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Sustainable fibre materials for replacing plastics in 3D-forming applications

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Abstract

Plastic is a very broad family of materials that may provide a wide array of mechanical properties depending on the plastic or production method in question. This is why many industries have chosen plastic as their material of choice for the production of anything from plastic bags to underground piping. There is, however, a prominent issue concerning the heavy environmental impact of plastic. This is both due to the processing of crude oil and lack of biodegradability which in turn impact nature if the plastic is not processed in a proper waste disposal stream. In some applications such as food trays and food containers, moulded pulp has been used as a sustainable and more environmentally friendly alternative to conventional plastic products for quite some time already. The applications of moulded pulp do however not expand very far at the current time and that is why this thesis seeks to assess the properties of moulded pulp samples to evaluate the potential for replacing plastics in new mass production settings. Moulded pulp does not only have an environmental motivation but it also shows promise of being more economically viable in certain settings and the paper industry is always looking for new applications for them to use the pulp they are manufacturing. There are, of course, many applications where it is not viable or desired to replace plastic at all but the motivation is strong for replacing plastics in applications where it is possible. This is especially due to a general shift in society towards environmental awareness which is a major driving force in changing industries. If people want change, the industry will in most cases have to adapt in order to remain profitable.

For this thesis, the focus was on assessing the mechanical properties of moulded pulp and how the fibres and the material behave when thermoformed in a fashion similar to that of thermoformed plastic. This was primarily done through the means of tensile testing of paper sheets and moulded specimens. In addition to this, the effect of reinforcement from microfibrillated cellulose (MFC) was also explored.

In the end, the results of all the tests were very promising and the strength of the moulded samples was on par with quite a few of the most common plastics used today. The thermoforming, which is one of the most promising methods for moulding paper, also showed its capabilities of producing excellent samples and the future development of thermomoulding processes could result in highly efficient moulding methods down the line. This thesis did not have any specific applications in mind when the research was conducted but when considering the results it is clear that with further research, this material could possibly be used for a wide array of applications and it could possibly be used in a laminate construction to greatly enhance the strength and make the material viable for even structural components.

Sammendrag

Plastikk er et begrep som omfatter et stort spekter av materialer som kan tilby vidt forskjellige mekaniske egenskaper avhengig av type plastikk eller produksjonsmetode. Dette er grunnen til at mange industrier har valgt plastikk som materiale til alt fra bæreposer til avløpsrør. Dessverre har plastikk en del negative effekter på miljøet. Dette er både på grunn av prosessene tilknyttet opphenting av råolje og den biologiske nedbrytbarheten hos plastmaterialene. Det eksisterer allerede papir-baserte produkter, slik som innpakning eller beholdere til mat, men støpte papirprodukter har likevel et relativt begrenset bruksområde per dags dato. Det er derfor dette prosjektet ønsker å undersøke støpt papir sitt potensiale for å erstatte plastikk i nye former for masseproduksjon. Støpte papirprodukter har ikke bare en miljømotivasjon men denne type produkter har også vist potensiale til å være gunstige økonomisk og papirindustrien er alltid på jakt etter nye bruksområder for all papirmassen som produseres. Det er selvfølgelig ikke alle områder hvor det er mulig eller ønskelig å erstatte plastikk men motivasjonen er sterk for å erstatte plastikk der det er mulig. Dette er spesielt på grunn av et større fokus på miljøet i samfunnet. Samfunnet har stor påvirkningskraft på industrien så dersom det er et ønske om forandring så må industrien tilpasse seg for å forbli lønnsomme.

I denne oppgaven var fokuset på å evaluere de mekaniske egenskapene til støpt papirmasse og hvordan et slikt materiale oppfører seg når det blir termoformet på en lignende måte som termoformet plastikk. Dette ble primært gjort gjennom strekktesting av både pressede papirark og støpte testeksemplar. I tillegg ble effekten av forsterkninger med mikrofibrillert cellulose (MFC) utforsket.

Resultatene viste seg å være veldig lovende og styrken til de støpte prøvene var på høyde med flere av de vanlige plast-typene som brukes i dag. Termoformingen, som er en av de mest lovende måtene å støpe papirmasse på, viste også at det er en produksjonsmetode som er i stand til å produsere prøver i høy kvalitet. Med videre forskning kan disse termoformings-prosessene potensielt bli veldig effektive. Denne oppgaven hadde ingen spesifikke produkter som et krav når forsøkene ble gjort men resultatene tilsier at med videre forskning så kan et materiale som dette potensielt bli brukt i et vidt spekter av produkter. Ved å utnytte en laminatkonstruksjon kan det også være potensiale for å øke styrken betraktelig slik at det eventuelt kan benyttes i strukturelle komponenter.

Acknowledgements

I would like to thank my supervisor Martin Steinert for introducing me to this project and for welcoming me as a part of TrollLabs. I would also like to thank my co-supervisor Gary Chinga Carrasco for helping me navigate the world of wood pulp fibres and staking out the course of this thesis, and my co-supervisor Jørgen Blindheim for helping me with developing some of the practical test setups. Special thanks go to Norske Skog, represented by Dag Molteberg, for supplying the project with the necessary raw materials. Moreover, I would like to thank all the lab technicians and workshop engineers at PFI and MTP for assisting me and answering my questions through the course of my experiments.

Last, but not least, I would like to thank all my friends, family and my girlfriend for being there for me not only through the course of this thesis but during the last six years of studies leading up to this. I sincerely thank you all!

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Abbreviations

TMP	=	Thermo-mechanical pulp
MFC	=	Microfribrilliated cellulose
DIP	=	De-inked pulp
CTMP	=	Chemi-thermo-mechanical pulp
PFI	=	Paper and Fibre Research institute
MTP	=	Department of Mechanical and Industrial Engineering
DTU	=	Technical University of Denmark
MDF	=	Medium Density Fiberboard
CNC	=	Computer Numerical Control
CAD	=	Computer-Aided Design
CAM	=	Computer-Aided Manufacturing
CAE	=	Computer-Aided Engineering
PID	=	Proportional-Integral-Derivative
SSR	=	Solid State Relay
ASTM	=	American Society for Testing and Materials
ISO	=	International Organisation for Standardisation
PLA	=	Polylactic Acid
WFRC	=	Wood-fibre-reinforced Composite
WPC	=	Wood-Plastic Composite
SEM	=	Scanning Electron Microscope
L&W	=	Lorentzen and Wettre
UTS	=	Ultimate Tensile Strength
PP	=	Polypropylene
PE	=	Polyethylene
HMDS	=	Hexamethyldisilazane
PHA	=	Polyhydroxyalkanoate
PPS	=	Parker Print Surf
EDM	=	Electrical Discharge Machining

Chapter 1

Introduction

1.1 Background

This thesis is a continuation of ”Zero Footprint Material Production”, a 15 ECTS pre-master project conducted at NTNU, Trondheim over the course of the fall of 2016. It was conducted by Eirik Ulsaker Jacobsen under the supervision of Martin Steinert and the co-supervision of Jørgen Blindheim. The pre-master project can be found in its entirety in Appendix F and will be referred to as Jacobsen (2016) in this thesis. That project came to be as a way to explore the possibilities of creating a sustainable material that could potentially replace certain plastics in mass production. This was motivated both by the environmental aspect of replacing plastics and a desire from the paper industry to find new applications for the large amounts of pulp being produced. The exponential growth of technology in recent years has led to certain qualities of paper seeing a noticeable decrease in the demand and thus forced the paper industry to find new applications where paper is needed. That is why this thesis intends to further explore if sustainable fibre materials have the properties needed to make their way into new industrial production settings.

N.B. Initially, this project wanted to explore the possibility of using recycled fibres as well. The recycled pulp was obtained from a local supplier instead of Norske Skog and Section 4.1 includes some tensile test results on paper sheets made from this recycled pulp. Unfortunately, the supplier was unable to supply any more pulp than what was used for the initial testing so no further testing was done with recycled pulp and this thesis focuses solely on the thermomechanical pulp (TMP) and Kraft pulp because of this. The results of the Recycled tensile tests are however still included as it provides some interesting information when compared to the other results from that test.

1.2 Problem description

This master thesis aims to evaluate if wood pulp fibres have the necessary mechanical properties to be used for 3D-forming applications in a mass-production setting with the goal of being able to replace some plastics in the future. The two pulp types that will be explored are thermomechanical pulp (TMP) and Kraft pulp. This includes tensile testing and an assessment of the moulding process itself. In addition, this thesis will look into whether microfibrillated cellulose

can be added to increase the strength without compromising the dewatering of the pulp during 3D-forming. This thesis research was conducted in partnership with Scandinavian Business Seating with raw material supplied by Norske Skog.

1.3 Objectives and scope

This thesis has the overarching goal of assessing if moulded pulp has the potential to be used as a substitute for some plastic products in a non-specified mass production setting. Specifically, the thesis will focus on evaluating the mechanical properties of moulded pulp and how the fibres behave when thermoformed in a fashion similar to that of thermoformed plastic. The concrete objectives for this thesis are the following:

- Produce dewatered and hot-pressed model sheets for assessment of basic mechanical properties of TMP and kraft pulp both with and without MFC as reinforcement.
- Produce standardised tensile test samples for mechanical testing and for comparison to plastic samples.
- Assess the mechanical properties of the moulded samples (tensile strength, elongation, modulus etc.).
- Develop and assesst a process for thermoforming pulp.

1.4 Initial research and testing

Thermoforming has been established as one of the best ways of moulding pulp [1] and Jacobsen (2016), the precursor of this thesis, focused on experimenting with the thermoforming of pulp to see if it was possible to make tensile test samples that would resemble plastics both in how they were moulded and how they were tested. The goal of this was to assess the mechanical properties on a platform more close to what is known in the world of plastics. Thermoformed mould-dried pulp is known for creating products with the closest resemblance to thermomoulded plastics, with higher density and smoother surfaces. The different types of moulded pulp are covered in Section 2.4.1. Through the initial attempts to mould TMP fibres some issues with the density of the fibres were discovered which essentially made it impossible to dry the fibres properly due to the water being unable to boil off and dissipate. This was solved by developing a filtering system that would drain off some of the fines in order to allow for a less dense structure that would accommodate the dissipation of water. This filtering system utilises the same concept as most processes for making paper or pulp products where a low-concentration pulp is filtered down to the desired shape. This also aids in creating a homogeneous and interlocked structure in the fibres. This filtering system has since then been improved upon for a more efficient process which is covered in Section 3.3. The concentration of the pulp in these types of filtering processes varies but in general it is 1 - 4 wt% for most pulp-moulding applications [1] and 0.2 - 0.5 wt% for paper sheet making aimed at physical testing [2]. For the mould-filtering process used in this thesis, it was desirable with a concentration within the limits for the paper sheets as this ensures that the fibres are able to filter properly. Jacobsen (2016) also discovered some issues with the mould that halted the progress of that project. These issues and solutions to them are discussed further in Chapter 3.1.

Chapter 2

Theory and literature review

2.1 Plastics and sustainability

Plastic has a long history and it was already in 1907 that the world was introduced to the first fully synthetic plastic which was created by Leo Baekeland and appropriately named Bakelite [3]. Plastics have since then become a big part of modern society due to their potential for excellent mechanical properties in combination with low weight. There is, however, a lot of controversy surrounding the environmental impacts of plastic. In 2007 there were 260 million tonnes of plastic produced globally with 24.6 million tonnes of post-consumer waste in Europe alone. Out of this, half was deposited in landfills while $30\,\%$ was used for energy recovery (the process of creating energy from burning waste) and only $20\,\%$ was recycled [4]. However, based on the 2016 report from the Association of European Plastic Manufacturers [5] there has been a positive change in European plastic waste management. In 2014 there were 25.8 million tonnes of plastic waste in the official European waste streams and the European Plastic Manufacturers claim that they had a recovery rate of $29.7\,\%$ recycling, $39.5\,\%$ energy-recovery and only $30.8\,\%$ landfill. This is a positive development but these numbers are still only based on the official waste streams in Europe, a region that is actively pursuing better recycling and waste handling solutions. What about the plastic that is dumped straight into nature or the waste disposal rates in developing countries? In addition to this, there is a difference between thermoplastic and thermoset plastics. The latter can not be softened by heat which makes recycling a lot more challenging than for the thermoplastics which can be reheated and remoulded to allow for easier recycling and reuse. Moreover, there is also the issue of most plastics being oil-based.

