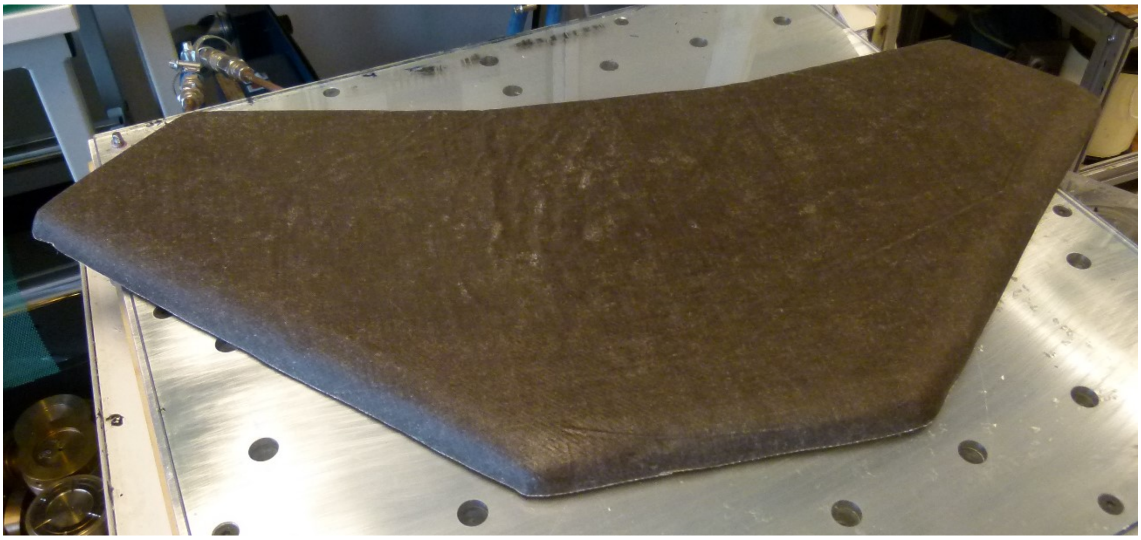

Figure 3.34: *The CRU top cover after finishing treatments to smooth outer surfaces.*

The sustainable prototype of the CRU was showcased during the Luxturrim5G project finale, The Impact Day, held at Nokia's Karakallio premises (27.5.2019). The aim was to demonstrate the usage of alternative materials to decrease the environmental impacts of different products and still have the required performance. For the sustainable version of the prototype achieved in this thesis, the most important goal was the usage of flax fibres instead of traditional carbon or glass fibres. Also, the applied epoxy resin was partly manufactured off bio-based polymer components so that the environmental impact of the product is clearly reduced. To increase the sustainability of the 5G CRU in future, the matrix could be re-selected from epoxy to certain well-adjusted biodegradable thermoplastic material, like PLA, or some liquid reactive thermoplastic resin. However, this type of re-selection will force to re-design the CRU manufacture as well.

4. RESULTS

The results in total are divided to three different topics of the thesis: CRU case study, the processing effects of materials, and the effects of soil burial. In the first chapter, CRU case study, I go through the findings of the literature survey about the flax fibre reinforced composites, and the impact of other factors, that should be taken account when re-designing the CRU through material selection. In the second chapter, Processing effects of the materials, I go through the observations I made during the fabrication of the laminates for the work. In the third chapter, Effects of soil burial, I go through the results from the mechanical testing of the materials and how the soil burial testing did affect the mechanical properties.

4.1 CRU case study

At the moment, the automotive industry is using more and more NFRP components in their new car models. Although the usage of natural fibres in composites is still at its early stages, the materials could be a decent competitor to traditional composite materials like GFRP or CFRP. Compared to these materials, with the usage of flax fibres, we could achieve environmentally more sustainable products compared to either of the composites using traditional reinforcements. Nowadays and in future, the recycling and recovery of traditional composites poses a major problem. The recycling of GFRPs isn’t economically viable due to the low price of virgin glass fibres and even when the fibres are recycled, the mechanical properties deteriorate a lot. The recovery process through incineration for GFRPs isn’t so effective due to the high glass concentration of the composites. For these reasons, GFRPs are being dumped to landfills. In future, for example, when wind turbines come to their end-of-life stage, people will already have lots of waste to deal with.

From the mechanical point of view, properties of NFRPs are somewhere between glass and carbon fibre reinforced composites depending on the exact optimization criteria. When compared to GFRPS, natural fibres have in average better aspect ratio for stiffness. By substituting glass fibres with natural fibres, we could achieve lighter structures with required stiffness especially in chopped fibre composites. When the mechanical properties of natural fibres are compared to mechanical properties of carbon fibres, carbon fibres have much better mechanical performance. However, natural fibres could offer a cost-efficient option in situations, where lightness vibration damping are desired properties but the mechanical performance doesn’t require the usage of carbon fibres.

Based on the literature survey, the flax fibres as reinforcing element has its own problems regarding the usage in the CRU design. The first of challenges is the uncertainty of the quality of the flax fibres and another is the long-term durability in outdoor atmospheric conditions. One of the major physical problems for the flax fibres seems to be its hydrophilic nature. In the 5G application case, where air humidity could enter the flax fibres in the composite, it could cause severe deterioration as observed in the studies in current literature. Also, the negative effect of the freezing-thawing cycles would cause further durability problems in the regions of the world where freezing temperatures occur frequently. The UV stability of the NFRP seems to be decent due to the presence of the gum in the fibres, which can absorb the radiation thus protecting the fibres and the polymer.