Oil is a finite resource that is the cause of a lot of controversies. Not only is the reintroduction of oil into our eco-system believed to cause environmental disturbances but the retrieval and refining of oil raise many ethical and environmental questions, especially when considering certain methods like hydraulic fracturing or oil-sand-retrieval. One solution to these issues is a shift towards bioplastics. Bioplastics, like polyhydroxyalkanoate (PHA) or polylactic acid (PLA), are plastics that may provide mechanical properties similar to oil-plastics but they are made from natural and renewable sources such as starch or soy [6]. Some of these bioplastics even claim to be biodegradable or compostable. In addition, there are also bioplastics that are essentially bio-based versions of their oil-derived counterparts. Examples of these are BioPE and BioPP which are chemically equal to polyethene (PE) and polypropylene (PP), which are both common plastics. BioPE and BioPP are thus biobased and durable but not biodegradable.

Bioplastics are however not entirely without flaws either and chapter 2.6 covers some of the issues surrounding biodegradability and compostability.

Wood fibres and pulp, on the other hand, has shown a potential for creating environmentally friendly materials. Wood is a renewable resource and it can be used to create materials with good properties in terms of biodegradability and compostability. The idea behind this thesis is that certain plastics used in mass-production perhaps could be replaced by a biodegradable and sustainable material that hopefully would have positive long-term effects on the environment. There are some issues with the energy-consumption of pulp production and especially with TMP-production [7] but this is an area where pulp-manufacturers are trying to improve. In addition, with the current shift in society towards renewable energy, these issues of energy consumption should have less of an impact in the future.

2.2 Introduction to fibres and pulp

Paper has been an integral part of society for centuries and archaeological paper fragments have been dated as far back as to the 2nd century BC [8]. The process of making paper from pulp is believed to have been conceived as early as the 2nd century AD [8]. In modern society the applications of pulp and wood fibres have expanded far beyond the scope of just plain paper and new technology allows for different forms of treatment and production processes. Modern pulping is done by chemically or mechanically separating cellulose fibres from wood, crops or waste paper with multiple variations of these two main processes. Three very common methods are thermo-mechanical pulping (TMP), chemi-thermo-mechanical pulping (CTMP) and chemical pulping [9]. These methods all yield pulps with different chemical and structural characteristics which may be of use for different applications, such as packaging, tissues or printing paper. There is also a process that involves pure mechanical pulping but it is not as common since it puts a lot of stress on the fibres which could lead to them being partially destroyed in the process. It does, however, allow for the pulping of harder wood types.

In terms of production numbers Table 2.1 shows global pulp production by category in the year 2000. It is quite clear that the chemical kraft process (also known as the sulphate process) is the most commonly used method for pulping. In the second place, we find the mechanical processes with TMP being the prominent process [10]. The TMP process uses high-temperature steam to soften the fibres before the mechanical refining which helps create a stronger pulp than a purely mechanical refinement would be able to. Unfortunately, this comes at the cost of an increased energy consumption during the production process. Norske Skog has the capabilities to produce TMP and de-inked pulp (commonly referred to as recycled pulp) but they order the kraft pulp they need from other facilities [Personal correspondence, 05.12.16]. They are also able to produce microfibrillated cellulose (MFC) from kraft pulp and are currently experimenting with producing MFC from TMP. MFC and its impact on fibrous materials will be covered in Section 2.3

2.3 MFC

MFC is a form of nanocellulose extracted from wood fibre pulp which was first defined by Turbak et. al. [11] and Herrick et al. [12] in 1983. MFC is composed of micro-/nanofibrils

Table 2.1: World pulp production in the year 2000 [10]

Pulping Method	**Production [M tonnes]**
Chemical	131.2
-*Kraft*	117
-*Sulfite*	7
-*Semichemical*	7.2
Mechanical	37.8
Nonwood	18
Total Virgin Fibres	187
Total Recovered Fibres	147
Total Pulp	**334**

which are tiny fibres in the structure of the cellulose that can be extracted from the pulp. Figure 2.1 shows pictures from a scanning electron microscope (SEM) with some good examples of microfibrils in a fibrous structure. The use of MFC has been widely researched and multiple studies have shown that MFC has a significant potential for increasing the strength of paper [13–16]. This potential is very interesting for this project since it could potentially improve the mechanical properties of the new material. The hypothesis is currently that MFC would provide a boost to the tensile strength of the moulded samples at the cost of making dewatering more difficult. The tests conducted for this thesis aimed to find out if this trade-off is favourable or if MFC in its current form is not viable as a structural improvement for this material. MFC is, however, a very general term and there are a number of different processes which are yielding very different fibrillated materials. For this thesis, the term MFC is used as a general umbrella term to keep things consistent but Section 2.3.1 will clarify some of the terminology surrounding MFC.

2.3.1 Characterisation of MFC

In the early history of microfibrillated cellulose the term MFC was fairly sufficient to cover this new fibrillated material but as research continued, a wide variety of terms started emerging such as nanocellulose, nanofibrillated cellulose, nanofibres, nanofibrils and microfibrils [18–22]. Not only are many of these terms very similar, but they have been used without any objective definition of the terminology. Chinga-Carrasco, G. (2011) [17] attempted to define some of these terms in an objective manner and Table 2.2 shows a systematic representation of how current literature applies different terminologies. Looking at this table there is a clear difference between the biological structural terms and technological terms. For technological use, the term nano has been established to refer to the range of $1\,\mathrm{nm}$ to $100\,\mathrm{nm}$ ($0.001\,\mu\mathrm{m}$ to $0.1\,\mu\mathrm{m}$) [23]. For the biological structures, however, the term microfibril is used for fibres smaller than $0.035\,\mu\mathrm{m}$ which in technological terms would be considered nano. In the smallest size group of the biological structures, the term *elementary fibril* can be found. According to

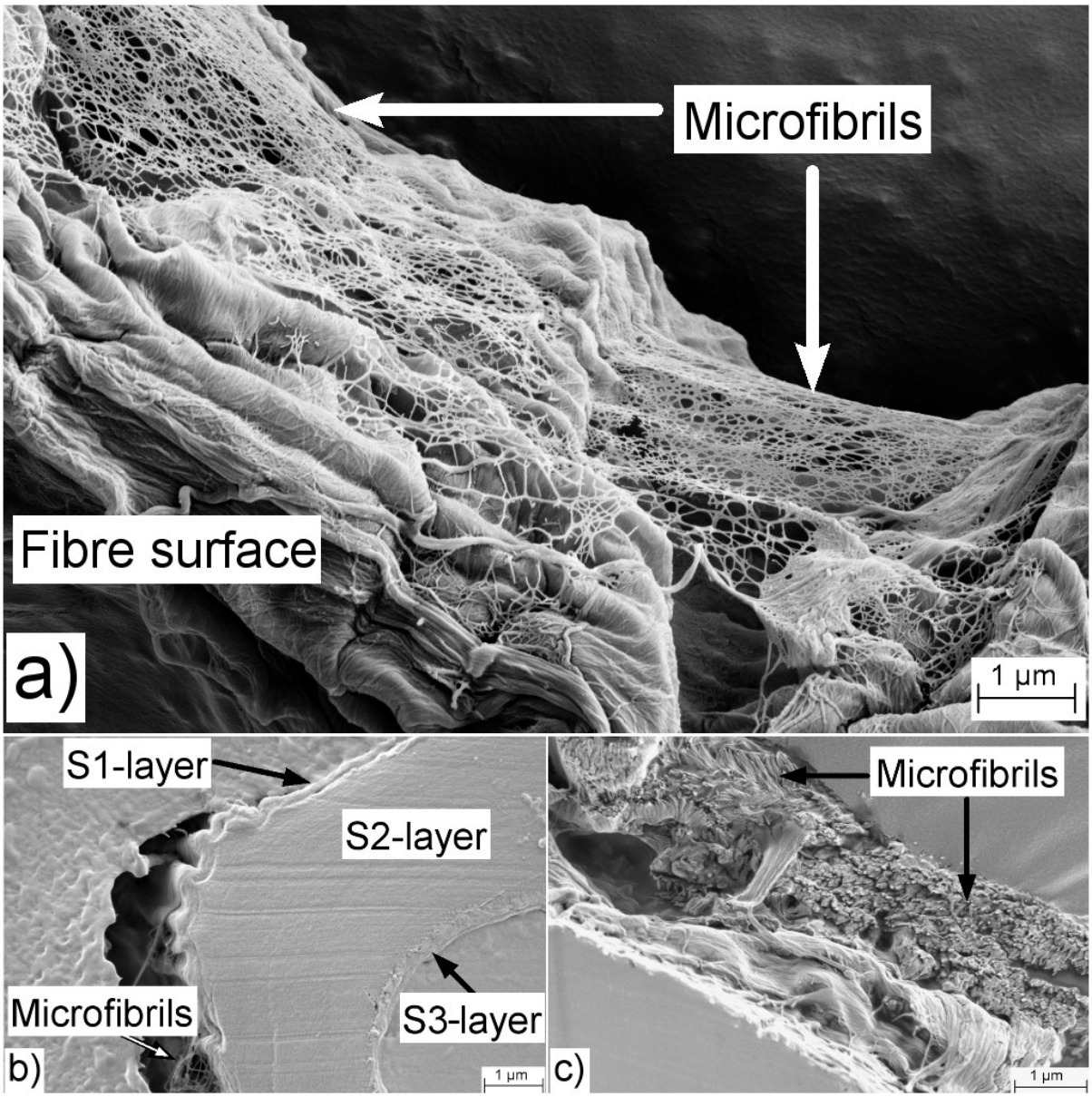


Figure 2.1: SEM-images of MFC, Figure 1 from Chinga-Carrasco, G. (2011) [17]

Table 2.2: Components of microfribrillated cellulose

Diameter [μm]	**Biological structures**	**Technological terms**
10 to 50	Tracheid	Cellulose Fibre
<1	Macrofibrils [24, 27, 28]	Fibrillar, Fines, Fibrils [29, 30]
<0.1		Nanofibril, nanofibres [18, 20, 31, 32]
<0.035	Microfibril [24, 33]	
0.0035	Elementary fibril [24–26, 33]	

This table is reprinted from Chinga-Carrasco, G. (2011) [17]

Meier [24], the elementary fibril is one of the cellulosic components of the wood fibre wall structure, together with the microfibril, macrofibril and the lamellar membrane. This element was reported to have a $3.5\,\mathrm{nm}$ diameter and was applied after the terminology of Frey-Wyssling [25]. Heyn [26] later reported that this elementary fibril was a universal structural unit of natural cellulose as it has been found to occur in several different types of cellulose. Meier [24] also reported on how microfibrils consisted of elementary fibre clusters with the size of the microfibrils always being multiples of $3.5\,\mathrm{nm}$ up to a maximum of $35\,\mathrm{nm}$. The difference between the use of the term micro in these biological structures and in the term MFC is one of the sources for the confusion around the terminology surrounding MFC. The term MFC is used for materials that may be composed of nanofibrils, fibrillar fines, fibre fragments or residual fibres. [17] The implication of this is that the term MFC does not necessarily have any connotations regarding the presence of any microfibrils, nanofibrils or other cellulose nano-scale structures. Moreover, depending on the production process, which is covered in detail in Section 2.3.2, MFC may also contain nanofibrils as its main component. In summary, there are good definitions of the terms used for this type of fibrillated cellulose but the term MFC should perhaps be taken with a grain of salt as a detailed characterisation is usually needed to really understand what type of fibrils it is referring to.

2.3.2 MFC production

The difference in how the MFC is extracted and treated is one of the major reasons why masses referred to as MFC often have completely different characteristics. The extraction process has essentially two main steps: A mechanical and/or chemical pre-treatment and a second process such as homogenisation or Masuko grinding to further fibrillate the material. Processes to disintegrate cellulose structures have been seen as early as 1950 [25, 27, 34]. In addition, Colvin and Sowden [35] reported on a homogenisation process that used beating to open the cellulose structures to expose the microfibril structures. It was then Turbak et al. in 1983 [11] who introduced a homogenisation process to fibrillate cellulose for commercial use. This homogenisation process, in combination with an initial refining process, is widely used to create the fibrillated mass that is referred to as MFC. The initial refining or pre-treatment is usually done once but the second step can be done multiple times to achieve an even higher degree of fibrillation [17]. For the homogenisation process, the pressure of the homogenisation also affects the level of fibrillation. The MFC mass used in this project was part of a batch from

Norske Skog that only had a single pass through the homogeniser at a pressure of 800 bar. [Personal correspondence 03.05.17] This means that the mass most likely consists of a mix of different size fibres and may have a relatively low degree of fibrillation. Masses with higher degrees of fibrillation have a more homogeneous mass of nanofibrils which may help raise the strength of paper but a higher degree of fibrillation also amplifies the issues of dewatering covered in Section 2.3.3. This is why the single pass mass used in this project is most likely a good fit for the project as it should provide a strength improvement without causing extensive issues with dewatering. The MFC from Norske Skog is currently being extracted from imported Kraft pulp but they are trying to develop a method for extracting MFC from the TMP they are making in their own facilities [Personal correspondence 05.12.16].

2.3.3 Dewatering

The ability to dewater or drain the pulp is a very important characteristic in paper making and pulp moulding since it directly affects the production time of a paper sheet or a moulded part. In mass-production applications it is paramount that the draining time is kept to a minimum in order to keep production times low, allowing for a more profitable process. Taipale et. al. [13] had the goal of assessing the mechanical properties and drainage times of kraft pulps that were mixed with varying amounts of MFC. They found that there were a number of parameters that affected the drainage time of the pulp mixed with MFC such as pH, salt content or type of cationic polyelectrolyte. However, it was found that an increased MFC content did not only provided significant strength improvements to paper made from the pulp but it also created a just as significant increase in the drainage time. This is the reason why MFC as an additive has seen limited use in commercial moulding processes since it increases the lead time on production considerably. However, there is a possibility that further research and optimisation of the production processes could allow MFC to provide increased strength without compromising dewatering too much.

2.4 Current state of moulded pulp

Moulded pulp products have been around for quite some time with one of the earliest known patents for a method for making pulp-products dating back to 1890 [36] and the first machine for creating such products being patented in 1903 [37]. Pulp-based products have seen an increase in popularity since then, especially in periods of increased environmental awareness in society. In recent years, the environmental demands from both customers and governmental regulations have put pulp-products on the radar of many companies looking to shrink their environmental footprint. Moulded pulp is, however, still an area that is somewhat under-researched and the current use is mostly limited to packaging and various forms of food-related products (food-trays etc.). Examples of this type of products can be seen in Figure 2.2.

2.4.1 Moulded pulp production

In its current state, moulded pulp has four main categories which are classified according to the International Moulded Fiber Association guidelines: [38]

Figure 2.2: Examples of moulded paper products

- **Type 1 - Thick Wall** Manufactured using a single mould with product wall thickness from about $5\,\mathrm{mm}$ to $10\,\mathrm{mm}$. One surface is relatively smooth, with one side rough. Primarily used for support packaging of non-fragile, heavier items. (vehicle parts; furniture, motors etc.). As well as, plant, floral and nursery pots and containers. Oven dried.
- **Type 2 - Transfer Moulded** Manufactured using one forming mould and one transfer mould with product wall thickness from about $3\,\mathrm{mm}$ to $5\,\mathrm{mm}$. Surfaces are relatively smooth on one side. Most common use is for egg cartons and trays. New designs are used for many types of electronic product packaging such as cell phones, DVD players etc. Also, used for hospital disposables, electrical appliances, office equipment, tableware and fruit and drink trays. Oven dried.
- **Type 3 - Thermoformed/Thin Wall** Manufactured using multiple heated moulds with product wall thickness of about $2\,\mathrm{mm}$ to $4\,\mathrm{mm}$. Surfaces are smooth and forms are well detailed with minimal draft angles. Products are dried in the mould and no oven curing is needed. Due to the hot mould pressing process, the walls are somewhat denser. Type 3 thermoformed fibre products closely resemble Thermoformed plastic material.
- **Type 4 - Processed** This type refers to moulded fibre products that require some type of secondary or special treatment other than simply being moulded and cured.

In addition to these four general types of products, there are also a number of different processes available within these four definitions. Currently, the two most common types in commercial use are stamping and vacuum assisted forming (which is a type of thermoforming) [39]. In addition to this there are also other emerging processes such as deep-drawing, hydroforming and hot pressing.

The first setup created for moulding pulp was developed by Keyes [40] in 1904 by utilising a two-part mould design. One part of the mould is dipped in a suspension of fibres before the second half is introduced in order to compress the fibres. Excess water is drained out by a vacuum. Once the product has been properly formed, the product is then moved to a separate oven to dry. This is essentially how vacuum forming is still done today. This process is however quite energy-consuming with the drying step being responsible for a large portion of both the time and energy consumed. One solution to this problem is an in-mould drying process called impulse drying. This process was introduced in the early 1980s by Wahren [41] and quickly caught the attention of the paper industry. This method works by combining the mechanical compression of the fibres with intense heat in the ranges of $200\,^{\circ}\mathrm{C}$ to $400\,^{\circ}\mathrm{C}$ [42]. This form of drying could lead to both faster and more energy efficient moulding but there are unfortunately some runnability issues that three decades of research has not yet been able to solve completely. These issues are mainly related to web delamination and paper adhesion to the hot surface [42]. However, there is still research being conducted in order to make impulse drying a viable option.

2.4.2 Mechanical Properties

Due to the biological nature of paper, the mechanical properties are heavily influenced by factors such as fibre length, humidity, temperature and the parameters of the moulding process. High humidity is especially troublesome since moulded pulp will sustain great losses in strength when humidity rises. This is due to hydrogen bonds in the inter-fibre bonds of the

moulded pulp [43]. Because of this variation in properties, the deriving of precise engineering equations is challenging and most experiments or design parameters are usually tailored to specific applications. Ji et al. [44] attempted to study the mechanical behaviours of moulded pulp with the help of a scanning electron microscope. The results from these experiments showed that the moulded pulp material was not only elastic-plastic but also had viscous characteristics. The visco-elastic nature of moulded pulp is known to cause a defect known as post-forming instability, which is covered in Section 2.4.3. There were also signs of irregular characteristics due to voids, impurities and random fibre fraction orientations. These irregularities could be controlled completely in future moulding processes, which would provide more homogeneous products with fewer variations. If parameters such as fibre orientation could be controlled completely, the mechanical properties could be more generalised and the properties could even be controlled by adjusting the fibre orientations in the same way as in materials such as fibreglass or carbon fibre composites.

2.4.3 Defects in moulded pulp

Moulding pulp is by no means an easy task. The varied and biological nature of the fibres leads to a whole new range of issues with moulding. There are a number of sources of error that could affect the moulding process and it is important to be able to distinguish if the result is satisfactory. Having the ability to do this characterisation is important to assess material properties and the effect of specific parameters of the moulding process. Looking for defects in the sample is an excellent way to identify and diagnose issues with the material or the process. Some of the most common defects and their implications on moulding are summarised below.

Post-forming instability

Due to the visco-elastic-plastic nature of paper, moulded paper may experience post-forming deformations as a result of elastic recovery and excessive or improper drying. These issues are most prominent in certain moulding processes but may appear in any moulding process since it is in part caused by an inherent property of paper. Because of this, deflection and spring back of shapes are very common defects and it is usually caused by moisture being absorbed after the paper is released from the mould, which leads to the release of mechanical stress, causing hygro-expansive deformation [39]. Fortunately, there are ways to alleviate this, either by utilising fibres with more plastic behaviour or ensuring that the drying process is properly tuned to prevent post-forming deformations. Adjustment of pre-moulding dryness and post-moulding conditioning have proven to be a successful forms of instability-mitigation [45]. For the experiments in this thesis, the presence of these types of deformation in the moulded samples would suggest that some of the moulding parameters would have to be looked at and reassessed.

Cracks

Cracks are not only some of the most obvious defects in moulded samples but they also have a tremendous impact on the functionality of the sample. Cracks are caused by stress in a certain area rising above the strength of the fibres, leading to a mechanical failure. In most cases, the cracks are the result of tensile stress but depending on the mould, there might be more

complicated stress states causing cracks to appear [1]. Cracks were however not expected to be an issue for the moulding of the samples in this particular thesis project since the samples would be under a constant pressure with no tensile force applied. However, this means that any sign of cracks in the samples should be taken as a sign of a major failure somewhere in the process.

Wrinkles

Wrinkling is a defect commonly seen in many of the current paper moulding applications (packaging material etc.) and is caused mainly by compressive forces acting transversely on the paper [46–48]. Wrinkling creates an uneven surface on the material which has quite an impact on the visual quality of the moulded material. Depending on the application, this form of visual defect could render the product useless. In sliding blank forming, adjusting the blank holder force has proven to help preventing wrinkling with the higher force resulting in a lower probability of wrinkles and buckling [45]. This does, however, come at the cost of an increased probability of cracks due to the increased force. Wrinkles were, like cracks, not expected to be an issue for this project due to the shape of the samples and the way the force was applied. However, in general it is still a warning sign if it were to occur.

Water pockets and discoloration

Due to the density of certain fibre types, pockets may appear in the sample when water and vapour are unable to escape. This results in spots or clearly defined areas on the sample surface which will result in a distinctly different surface finish. This was already seen in the initial testing of Jacobsen (2016) and is usually a sign that the moulding parameters need to be adjusted. This is most likely the defect that will be most prominent in this project, together with post-forming instability. This is especially due to the high temperatures the samples are subjected to during moulding. The combination of high temperature, dense fibres and a high mould pressure creates a state where trapping water vapour is very likely. The high temperatures may also cause discoloration but this was not expected to be a prominent issue as the temperatures would be kept well within the limit of what the fibres can handle.

2.5 Future of moulded pulp

Since the early 1900s, various research has been focusing on moulded pulp and its applications. This has been boosted by the increased focus on environmentally friendly solutions and materials in our modern society. As mentioned in Section 2.4.2 there is a drive towards finding ways to generalise the mechanical properties of moulded paper products in terms of specific engineering equations but due to the nature of fibres, there is still a lot to do in this regard. This does, however, not mean that there can not be made any progress in other aspects of paper moulding. The focus on specific applications is still very viable and there are already a number of innovations that are under development or entering the market. Section 2.5.1 and 2.5.2 covers some of these new innovations. In addition to this, there is also research being conducted regarding waterproofing and degradation of these type of products. This is covered in Section 2.5.3.