From the safety point of view, the fabrication of the flax fibres appears to be safer compared to the glass fibres. However, problems in the safety could be caused by the poor flame resistance of the NFRPs. The cellulose fibres are very flammable materials and, in average, polymers are also able to sustain the fire if they are ignited. If the NFRPs would be used in CRUs, they could be major flame hazard as the flames in the cities could spread through them. Also, as the CRU contains the radios used in the 5G network, the destruction of the 5G equipment could cause communication problems through the weakening of the mobile network.

If flax fibres would be used in this application, perhaps certain treatments should be done to modify the material. For a start, some treatments should be done to enhance the adhesion between the fibres and the polymer. As flax fibres are hydrophilic materials and traditional polymers are hydrophobic materials, the adhesion between these two is poor which would then require modification of polymer or the flax fibres (like a silane treatment). This would offer better mechanical properties to the material but also prevent it from aging in some degree. Good adhesion between the fibres and the matrix was also found to improve the water-resistance of the NFRPs. Also, some UV stabilizers should be added to the system as the lifetime of the product has to be many years although the gum in the fibres could protect the composite from UV radiation. For the safety point of view, the flame resistance of the components made from NFRPs compose a major fire hazard. One way to prevent this would be to add flame retardancy agents to the composites. However, these agents have been observed to have a major negative impact on the mechanical properties of the composite materials. Another method would be to coat the components, which could be seen more favourable from the aspect of the mechanical properties. In either case, some coating would be applied on the component to at least to increase the permeability of water and to adjust aesthetics.

One aspect of the long-term durability regarding the work here is the possible biodeterioration of the material. Although the polymer itself wouldn't degrade, the cellulosic fibres can be harmed by microbiological activity which could lead to preliminary failure of the material. To prevent this, biocides could be added to the system, but also good adhesion between fibres and the matrix through certain treatments would prohibit biodeterioration. Rosin, for example, should partly prevent the microbial activity and also be biosourced material. However, during this thesis, the addition of the rosin was observed to have a negative effect on other factors like absolute mechanical properties and the processability of the composites. In future, the processing parameters could be optimized to create completely bio-based composite structures.

The current (original) design of the CRU would be problematic to produce from continuous fibre reinforced polymers at a high volume. The top cover could be produced with a RTM or VARTM process, but the hull would cause some challenges. For the solution, there are basically two options: to change the material to short fibre reinforced polymer that could be injection moulded or to change the design of the CRU shell. At this moment, the CRU could be fabricated with a bladder mandrel (inflation) but still the desired volume of the production of the CRUs would be hard to achieve using continuous fibres with thermoset matrix. Simpler design could solve the problem which would then allow the usage of traditional methods like a RTM or VARTM with thermosets of thermoforming with thermoplastic composites. If short fibre reinforced composites would be used in future, the mechanical properties of the material could cause some re-design (wall thickness change) but the fabrication of the CRUs current (original reference) design could be achieved at big volumes through injection moulding.

The fabrication of the sustainable CRU prototype showcased the possibilities in the usage of alternative materials, which could at the same time offer the required mechanical properties and have a reduced environmental impact. As mentioned before, flax fibres have good mechanical properties, are relatively cost-efficient, and have theoretically abundant source. To increase the sustainability of the product, the epoxy resin could be changed to some more sustainable materials like PLA or some liquid thermoplastic resin.

4.2 Processing effects of materials

In the fabrication study of the materials for the thesis, certain observations were made in their behaviour and effect on the processing parameters. Especially, the pine gum rosin (Forchem) as an additive caused a lot of adjusting in certain material series for the thermoplastic materials. During the first step to produce the thermoplastic NFRP, rosin had to be first compounded with PLA and Mater-Bi®. Due to the low melting point of rosin, it tended to melt in the hopper before entering the screw. This premature melting of the rosin particles choked the hopper, so that rosin and polymer granulates didn't enter the extruder properly. This was somewhat fixed by properly adjusting the feeding speed of the granulates, so that the screw inside the extruder was partly empty throughout the run to avoid the rosin exiting from the extruder into the hopper section. Due to the high temperatures during the extrusion process and shearing of the materials, there probably has been degradation of rosin, PLA, and Mater-Bi® in some degree.

Rosin was observed to have major impact on the processing parameters compared to pure materials. For the materials (blends) containing rosin, the optimization of the processing parameters was a longer process and it was found that same parameters could not be used with pure material and the blend containing 10-m% of rosin. PLA caused problems during the injection moulding due to the small heat shrinkage so that the ejection mechanism in the mould couldn't detach the specimen from the mould. Thus, the detaching had to be assisted manually. For PLA containing rosin, a longer holding pressure with higher time were observed to fill the mould completely so that the filling of the mould was more about controlling the pressure in the mould (instead of the speed of the screw). Higher pressures were observed to cause severe flashes and sinks at the same time to the specimens.

Problems were also observed during the hot-pressing of the NFRP laminates. The first effects observed during the 'training' of the usage of the hot-pressing machine were notices made in the usage of flax fibre fabrics. In Figure 4.1, it can be seen a close-up image from one of the trial pressing laminates done with flax and HDPE. There are two major issues in the figure that also had to be taken into account during the pressing:

1) higher pressures were observed to damage the fabrics and,
2) moisture in the fabrics.

It was observed that higher pressures broke down the flax fabrics. Another issue was the bubbles inside the polymer caused by the moisture of the flax fibre fabrics (even after dehydration). For these reasons, low pressures and proper drying of the fibres had to be used to fabricate NFRP coupons by hot-pressing.