Figure 2.3: Green Fiber Bottle prototype [50]

2.5.1 New paper products

Although the current state of the moulded pulp industry is mainly focused on the traditional applications of packaging, food trays etc. there are attempts at innovating and finding a broader use for moulded pulp and recycled paper. Multiple companies are attempting to create containers that use higher percentages of paper in their structural parts. These containers do, however, still have a need for some sort of plastic or non-paper material to provide protection against moisture. One example of these products is the ”Paper Water Bottle” [49]. This water bottle has a structural shell made entirely of moulded pulp but there is still a liner inside made from recycled plastics. It does, however, use significantly less plastic than traditional bottles and this type of concept is being seen more frequently in recent years. This is already a step in the right direction but there are some that seek to completely eliminate the need for plastic. One such project is the ”Green Fiber Bottle Project” from DTU in Denmark in association with Carlsberg A/S [50]. They are attempting to create a bottle made entirely from pulp that is 100% biodegradable and void of any type of plastic liner or similar solution. This is very similar to the approach that this thesis is taking to fibre-based materials and it is the next step that is required to obtain sustainable materials and products. A picture of a Green Fiber Bottle prototype can be seen in Figure 2.3.

2.5.2 Wood-fibre-reinforced composites

Another type of material stemming from the idea of biodegradable and sustainable alternatives to plastic are wood-fibre-reinforced composites (WFRC). This includes wood-plastic composites (WPC) and materials like MDF, which is a mix of wood fibres and glue. With the emergence of bioplastics on the market, some companies are looking to create products combining wood fibres and polymers. Durapulp from Södra [51] is an example of such a material which combines wood fibres with PLA in order to create a stronger plastic material. These materials are very strong and durable with excellent water resistance due to the use of bioplastics but there are currently issues with the use of certain bioplastics. These issues are covered in detail in Section 2.6 but the bottom line is that even though bioplastics are better for the environment due to them not being oil-based, they are still plastics which may suffer from a lack of biodegradability. This is the reason why this project seeks to explore the possibilities of pure fibres and natural biodegradable materials.

2.5.3 Waterproofing and degradation

The hygroscopic nature of paper fibres puts issues relating to waterproofing high on the list of required improvements to paper-based materials. A general-purpose paper based material would have to be more resistant to changes in humidity or direct contact with water compared to pure fibres. The issue of waterproofing is not within the scope of this thesis but Section 4.6.1 provides suggestions for methods and further research that could help solve this issue. In addition to the effect of humidity, paper is also highly susceptible to environmental degradation from processes such as photodegradation, biodegradation and thermal degradation [1]. Ou et al. [52] observed a reduction in the degree of polymerisation by 50%, a 10% to 16% decrease in weight and a 10% to 30% decrease in tensile strength due to ultraviolet irradiation. In addition, Sørensen et. al. [53] saw that large initial creep deformations were induced by humidity and temperature variation. However, these characteristics are what allows these materials to be biodegradable, which is a crucial aspect of this thesis. Therefore, the extent of the waterproofing and degradation protection has to be limited to a point where the protection is sufficient while still maintaining a level of degradation that is satisfactory from an environmental perspective.

2.6 Biodegradability and compostability

Biodegradability and compostability are important terms for discussing the future of sustainable materials, plastics and other commonly used materials with heavy environmental impacts. They are also used to define important characteristics of sustainable and environmentally friendly materials. Compared to biodegradability, the term compostability is seldom used to describe materials and products because it usually implies very specific characteristics. However, these two terms should not be confused. The Merriam-Webster dictionary defines biodegradability as the following:

Capable of being broken down especially into innocuous products by the action of living things (as microorganisms).

This definition is true both for compostable and biodegradable materials but that does not mean they are interchangeable or imply the same material characteristics. The difference lies in the details of the process and how quickly it happens. Some industries have implemented standard to govern this difference and for the European packaging industry the following definitions from Pagga (1998) [54] is in place:

> ***Biodegradation*** *is a degradation caused by biological activity, especially by enzymatic action, leading to a significant change in the chemical structure of a material.*

> ***Compostability*** *is a property of a packaging to be biodegraded in a composting process. To claim compostability it must have been demonstrated that a packaging can be biodegraded in a composting system as can be shown by standard methods. The end-product must meet the relevant compost quality criteria.*

The standard methods mentioned for judging the compostability of a material is covered in the *EN 13432* standard [55] which dictates the following:

- **Chemical composition:** The standard sets limits for volatile matter, heavy metals (Cu, Zn, Ni, Cd, Pb, Hg, Cr, Mo, Se, As) and fluorine

- **Biodegredation:** Chemical breakdown of materials into CO2, water and minerals. Pursuant to the standard at least $90\,\%$ of the materials have to be broken down by biological action within 6 months.

- **Disintegration:** The physical decomposition of a product into tiny pieces. After 12 weeks at least $90\,\%$ of the product should be able to pass through a 2 x 2 mm mesh.

- **Quality of the final compost and ecotoxicity:** The quality of the compost should not decline as a result of the added packaging material. The standard specifies checking this via ecotoxicity tests: this involves making an examination to see if the germination and biomass production of plants are not adversely affected by the influence of composted packaging.

This standard requires all of these criteria to be met for a material to be deemed as compostable which for a lot of ”biodegradable” materials, like certain bioplastics, will prove to be difficult to achieve. Some materials may be industrially composted but that raises the question of whether an industrial composting process is more sustainable than recycling. In terms of sustainability, the goal is to use and process materials that can degrade without having significant impact on the environment whether the material continues its life-cycle in different forms or ends it at a landfill. This would mean that it would have to be compostable and not just biodegradable. Bioplastics are very popular as a new ”bio-friendly” alternative to conventional plastics but some bioplastics still have the same chemical structure as fossil-oil based plastics (e.g. BioPE and BioPP) which in turn creates issues with biodegradability and compostability. For this thesis, the use of bioplastics will not be explored and the focus will rather be on exploring the capabilities of a pure fibre-based material.

2.7 Tensile testing

In order to evaluate the moulded samples that were to be made, Jacobsen (2016) conducted research on which type of standardised testing would be the most fitting for the project. Tensile testing was chosen over 3-point flexure tests due to the following reasons:

- Tensile testing provides a homogenous load over the sample cross section which is favourable for a fibrous material like this.
- A 3-point test applies a force directly to the test surface which could affect the fibre samples differently than plastics in ways that could be difficult to account for.
- Flexure testing requires a thicker sample which would make moulding a solid and strong sample significantly more difficult.

Due to issues with the mould no tensile testing was done by Jacobsen (2016) and the task of conducting these tests were transferred over to this thesis.

Tensile testing is one of the fundamental test forms for assessing the basic mechanical properties and behaviours of a material [56]. Tensile testing is done by taking a sample of the material that is to be tested and secure it in a test rig which then applies a tensile load until failure of the sample is achieved. Tensile testing is a well-established test method with a number of different standards that governs sample dimensions and test parameters. This thesis has followed two different standards for its tensile testing. For the assessment of basic mechanical properties of the fibres the ISO 1924-3 [57] standard for tensile testing of paper sheets was used in combination with a *Lorentzen & Wettre tensile tester* (see Figure 2.4). However, for the moulded samples the goal was to compare the results directly to plastic samples so the ASTM D638 [58] standard for tensile testing of plastics was followed and the tests were done with a *MTS Criterion Model 42* test rig (see Figure 2.5). This form of tensile samples is often referred to as *dogbones* due to their characteristic shape with two wider grip sections and a narrow middle section.

After the tensile testing, a stress-strain curve is usually drawn from the test data in order to assess the mechanical properties of the sample. An example of this type of curve can be seen in Figure 2.6.

The initial part (a) is the linear elastic region which implies that if the sample is unloaded while still in this region it will return to its initial shape with no permanent deformation. The slope of this linear region characterises the material stiffness, represented by the Young's modulus, often written as E. The Young's modulus is derived from Hooke's law and is calculated by dividing the tensile stress in the elastic region by the corresponding extensional strain according to Equation 2.1.

$$E = \frac{\sigma(\epsilon)}{\epsilon} = \frac{F/A}{\Delta L/L_0} = \frac{FL_0}{A\Delta L} \tag{2.1}$$

Once the sample reaches the end of the linear elastic region it will start to yield and plastically deform, meaning that any deformation is irreversible. This region is marked (b) in the figure and the initiation point of the yielding is referred to as the yield strength σ_{ys}. At the top of the curve, the ultimate tensile strength is found, σ_{UTS}. This signifies the greatest stress that the sample was able to withstand. For a brittle material, the fracture point, σ_f, is often close

Figure 2.4: L&W Tensile Tester

Figure 2.5: MTS Criterion Model 42 tensile test rig

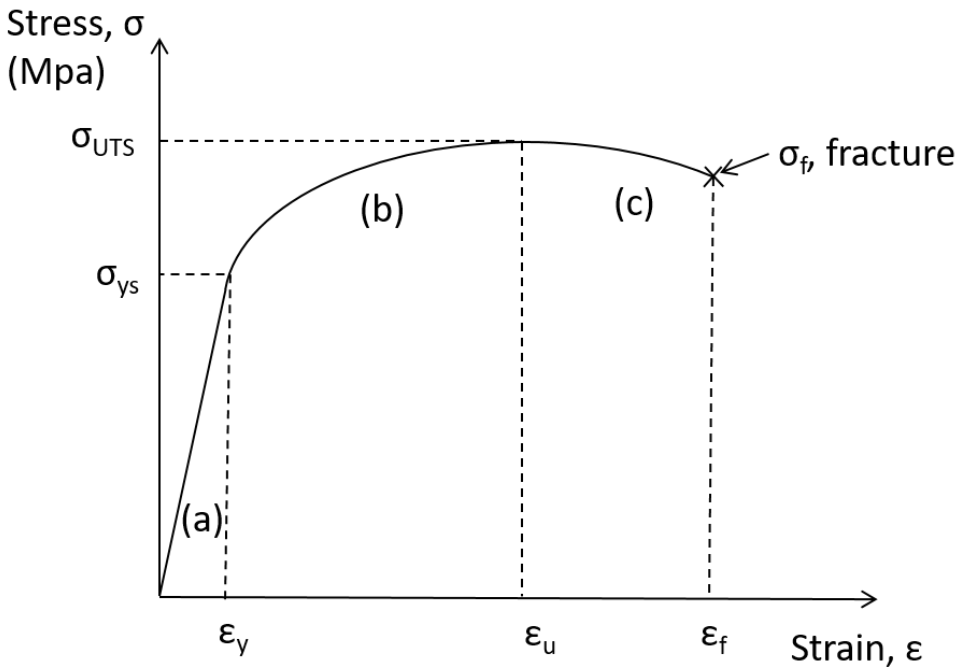


Figure 2.6: Engineering stress-strain curve of a typical ductile material, courtesy of Anders Bratli Wold

to the ultimate tensile strength. For a ductile material, on the other hand, the stress will keep decreasing after reaching the ultimate tensile strength while the strain is increasing. This is the region (c) in the figure and is due to necking in the sample, which is a narrowing of the cross-sectional area of the sample.

The visco-elastic-plastic nature of fibre materials implies that the stress-strain curves that will be drawn from the testing done in this thesis will not likely look exactly like that in Figure 2.6, but the concepts of the stress-strain curve still remain the same in terms of assessing the mechanical properties.

Chapter 3

Experiments and prototyping

3.1 The mould

During preliminary research, Jacobsen (2016) uncovered a number of issues related to the aluminium mould that was used for the initial testing. These issues were covered in detail in Jacobsen (2016) but the following is a summary of the main issues.

- Milling to the depth required for the mould caused inaccuracies and tool issues with the CNC mill.
- The dimensional tolerance was too large (as a result of design combined with the milling-inaccuracies) causing the mould to jam and allow fibres to creep up the sides of the mould.
- Aluminium turned out to be too soft as a mould material which over time caused a number of issues with jamming.

A number of possible steps were suggested in order to solve these issues with the most important one being a move from aluminium to proper tooling steel. The workshop at MTP Gløsuahgen had a block of Uddeholm Calmax [59] available which is a well rounded tooling steel with good wear resistance. In addition to this the tolerance of the mould was decreased from $0.2\,\mathrm{mm}$ to $0.1\,\mathrm{mm}$ in order to provide a tighter fit that should be able to solve some of the jamming issues experienced with the aluminium mould. The mould was also redesigned to have a detachable bottom. The purpose and benefit of this were discussed in depth in Jacobsen (2016) but the most important objective was to make the milling process easier. After consulting with the workshop engineers at MTP Gløshaugen it was concluded that there still was a chance that they would not be able to achieve the tolerance and accuracy needed for this mould due to the hardness of the tooling steel and the depth of the mould. It was suggested that it could be done a lot more accurately and precisely by doing wire electrical discharge machining (EDM). This process is often called spark machining or spark eroding. It works by pulling a wire through the work-piece and essentially cutting the steel by rapid current discharges between the workpiece and the wire [60]. This is a slow but very accurate machining process. This type of equipment was available at MTP Valgrinda so the job of machining the mould was transferred to the MTP Valgrinda workshop. Initially, it was only the female part of the mould that would be spark eroded but after an evaluation of the models, it was decided to also machine the male part in two separate pieces. This would ensure the best possible fin-

ish and accuracy. Spark eroding is a very time-consuming method but the results it provides greatly outweigh the time cost of the process. The finished mould can be seen in Figure 3.1 and 3.2. These images show the mould after quite a bit of use so it has begun to show some wear but this is just on the outside. The inside of the mould, which has been covered with a release agent, is still in excellent condition. The results of the spark eroding were very satisfactory and there were no jamming issues present with this new iteration of the mould. The tooling steel has also proven to be very wear resistant and in combination with a release agent the inner moulding surface has been kept in good shape and free of corrosion. The release agent used for this mould was *Chemlease 2185* from Chemtrend [61]. This is a release agent with very low viscosity, no significant buildup and a good wear resistance. This is very desirable for a mould with a tolerance as tight as the one used for this project. According to the manufacturer this particular release agent should be able to handle temperatures reaching 230 °C - 250 °C once cured [Personal correspondence, 7th March 2017], which was more than sufficient for this project. The mould made from the Calmax steel worked quite well to the extent of this thesis but in regards to future pulp moulding, a different material should be used. The mould has started to show signs of corrosion since the steel was not stainless or inert to the pulp, which are important characteristics that a more permanent mould would need to have.

Figure 3.1: Spark eroded steel mould disassembled

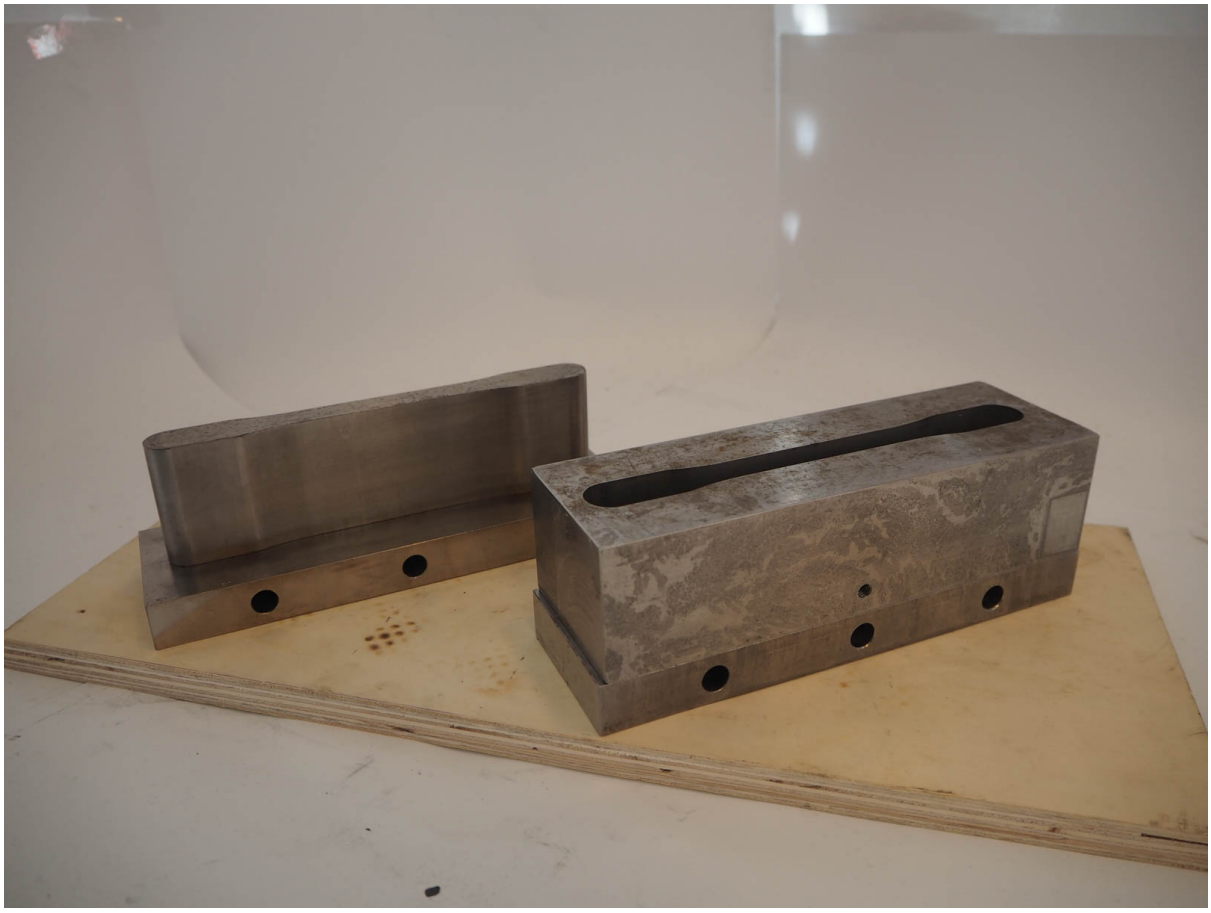
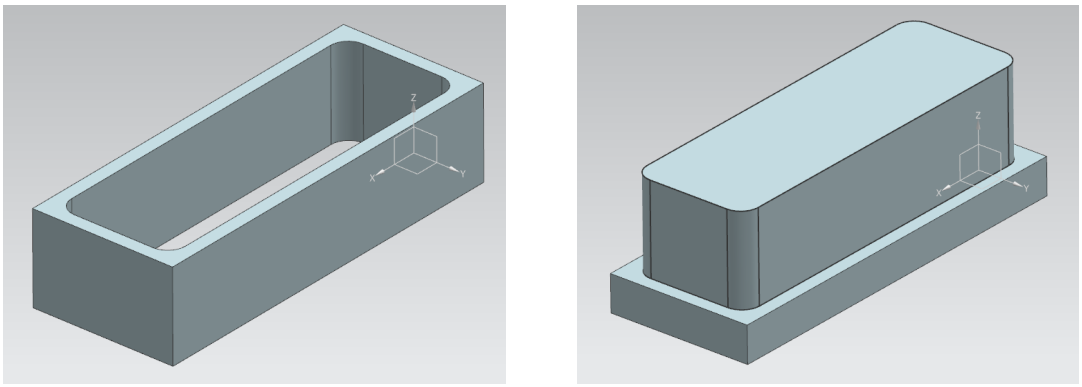


Figure 3.2: Spark eroded steel mould assembled

3.2 New mould design and laser exploration

While the mould described in Section 3.1 was being produced, a new design idea for a second mould was devised. The original mould was only capable of producing a single sample at a time so there was a desire for a solution that could create multiple samples at a time. The new concept revolved around a mould that would function in the same way as the current one but instead of moulding a single dogbone shape, it would mould a larger sheet from which multiple samples then could be laser cut. Figure 3.3 shows the early concept models of the female (a) and male (b) parts created for this new mould. The software used for this was Siemens NX 11. The tensile sample shape was only $19\,\mathrm{mm}$ wide so multiple samples could potentially fit on a relatively small sheet. Before this idea could be developed further, a test with the laser cutter had to be conducted first to make sure that the laser would be able to cut through the fibres properly. Some old samples of TMP were subjected to a simple cut from the laser using pre-existing parameters used to cut MDF. The cut, seen in Figure 3.4, was very clean but it was clear that the laser had not only cut through the fibres but also heat treated them in some way making the cross-sectional area touched by the laser seemingly harder than the uncut edges. For this project, the effect the laser had on the edge meant that it could not be used to cut samples to be used for tensile testing as there was no way at this point to estimate the effect the laser-affected edge would have on the tensile properties of the sample. Exploring this effect would require extensive research which was far outside the scope of this thesis. These findings



(a) Female **(b)** Male

Figure 3.3: CAD models of the new mould concept

did, however, raise an interesting question: Could some sort of treatment by a laser make the surface of the material more resistant to water or alter the mechanical properties? Because of this, it was decided that a non-extensive probing experiment would be conducted to see what kind of effect the laser could have on the fibre samples. Waterproofing was not within the main scope of this thesis but some light exploration of this concept could add another point to the list of future solutions to the issue of waterproofing or improving the properties of a pure fibre material. The samples used for the laser experiment consisted mainly of used tensile samples that had already served their purpose. This experiment is covered in Section 3.9.

3.3 Filtering improvements

The filtering system used by Jacobsen (2016) was created in order to filter the pulp straight into the mould, thereby creating a homogeneous mass that was ready to be moulded. The filtering system, pictured in Figure 3.5, did work quite well at the time. It did however become clear that for making multiple samples and larger batches, this simple gravitational filtering would be too time-consuming. The solution to this was the addition of a vacuum to drain off the water more efficiently. Co-supervisor Blindheim was already planning some experiments with the type of vacuum moulding described in Didone et al. (2017) [1] so the effort of these two ideas was combined into creating a vacuum system that could work for both the sample filtering and the vacuum moulding.