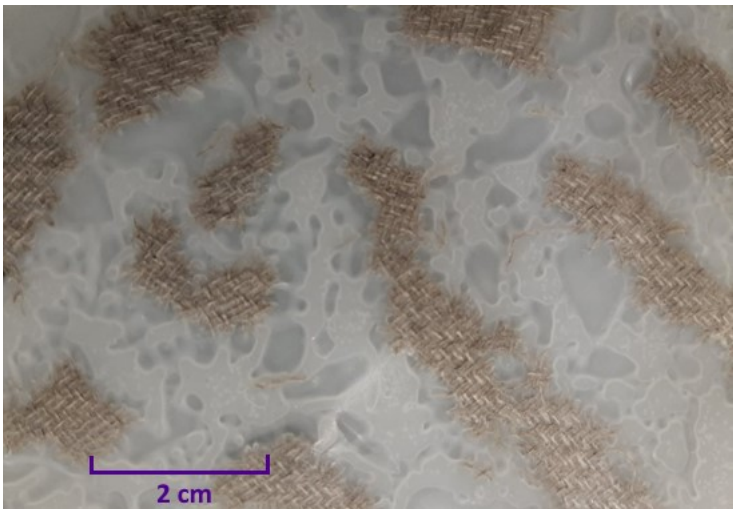

Figure 4.1: *Close-up from the flax – HDPE practice specimen.*

Just as during the extrusion and injection moulding, rosin caused major effects on the hot-pressing of laminates. Due to the low melting point of rosin, it had impact on the melting range of PLA containing rosin that then affected the melt viscosity. For hot-pressing, a blend was observed to have too low viscosity, which then had impact on the quality of the NFRP laminates. The more rosin the composites had, the worse was the quality of the fabricated laminates. In Figure 4.2, a close-up image is presented from the surface of rosin-treated flax with PLA compounded with rosin. As it can be seen, there are lots of cavities in the laminates that were not observed with pure PLA (hot-pressed sheets). For pure flax with PLA rosin blend, the composite quality was better compared to the RtFPR but small bubbles or voids could still be observed in the surface. In some cases, the laminas of the RtFPR laminates could be just ripped off from each other by hands indicating low interlaminar adhesion.

Figure 4.2: *A close-up image from the surface of a RtFPR laminate.*

Other than mentioned above, there was not any remarkable observations made during fabrication of the samples. Although rosin is natural material and has good antibacterial properties, it had a negative impact on the processing of the materials. In future, if rosin would be used, the processing parameters would need much more optimization and less rosin could be used during the rosin treatments of flax fibres.

4.3 Effects of soil burial

The materials during the thesis work were analysed by a method where they were buried in soil. This was made to see the effect of microbial activity on the materials and to see how rosin affected to the system from degradation point of view.

In Figure 4.3, the standard tensile test data is presented for the polymeric specimens. In Y-axis, is the force data given, and in X-axis, is the extension of the specimen per point of force pair. The black curves are the tensile test data of reference specimens before the soil burial testing and the purple curves are the data for the specimens that have been in the compost bin for two weeks (after drying). In the plots, graph a) is the data for pure PLA, graph b) is the data for PLA containing rosin, c) is the data for pure Mater-Bi$^{®}$, and graph d) is the data for Mater-Bi$^{®}$ containing 10-m% of rosin.

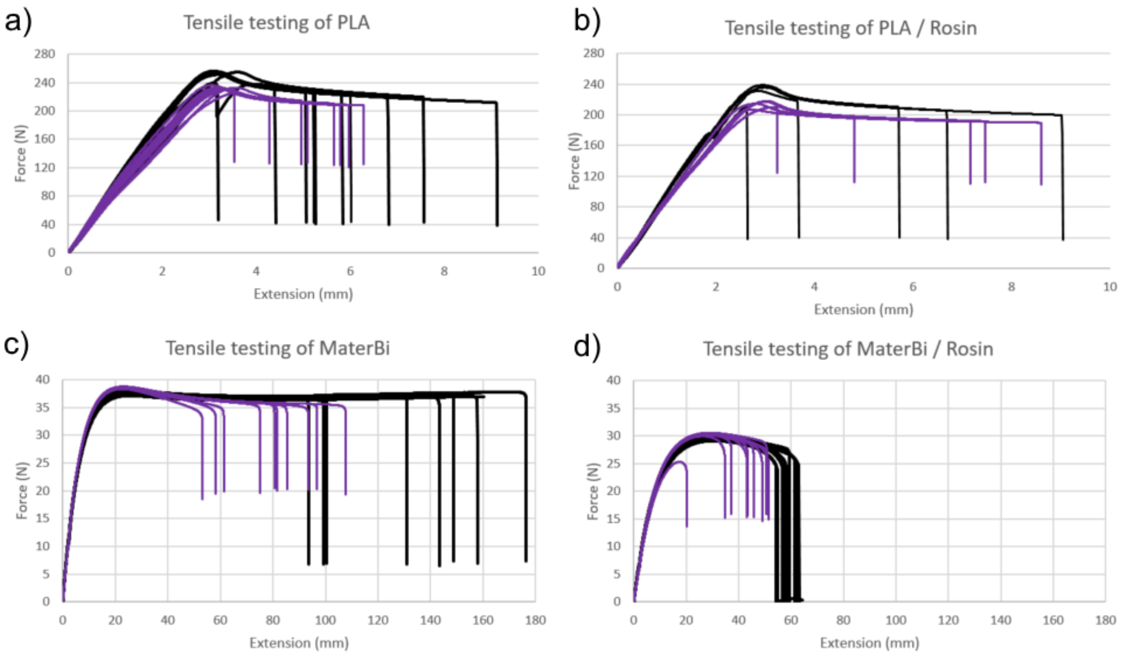

Figure 4.3: *Tensile test raw data for the polymeric materials: a) PLA, b) PLA rosin blend, c) Mater-Bi$^{®}$, and d) Mater-Bi$^{®}$ rosin blend. The black curves represent the reference (prior to 2 weeks of composting) data.*

In Figure 4.4, the pure raw tensile test data for the flax-reinforced composite samples is presented. On the Y-axis, is the force data, and, in X-axis, is the extension data per force pair. The black curves are the tensile test data of reference specimens before the soil burial testing and the purple curves are the data for the specimens that have been in the compost bin for two weeks (after drying). The curves are for following materials: graph a) data for the flax Epoxy (FE), graph b) is data for the flax PLA (FP), graph c) is the data for flax PLA + rosin blend (FPR), and graph d) is the rosin treated flax PLA + rosin blend (RtFPR).

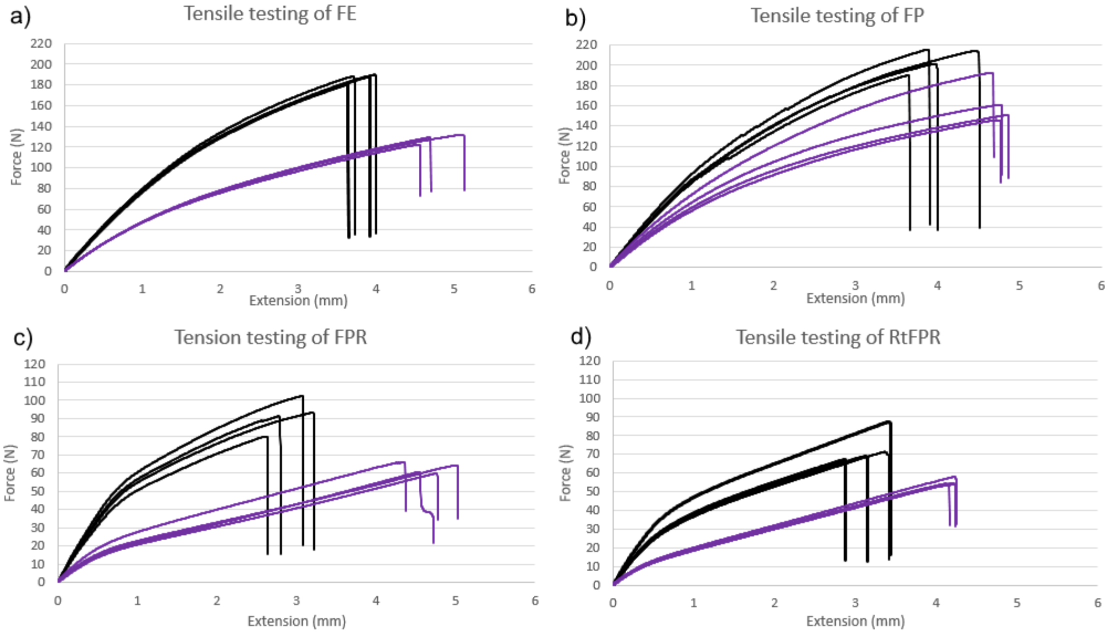

Figure 4.4: *Tensile test data for the composites. a) Flax – Epoxy (FE), b) Flax – PLA (FP), c) Flax – PLA / Rosin (FPR), and d) Rosin treated Flax – PLA / Rosin (RtFPR)*

For a more specific analysis of the effects of the soil burial, the values for the determined mechanical properties (engineering constants) for the polymeric materials are marked to Table 4.1. For each material the ultimate tensile strength before and after soil burial is marked, the average ultimate extension before and after soil burial, and the difference in these values due to the soil burial aging. For both types of PLA samples, the differences due to soil burial can be seen mostly as the loss of ultimate tensile strength. For Mater-Bi® samples, the ultimate extension is reduced but the ultimate tensile strength got improved.

Table 4.1: *Determined mechanical properties of the developed polymeric blends and pure materials before and after the soil burial (SB). The cod – R in the end of series name means that the material contains 10-m% of rosin.*

Material: Property:	PLA	PLA + R	Mater-Bi®	Mater-Bi® + R
Tensile strength, before SB (MPa)	61,5	56,5	9,28	7,22
Tensile strength, after SB (MPa)	56,9	52,3	9,46	7,30
Decrease in tensile strength (%)	7,62	7,56	-1,96	-1,18
Ultimate extension, before SB (mm)	5,81	5,52	134,4	58,3
Ultimate extension, after SB (mm)	5,16	6,26	79,4	45,1
Decrease in the extension (%)	11,2	-13,2	79,4	45,1

To analyse the effects of soil burial, values for the mechanical properties for composite samples are marked to Table 4.2. For each composite material, what has been marked are the ultimate tensile strength and interlaminar shear strength before and after the soil burial, and the change in the mechanical property for both of these measurands in percentage.