A vacuum can be achieved by simply pumping air out of a sealed container but in this case, with water present, it was desirable that water is not pulled through to the source draining the chamber of air. A separate steel chamber was made that would connect to the filtering system and the vacuum source. To pump the air out of the chamber, a vacuum cleaner was initially used but at a later stage, a vacuum ejector was acquired. A vacuum ejector creates a vacuum by utilising Bernoulli's principle which states that when the speed of a fluid is increased, the pressure is decreased [62, 63]. By running a high-pressure air stream through an ejector, a pressure imbalance is created which in turn creates a vacuum suction. A simple visualisation of this principle is shown in Figure 3.6. The airflow and thus also the vacuum can be controlled and it is a very efficient way of creating a complete vacuum. The vacuum ejector does, however, not move as much volume as a vacuum cleaner which turned out to be an issue for this particular

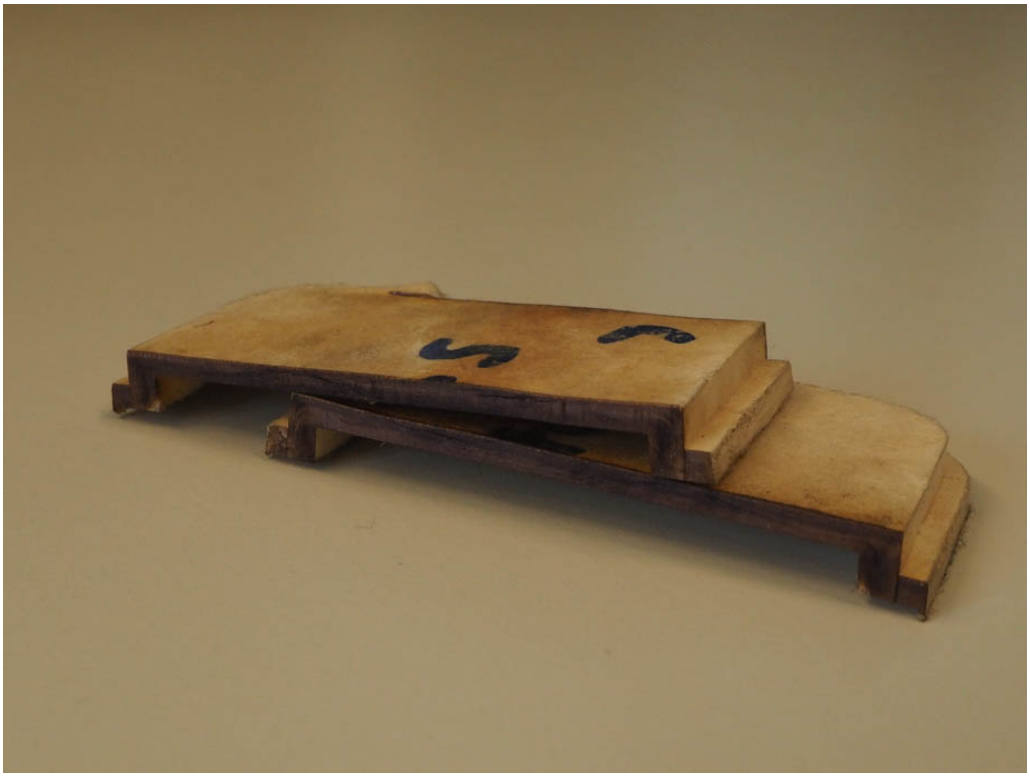


Figure 3.4: The laser cut edge of a TMP sample

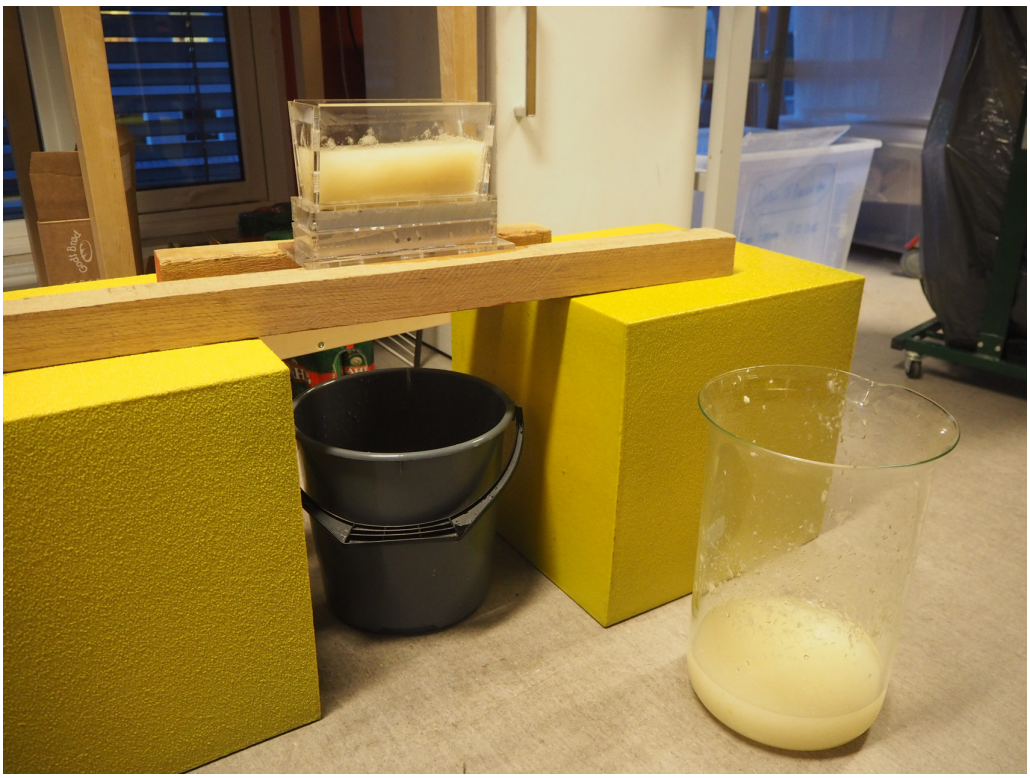


Figure 3.5: Filtering prototype

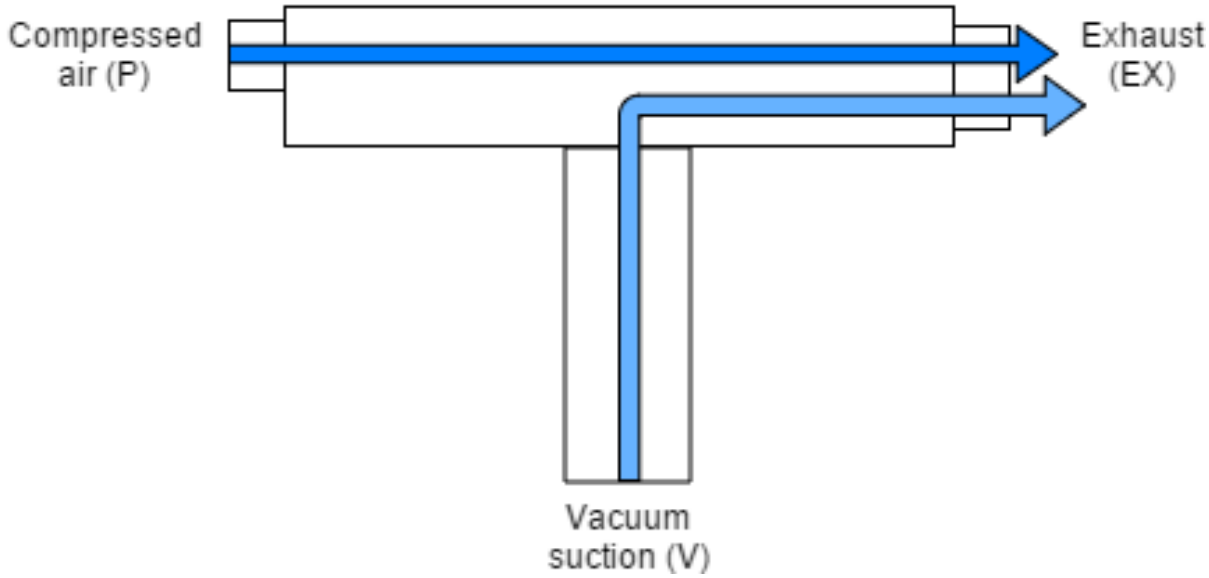


Figure 3.6: Vacuum ejector functional principle

process. The filtering system was not completely air tight but had small gaps through which air could enter. Because of these small gaps, the ejector was not capable of creating a strong vacuum suction because it drew a lot of air through these gaps. This was especially a problem at the end of the filtering process when the fibres had started to form a thick layer in the bottom of the mould, limiting the flow of water further. At this point, the vacuum just pulled air through the gaps and left the water untouched. The vacuum cleaner, on the other hand, moves a much larger volume of air which allowed it to maintain a certain amount of vacuum suction on the water, despite drawing air through these small gaps. It could have been possible to completely seal the filtering system to prevent the issues with the ejector but since the filtering system was taken apart for each sample it would have had to be resealed before each filtering. This would not be worth the time and effort compared to simply using the vacuum cleaner. Because of this, the vacuum cleaner became the primary vacuum source for this process. Figure 3.7 shows the complete vacuum assisted filtering system and Figure 3.8 shows a freshly filtered sample ready for thermomoulding. Further pictures of the filtering process can be found in Appendix A.

3.4 Paper sheet making

The process of moulding the tensile test samples was an important step to develop this material but it also involved an experimentation process, during which a lot of obstacles could appear, especially related to the moulding process itself. Because of this, a form of more reliable testing of mechanical properties was needed to establish baseline properties and behaviours of the different fibre types. Knowing these parameters could prove to be crucial to moulding the fibres effectively. This is why it was decided to make paper sheets for tensile testing as well. Paper sheet making is a process that is standardised through the ISO 5269-1 standard [2] and it is something that PFI has a lot of experience with. For this project, however, it had to be done a bit differently. Paper sheets are usually very thin and air dried but to be able to compare the results from the paper sheets and the moulded samples the paper sheets had to be created

Figure 3.7: Vacuum setup

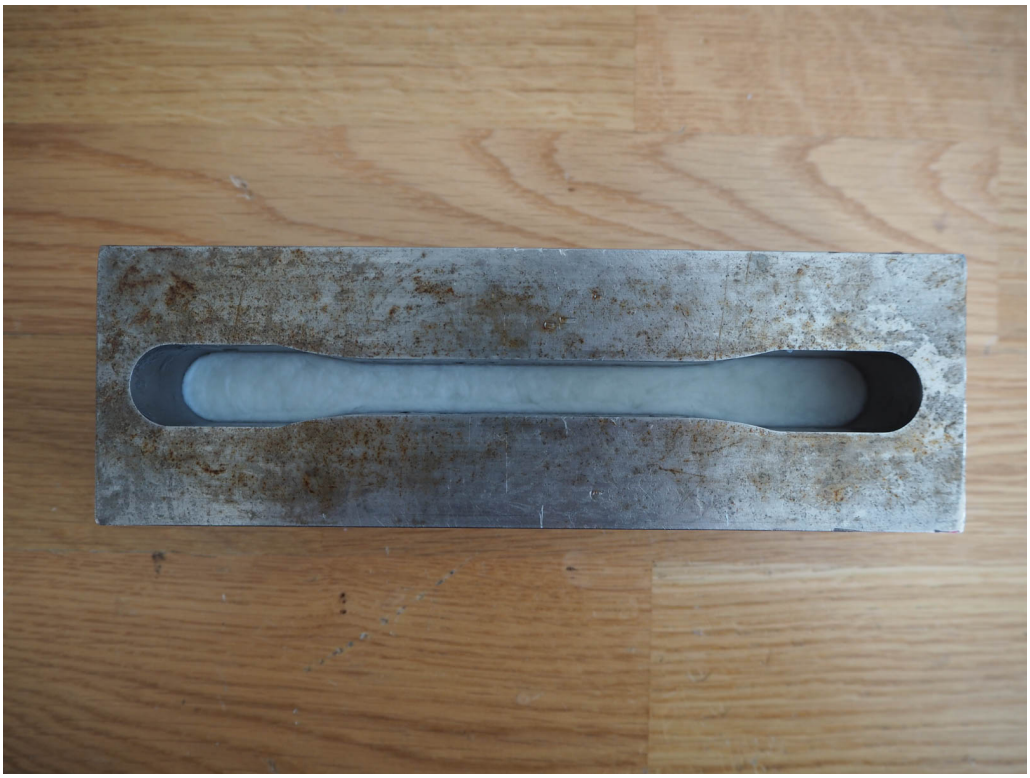


Figure 3.8: Filtered sample

in a similar fashion. This meant much thicker sheets that would be pressed and heated in the same manner as the tensile samples.The hypothesis was that this would create a similar fibre structure. These sheets would have a sheet-weight of $200\,\mathrm{g/m^2}$ which is significantly higher than the $50\,\mathrm{g/m^2}$ to $70\,\mathrm{g/m^2}$ the ISO standard usually dictates. The sheet former would create sheets that were $19.5\,\mathrm{cm} \times 19.5\,\mathrm{cm}$ which then translates to a fibre amount of $7.6\,\mathrm{g}$ per sheet. To ensure enough tensile samples from each batch it was decided to make four sheets per batch for a total of $35\,\mathrm{g}$ of fibres, which is close to the limit of what a single disintegrator can handle. The process for making these sheets had two distinct parts. The standardised sheet-making process in accordance with ISO 5269-1 [2] (this process is covered in detail in Appendix B) and the heated pressing of the sheets. The heated pressing was not something that had been attempted by PFI before so there was a lot of initial testing that had to be done in order to calibrate the method and find the right parameters to ensure the sheets came out with the desired finish and quality.