Table 4.2: *Mechanical properties of the developed composite materials before and after the soil burial (SB). FE = flax epoxy, FP = flax PLA, FPR = flax PLA-rosin blend, and RtFPR = rosin-treated flax PLA-rosin blend.*

Material: Property:	FE	FP	FPR	RtFPR
Ultimate tensile strength, before SB (MPa)	79,6	71,9	56,3	40,2
Ultimate tensile strength, after SB (MPa)	56,1	60,1	36,0	31,6
Decrease in ultimate tensile strength (%)	29,5	16,4	36,1	21,4
ILSS, before SB (MPa)	18,6	18,4	10,2	4,75
ILSS, after SB (MPa)	18,0	16,6	4,2	2,1
Decrease in ILSS (%)	3,3	10,1	58,3	55,7

5. DISCUSSION

5.1 The sustainable Camouflage Radome Unit

The work in the was divided to different topics, which of each are covered here separately starting by the fabrication of the sustainable prototype. As the idea was to fabricate a sustainable version of the metallic (steel) version, flax fibres and a partly bio-based epoxy were chosen as the materials for the prototype. The prototype itself was a demonstration specimen to showcase the possibilities in the field of materials science and using alternative materials in 5G applications. At least nowadays, it’s important to demonstrate the sustainable ways to produce different products, in this case the component of a smart light pole, to attract modern society. As the sustainable prototype was shown to public during the project finale, the Impact Day, it could draw attention of many people from different companies and media. Through publicity like this one achieved, the usage of the natural fibres in different products may increase as more and more people and industries are aware of this kind of alternative materials that exist and can be used in different load carrying multifunctional applications.

In this thesis case, where natural fibres would be considered as being used in the 5G CRUs, there must be more advantages compared to traditional materials like steel or GFRP. One of the most obvious one is the sustainability, and the second being light weight. Although, nowadays, the environmental friendliness and sustainable manufacturing are emphasized, this often comes with a higher price tag. As the smart light poles themselves are not the lowest cost products out there, the extra price tag though the usage of the natural fibres may not be a delightful factor. It can be argued that there may not really be any interest to pay for the extra price for more environmentally friendly products although, nowadays, the sustainable production is being hyped a lot. On the other hand, if the increase in the unit price is not that much compared to the base price tag, the sustainable CRU could be used as a business advantage over other competitors. Depending on the final customer of the product, they may have certain requirements about the sustainability of that product. For example, certain cities may promote sustainability in their decision making which could make sustainable products worth the extra price tag and be chosen over their competitors. Also, if more legislations or regulations would be set that determine raw materials of the products to be recyclable or bio-based, this would promote the usage of the NFRPs. The second factor, light weight, is not the most important factor in this application, although there might be a need for lightness as there are lots of other parts also added to the smart light poles increasing the pole sway. In moving objects, like cars, NFRPs are excellent materials as they are lightweight, relatively cheap, and provide required mechanical properties.

If thermoset matrix, like epoxy, is used in the product, the sustainability of the product is reduced significantly. It can't be recycled efficiently, but compared to GFRPs, it can be incinerated without the formation of ashes. From the sustainability point of view, steel products could be scrapped and re-melted to new products which would make them more sustainable solution (yet energy intensive). To create more sustainable products from NFRPs, epoxy must be changed to thermoplastic polymer for example, preferably to a biodegradable one. In the case, where we would have 100% biodegradable materials, we would be in an excellent situation. Through bio-based materials that can be biodegraded to form nutrients than can be used as fertilizers for plants, or gases and energy, we would be close to a cradle-to-cradle type of system.

To achieve this type of situation, lots of research should be done in the field of NFRPs. As the natural fibres are water sensible and have poor adherence with the traditional polymers, certain treatments must be done to the fibres or certain additives must be added to the system. Also, their long-term durability in atmospheric conditions is problematic. These problems can be somewhat avoided with the proper treatments, but the traditional treatment methods decrease the environmental sustainability of the products and can be very expensive. For this reason, it is important to study and research treatment methods which would be based on natural methods. Another factor would be the possible biodeterioration of the natural fibres. To prevent it, one method would be to add biocides to the system which would decrease or even stop the activity of the micro-organisms. However, traditional biocides can be harmful to the environment thus decreasing the sustainability of the product. One of the most severe problem in the civil engineering applications would still be the fire retardancy of the NFRPs due to the cellulosic fibres and polymeric matrix. The products should be treated by adding lots of fire retardants or adding fire retardant coating on top of the CRU which on the other hand would decrease the mechanical properties at some rate.

The current reference shape (geometry) of the CRU would be hard to achieve with continuous fibre reinforced composites without major investments to the processing. A simpler or modified design should be used with continuous fibre reinforced polymers so that it could be manufactured with traditional RTM equipment or by fast thermoforming. Although the current shape could be achieved with some methods, like bladder inflation, the manufacturing rate would not reach the desired (anticipated) rate. Another option would be to use short fibre reinforced thermoplastics to achieve a more complicated shape but the mechanical properties would be reduced remarkably.

5.2 Biodegradation of the materials

For the soil burial testing and analysis for the candidate materials, especially the effect of a rosin addition was studied and how it would affect the bacterial activity. For the analysis, ultimate tensile strength, flexural interlaminar strength and ultimate extension at breakage were used as measurands. In the reference state, rosin was observed to lower the mechanical properties of both pure polymeric materials and composite specimens. In Mater-Bi® specimens, the addition of rosin to material caused major decrease in the ultimate extension. Moreover, the adding of rosin to the composites made materials more porous as seen in Figure 4.2. This could partly explain the drop in the mechanical (ultimate) properties.

The tensile strength values of pure PLA specimens, both with and without rosin, only dropped slightly due to the soil burial testing and rosin did not seem to have any effect on the degradation process. For Mater-Bi®, major changes could be observed visually and mechanically due to the soil burial. It was noted from visual observations that specimens containing rosin were less stained and the only stains that could be spotted were small black areas here and there. The ultimate tensile strength was observed to increase slightly for both Mater-Bi® samples but the ultimate extension was reduced due to the soil burial aging (increased brittleness).