The press used for this, a *Fontune Presses LPB 300*, has three adjustable parameters; temperature, pressure and time. These range from $60\,\mathrm{kN}$ to $300\,\mathrm{kN}$, $0\,^{\circ}\mathrm{C}$ to $300\,^{\circ}\mathrm{C}$ and the built in timer goes up to 99 minutes and 59 seconds. For the initial rough calibration, four paper sheets were made and cut into four smaller pieces for a total of 16 samples to be used for calibration. The first three samples were pressed for 5 minutes at $300\,\mathrm{kN}$ with temperatures starting at $60\,^{\circ}\mathrm{C}$ and then increasing by $15\,^{\circ}\mathrm{C}$ for each sample with a fourth sample being pressed at only $150\,\mathrm{kN}$. These all came out somewhat moist so the next samples all had their parameters tweaked to see if higher temperatures or longer curing times would ensure that the samples were properly dried. After this initial testing a couple of things had been discovered:

- The temperature has to be at least $120\,^{\circ}\mathrm{C}$
- Higher pressure seems to have very little impact on how dry the samples turn out and the results seem to be best at the lowest possible pressure of $60\,\mathrm{kN}$.
- At high temperatures, it does not take more than a couple of minutes to dry the sample.

In the next round of calibration, there were issues with some of the sheets developing larger rough spots on the usually smooth surface of the pressed sheets. This was only present in the denser TMP sheets and was thus believed to be caused by water getting trapped due to the pressure. Examples of this can be seen in Figure 3.9. This was alleviated by increasing the temperature even further to $135\,^{\circ}\mathrm{C}$ and after a couple tests with the timing a final set of parameters were found: $135\,^{\circ}\mathrm{C}$ - 3 minutes - $60\,\mathrm{kN}$. These parameters gave very dry and compact samples with excellent surface finishes. After being pressed the samples were stored and conditioned in accordance with ISO 187 [64] to prepare for tensile testing. This standard dictates that the conditions for sample storage before testing should be a temperature of $23 \pm 1\,^{\circ}\mathrm{C}$ and a relative humidity of $50 \pm 2\,\%$. A sufficient conditioning time would be a minimum of 4 to 8 hours depending on sheet weight. For this project, all samples were conditioned overnight to ensure proper conditioning.

3.5 Sheet tensile test setup and sample preparation

For the initial tensile test, a *Lorentzen & Wettre Tensile Tester* was used. This rig tested one sample at a time until a predetermined number of samples had been tested and it then printed out complete test data and parameters for each sample and the average values of the full sample

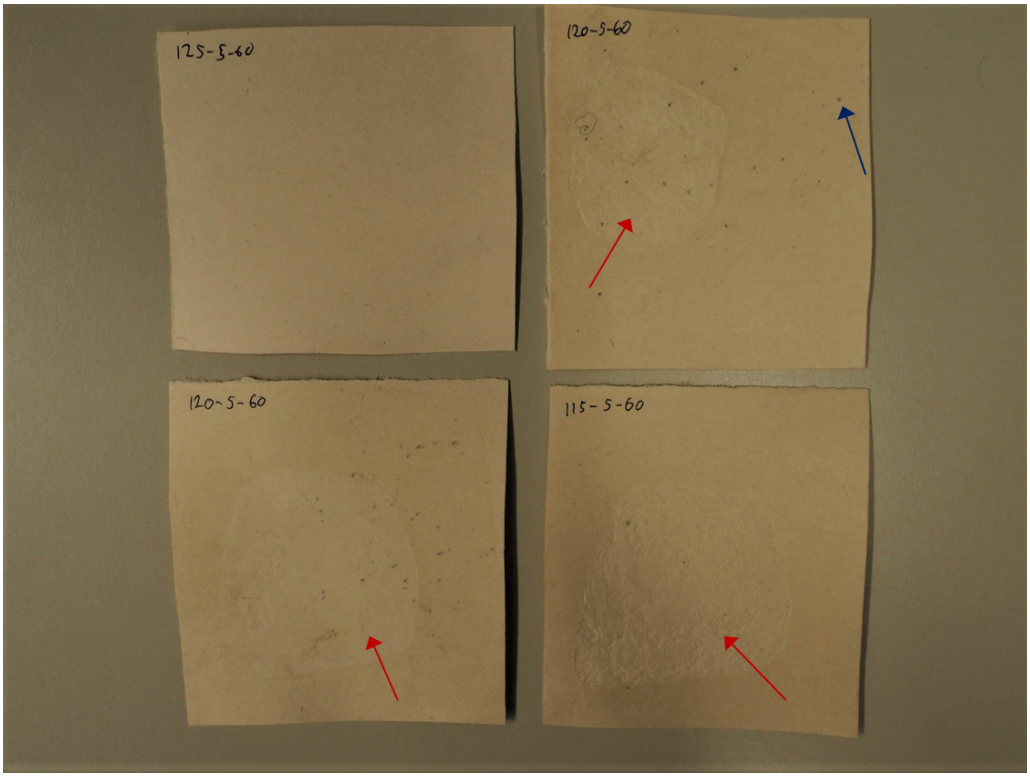


Figure 3.9: Spots on the hot-pressed sheets. The red arrows indicate the rough spots that emerged during the hot-pressing. The blue arrow indicates a minor defect that is believed to be a result of impurities either in the pulp or on the metal plates used for the pressing.

series. By utilising a serial monitoring software on an external computer it was possible to write every data point from the test to a text file as well. Unfortunately, the serial monitor only provided data on the force applied to the sample and not the corresponding elongation. Because of this, the elongation had to be estimated from the peak values provided by the data printed by the test rig. The test rig stretches the samples at a constant rate of $100\,\mathrm{mm/min}$ which should allow a linear estimation of the elongation to be made if the peak values of elongation and force are matched up correctly.

The sample strips for this rig were made by using a punching tool to punch out the sample strips from the paper sheets. These strips usually vary in length depending on the dimensions of the sheet but the length was not a parameter of importance since the tensile test rig had a fixed length between its grips of $100\,\mathrm{mm}$. The samples had a width of $15\,\mathrm{mm}$ along the entire length of the sample. Before being punched out, the sheets were first measured in terms of thickness and roughness. This was to ensure that there was consistency across the different sample sheets and to exclude any sheets that had parameters that deviated too much. Thickness and roughness were measured at five different points per sheet in the following order: upper left, centre, upper right, lower right, lower left. This was measured by using a micrometre and a Parker Print Surf test in accordance with ISO 534 [65]. The measured thickness and roughness of all sheets in this initial run can be seen in Appendix D. From a batch size of four sheets, three sheets were chosen and three samples were punched from each sheet. The thickness of each individual sample was then measured in order to calculate the cross-sectional area correctly. After the tensile tests had been conducted the samples were stored at the same climate as during the pre-testing period mentioned in Section 3.4. This was so that future inspection or microscopy of the samples could be conducted if necessary.

3.6 Mould calibration and testing

After the initial tensile tests, it was time to move on to the moulding and testing of dogbone samples. Once the mould was finished and properly prepared with a release agent, the work of calibrating and testing the mould began. There are a number of parameters that dictate the results from the moulding process, including applied force, drying time and fibre amount. The goal was to achieve a sample with a thickness of $3.2 \pm 0.4\,\mathrm{mm}$ [58] with a good surface finish and a homogeneous structure without any structural flaws or weaknesses. The first step was to calibrate the fibre amount needed for each sample. A starting point of around $7\,\mathrm{g}$ per sample was chosen based on previous moulding attempts from Jacobsen (2016). After a couple iterations it seemed like $7.5\,\mathrm{g}$ of fibres produced samples with the desired thickness. This was tested for both TMP and Kraft and in both cases, it returned a sample thickness around $3.2\,\mathrm{mm}$. After attempting to mould a full five sample series this did, however, turn out to be wrong. Due to unknown causes, the samples moulded with $7.5\,\mathrm{g}$ of fibres turned out too thick and a new round of calibration had to be done. This deviation might have been due to errors made in the initial calibrations but the cause is still unknown. After re-calibrating it was concluded that the TMP needed about $7.1\,\mathrm{g}$ to achieve the desired thickness. It was also discovered that the activated Kraft fibres produced significantly thicker samples than the TMP and thus a separate round of calibration had to be done for the Kraft fibres. This calibration landed at $6.5\,\mathrm{g}$ per sample. For all subsequent mouldings $7.1\,\mathrm{g}$ for TMP and $6.5\,\mathrm{g}$ for Kraft have been producing consistent and correct sample thickness.

These calibration runs were also used to evaluate the effect of pressure on the sample. In Jacobsen (2016), all the samples were subjected to a force of at least $50\,\mathrm{kN}$ but during the new mould calibrations the force applied to the mould was significantly decreased to $5\,\mathrm{kN}$ without any apparent change in sample density. There were, however, some issues with samples showing signs of wet pockets withing the narrow section of the sample. These would appear in the same spots each time with varying size and extension. Images of this can be seen in Figure 3.10. These spots would shrink when air dried causing the sample to get an uneven and sometimes warped structure. These spots would only appear on the TMP samples and never in the gripping sections where the mould had drainage holes. Drying time did not seem to affect the wet pockets either. Because of this, the hypothesis was that the force exerted on the sample during drying was preventing the water from escaping the centre of the sample. To prevent this from happening, it was attempted to adjust how the force was applied to the sample. After some testing, it was concluded that the optimal procedure would be to change the force during different phases of the moulding process. In addition to this, the samples would also be left a couple minutes longer during the pre-moulding vacuum filtering to ensure that as much water as possible was drained out of the sample. In combination, these changes solved the issues and provided samples with no sign of wet pockets or other irregularities. The initial samples also showed some signs of post-moulding instability where they would start bending slightly over time after moulding but once the parameters of the moulding process had been tuned so that the water pockets were no longer present, the post moulding instability also became a rare occurrence. The instability was most likely caused by improper drying so when the parameters were changed to properly dry the sample, the instability issues were also solved.

The final calibrated moulding process was done in accordance with the following procedure:

- A pressure of $5\,\mathrm{kN}$ is applied to compress the sample and the heating process is started.