As mentioned in the literature survey part about biodegradation, the degradation rate of PLA is very slow in atmospheric conditions and it would require a higher temperature for a faster degradation rate. During the two-week soil burial testing, there might have been slight hydrolysis as the tensile strength was decreased slightly for both material series. Due to the drop in the tensile strength being the same for both of the series, the rosin addition did not seem to have any clear effect on the degradation rate, which would in turn suggest that the bacterial activity did not have any effect on the degradation rate of PLA. For Mater-Bi®, rosin in the material blend seemed to have a positive effect on the resistance against bacterial activity as the change in the tensile strength was not that high and there were less stains in the material due to the soil burial. Although the drop in the extension is much higher for pure Mater-Bi®, any clear conclusions is hard to draw between the pure specimens and the specimens containing rosin due to the much lower extension for the specimens containing rosin in the reference state.

For the composite samples, the soil burial testing led to a negative impact on the tensile and interlaminar strength of each of the series. However, between the specimens, the drop in the tensile ultimate strength was different. The highest percentual decrease was observed for FPR. The second highest was observed for FE, then RtFPR, and finally the FP series. The specimens containing rosin had overwhelmingly highest drop in their interlaminar strength. After these, the FP is next, and only minor drop was observed for the FE series. The more there was rosin in the system, the more porous the composite was, which was observed to worsen the mechanical properties.

FE specimens seemed to have the second highest drop in the tensile ultimate strength. Referring to the literature survey, cured epoxy is not that susceptible to biodegradation. Although the flax fibres should go through certain amount of biodegradation despite the non-biodegradable matrix, the amount of degradation is remarkably high. In this case, the high degradation may be caused by water uptake of cured epoxy. Water molecules in the matrix cause swelling and plasticization which then worsens the mechanical properties, and, can affect the interface between the fibres and the matrix. (Ramirez et al. 2008) The reduction in the mechanical properties then may partly be caused by the degradation of the epoxy, but also the moisture in the epoxy may increase the degradation rate of the cellulosic fibres. The pure PLA system was not observed to degrade significantly during the testing, which would suggest that, in the FP composites, the degradation is mostly caused by the degradation of the fibres, whereas in the FE composites the degradation may also be caused by the degradation of the epoxy due to the moisture. However, the decrease in the interlaminar strength was lowest in the FE specimens.

The highest degradation in general was observed for the FPR specimens and the third highest rate was observed for the RtFPR specimens. One notable factor in these series was the high porosity of the specimens. During the soil burial, the moisture caused different layers to detach from each other. This could be seen for RtFPR specimens as the layers could be in some cases separated from each other with bare hands. Although RtFPR specimens were more porous than basic FPR samples, the drop in the tensile strength was lower. According to these observations, the rosin in the fibres prevented microbial activity as the more porous structure should increase the microbial activity and the degradation rate. When rosin is compounded with PLA, the antibacterial properties of rosin may not be so effective as the highest biodegradation happens in the natural fibres and not in the matrix.

The moisture in the compost seems to have major effect on the interface between the natural fibres and PLA. For all samples containing PLA as matrix, the colour of the specimen changed. This could be caused by micro-cracking in the materials, as water causes swelling in the fibres and when water is dried off, it might leave micro-cracks to the interface. This could explain the higher drop in the interlaminar shear strength of the PLA composites compared to the FE composites. In FE specimens, there was only minor change in the colour probably due to the better wetting of the fibres during the fabrication of the composites.

5.3 Future research

In future, there are certain factors that could be done differently regarding the current process of the thesis and, also, there are other topics that could be studied regarding NFRPs. One of the things that could be done differently regarding the current work is the usage of rosin in the composites, especially during the fibre treatment process. In the work, the fibre treatment was done using a 10 m-% rosin (and alcohol) solution. In future, the amount of rosin should be lowered to only a few percent. Also, pure epoxy specimens should also be buried to soil to see the possible effect of the moisture (in that soil) to the deterioration of the epoxy. This information could be then used to analyse the results of the soil burial much more efficiently.

In this thesis, especially the possible biodeterioration was studied. In future, if the study of the natural fibres is continued, suggested future topics are presented in Figure 5.1. In Figure 5.1, I have listed three fields where the natural fibres should be in-depth studied. The first one, Fire Engineering, includes the study on the fire retardancy properties of NFRPs. The second one, fibre treatments, include the study on the fibre treatment methods to improve the adhesion between fibres and the matrix. The third one, LCA, should be conducted on natural fibres as reinforcing elements to study the sustainability of the product made from NFRPs.

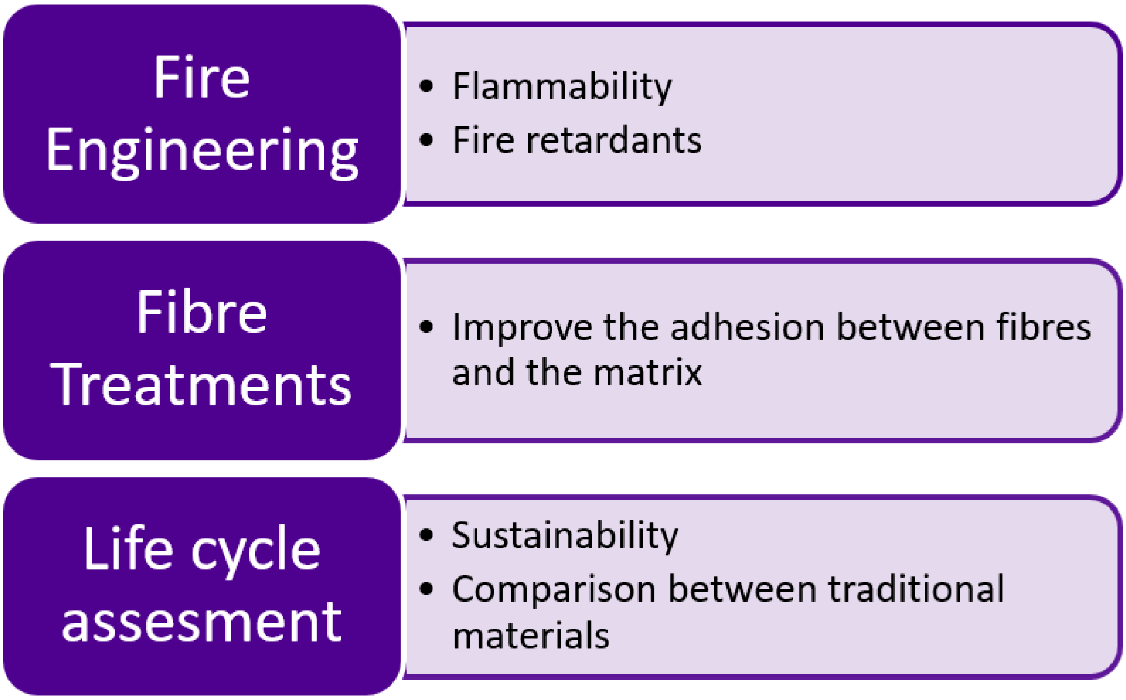

Figure 5.1: *Possible trends for the future research in the field of natural fibre reinforced composites based on the results and analysis of this thesis.*

The first topic, fire engineering, can be very critical when the material is used in a load-carrying application. As the load-carrying applications are often structural ones, the failure of the material in these situations can cause severe damage through the collapse of the system or the possibility of spreading the fire in the system quicker. The second topic, Fibre Treatments, would cover treatments done to fibres to improve the adhesion between the fibres and the matrix. Especially, socially and environmentally sustainable methods should be studied as a way to improve the adhesion between the fibres and the matrix as the adhesion has a major impact on the laminate's mechanical properties, moisture absorption, and biodeterioration. The last topic, Life cycle assessment, should be done to NFRPs in certain application to provide information for proving the sustainability of the usage of natural fibres and to prevent any possible 'green washing'. Currently, the research and development of bio-based and biodegradable materials, such as bio-based and biodegradable epoxies, is going on around the world. In future, these new materials could also be the way to go in the field of biocomposites.

6. SUMMARY

The thesis was divided to main topics, Camouflage Radome Unit and the soil burial, which are covered separately starting by the CRU. The fabrication of the prototype of the sustainable CRU was done by the data received from the Luxturrim5G project and the prototype was showcased during the Luxturrim5Gs project finale, The Impact Day. The aim was to show the possibilities in the usage of alternative composite materials. Through the publicity like this one, the usage of the NFRPs in different products may increase as more people and industries are aware of this kind of alternative composite materials which could be used in different load-carrying multifunctional applications. The natural fibres themselves pose certain problems in their long-term durability in atmospheric conditions. However, these problems can be somewhat avoided with proper treatments. These treatments should prevent the moisture absorption to the fibres which may cause swelling, and the water may cause fibres to be susceptible to microbial activity. Also, treatments should be done to fibres or to the polymer to enhance the adhesion between the hydrophilic fibres and the hydrophobic polymer. From mechanical point of view, NFRPs could be used without any major problems in the CRU. As the loads are not that high, even less than a millimetre-thick wall could handle the loads easily with the new shape based on a baseplate (according to the simplified calculations made during the thesis), and only problem in this application being the long-term durability of the composite.

During the soil burial, degradation can be seen in each of the composite specimens and in the Mater-Bi® specimens. In these conditions, the PLA was not observed to biodegrade substantially. However, flax fibres were observed to degrade no matter what the matrix material was. Rosin was observed to affect negatively the processing of the composites and the mechanical properties of the pure polymers and the composites. However, it was observed that the rosin had positive effect on the biodeterioration, and in future, more research should be done with the rosin treated fibres except the amount of rosin in the fibres should be changed to only a few percent.

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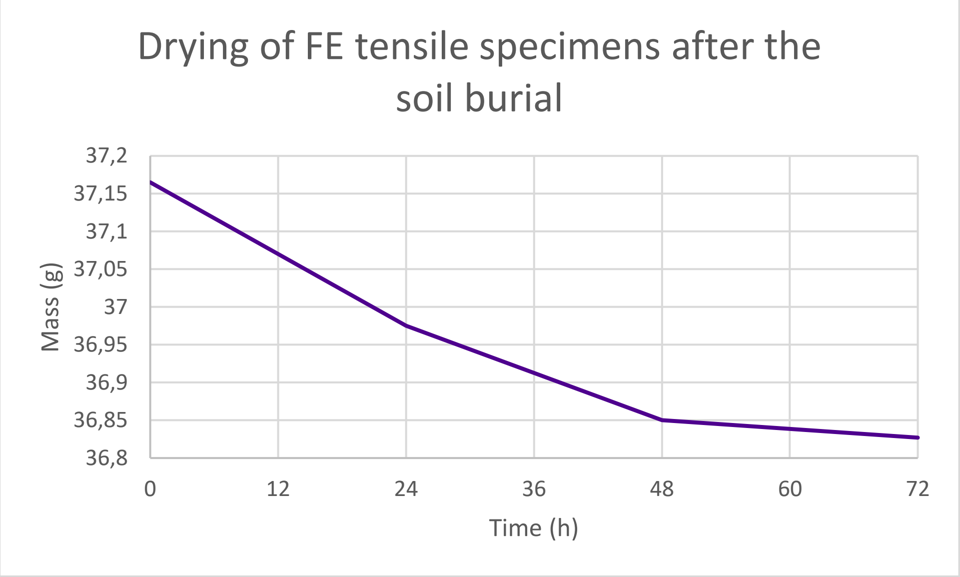
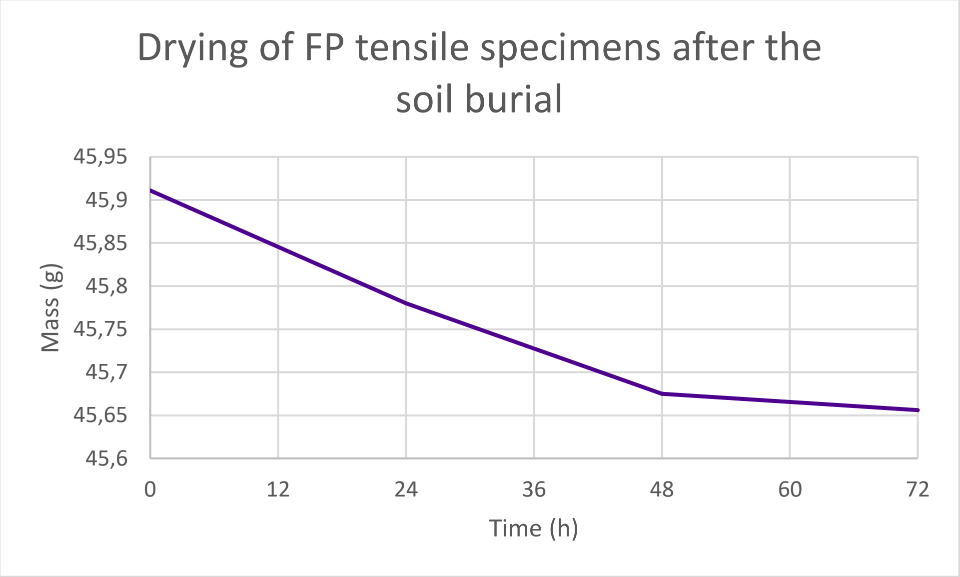
ANNEX 1: THE MAIN REQUIREMENTS FOR THE CAMOUFLAGE RADOME UNIT PER SPECIFICATION

Factor	**Requirements**
Position of the unit	Camouflage Radome Unit ca not be attached above the lamps due to the high absorbance of the lamps and disturbance of the signal caused by the surface of the lamps.
Signal windows	Fixed to the point in certain directions and comprising four windows (four sectors) per lamp. The radiation lobe for each signal is ±45 degrees to cover the whole 360 degrees. Horizontally 60 degrees are covered from the ground level for good connection towards buildings and streets.
Attachment	Drilled holes in the CRU are used to attach the unit to the lamp pole and the cover to the hull. The attachment must not increase the compliance of the system so that the sway at devices would increase.
Modularity	CRU has to be modular so that the access to the parts is as simple as possible and standardized in terms of connections and cable input-output ports (power and data).
Volume of the unit	The volume of the CRU has to be large enough for all the necessary devices and cabling and enough area for the heat sinks and thermal barrier distances. the size of the CRU is not fixed (predetermined) but a smaller structure could be camouflaged better with the overall lamp post.
Mechanical requirements	The total weight of the components inside the CRU is about 5.0 kg. The operation temperature inside the CRU housing should be kept between 0 and 40 degrees Celsius. The temperature range must not affect the load carrying capability of the unit (e.g. glass transition or heat deflection limit for polymers must be higher).

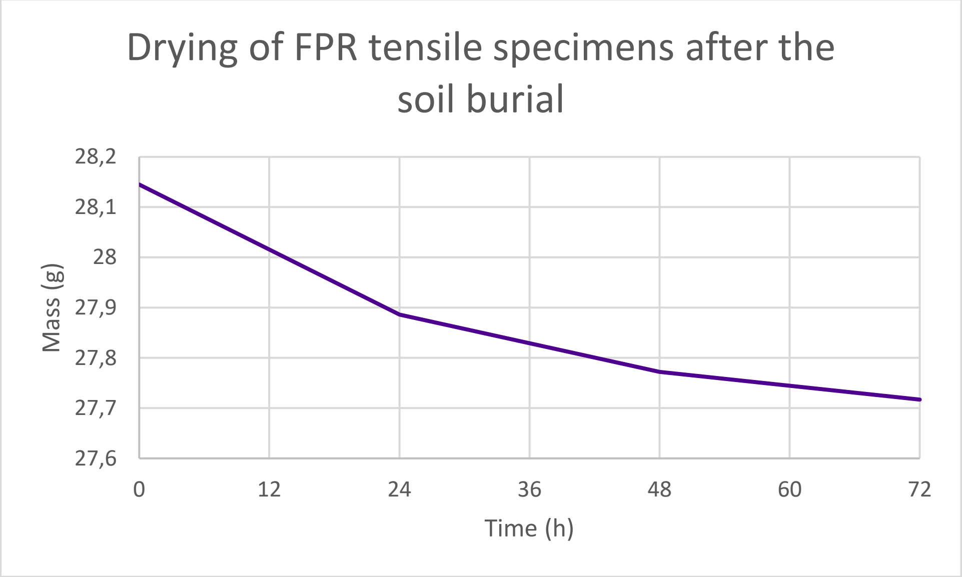
ANNEX 2: THE DRYING DATA FOR FLAX EPOXY TENSILE SPECIMENS



ANNEX 3: THE DRYING DATA FOR FLAX PLA TENSILE SPECIMENS



ANNEX 4: THE DRYING DATA FOR FLAX PLA + ROSIN TENSILE SPECIMENS



ANNEX 5: THE DRYING DATA FOR ROSIN TREATED FLAX PLA + ROSIN TENSILE SPECIMENS