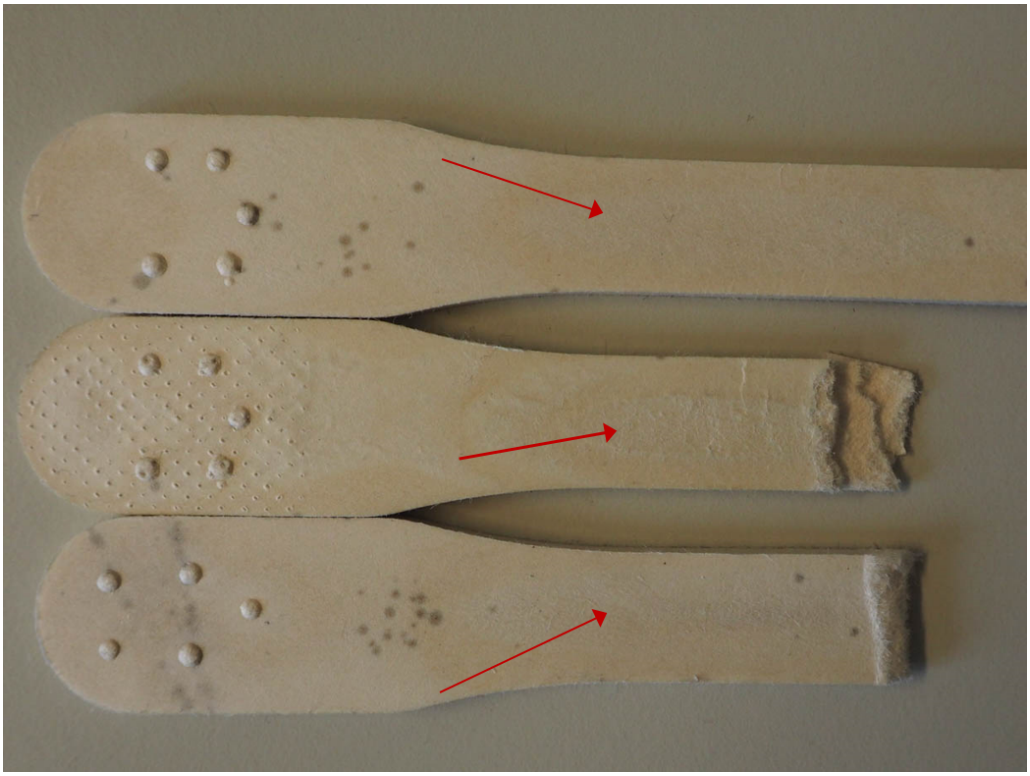


Figure 3.10: The red arrows point to varying degrees of dry spots on some of the early moulded samples. These samples were deemed defect and was used later to calibrate the tensile test rig, which is why they are fractured in this picture.

- When the water starts boiling, the pressure is released and just enough force is applied to prevent the sample from expanding.
- After most of the initial water has evaporated (when no steam or water is seen anymore) the force is increased to about $1\,\mathrm{kN}$ for the last phase.

These three phases take five minutes each with an extra five minutes added to the last drying phase for kraft fibres due to the kraft fibres absorbing more water during the filtering. A rough representation of the pressure and temperature at different times can be seen in Figure 3.11 and Figure 3.12 shows a picture of the setup in the middle of the moulding process. There was a clear tendency of the Kraft pulp being more difficult to compress than the TMP. The TMP would usually be fully compressed within a minute of the initial force being applied but the Kraft pulp would spend close to the full five minutes of the compression phase before the correct thickness was achieved. This does, however, make sense when considering that the Kraft samples do seem to retain quite a bit more water when filtering than the TMP.

Once the calibration was finished, a batch of TMP and a batch of activated Kraft was prepared with enough fibres to create two separate series of five samples, which is the minimum size of a series of samples according to ASTM D638 [58]. After moulding, the samples were conditioned in accordance with ISO 187 [64] just like the paper sheets before being tensile tested on the *MTS Criterion Model 42* tensile test rig. The results of these tests can be found in Section 4.2. Figure 3.13 shows examples of a post calibration TMP and Kraft sample. They both showed signs excellent surface finishes, high density and good perceived mechanical properties. The small spots of discoloration on the surface was a result of impurities in the mould due to pitting on the tool surface due to the steel not being inert to the pulp. They were

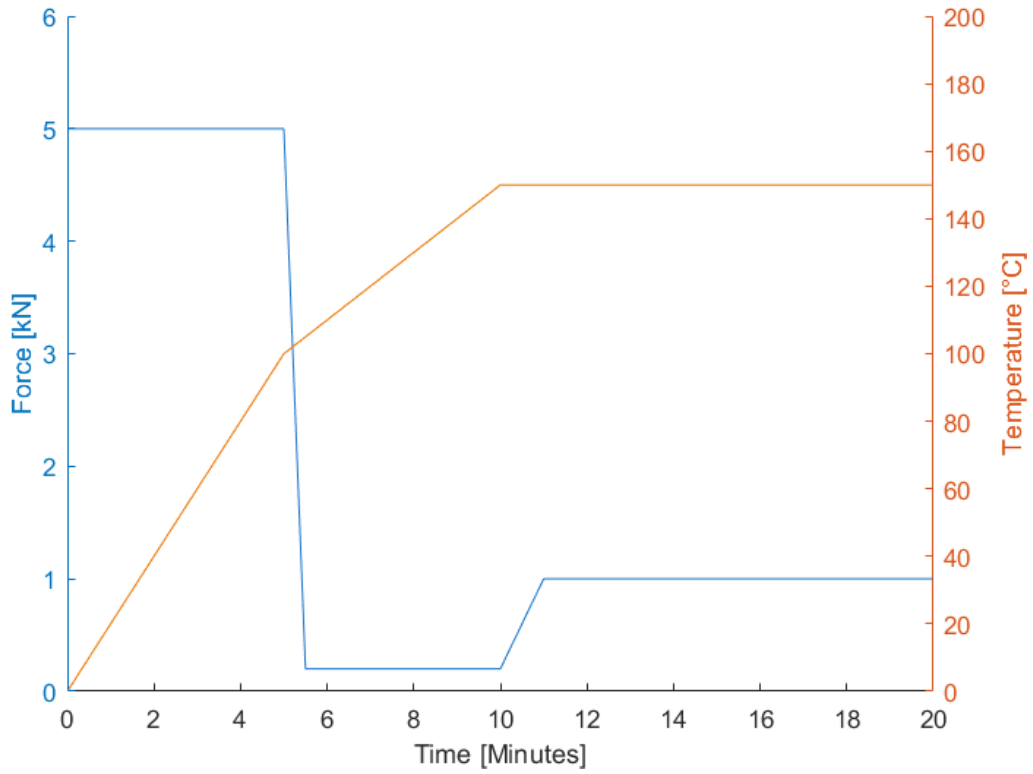


Figure 3.11: Moulding process force and temperature over time

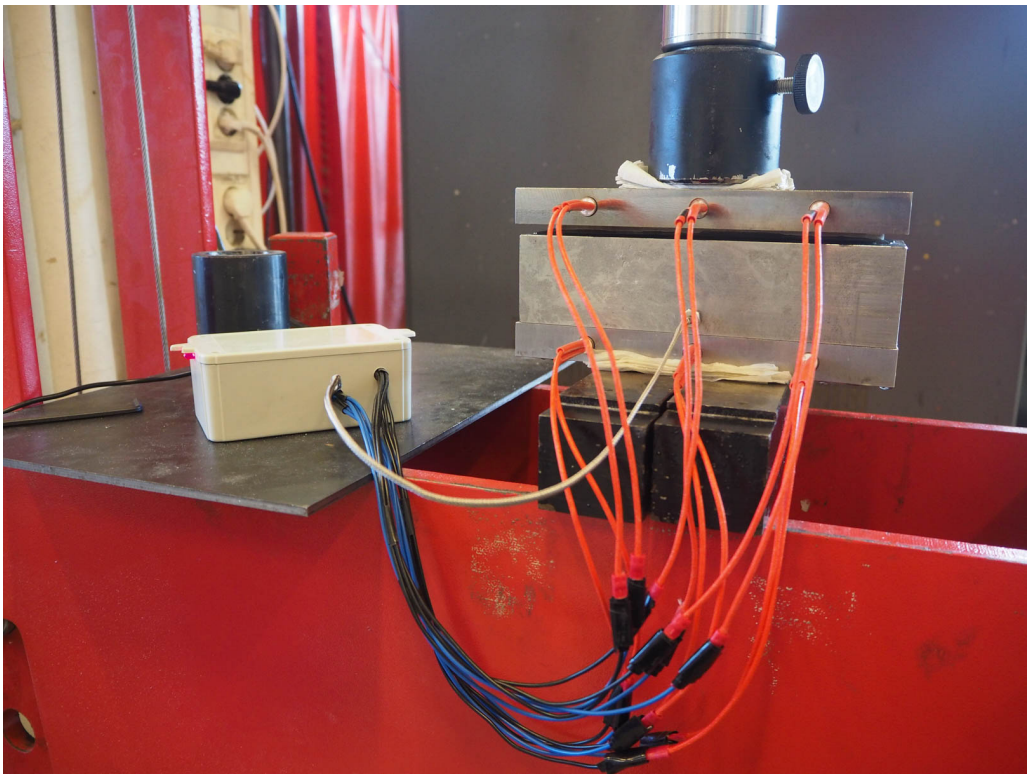


Figure 3.12: Moulding setup: After the pulp has been filtered down into the mould, the bottom is attached and the male mould half is inserted. The mould is then placed in a hydraulic press and a temperature sensor and six 250 W heating elements, controlled by a PID controller, is inserted to heat the mould.

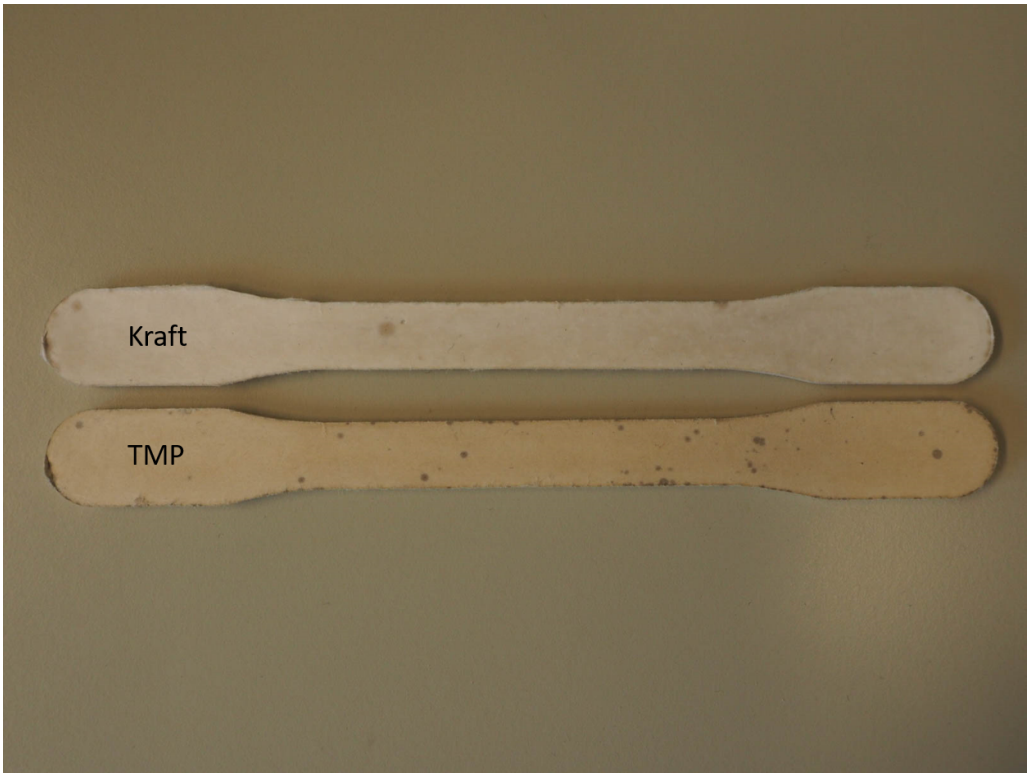


Figure 3.13: Moulded TMP and Kraft samples

purely superficial and was not believed to affect the structure of the sample.

3.7 MFC-reinforced tensile testing

After the initial tensile testing of paper sheets and dogbones made from pure TMP and Kraft pulp, the next step was to add varying amounts of MFC to the pulp to assess the effects of MFC on the tensile samples. There were three main questions that needed to be answered:

- Does MFC increase the tensile strength of the samples?
- What effect does MFC have on the ability to dewater the pulp?
- How does MFC affect the moulding process?

The pulps that were to be used for these tests were the same TMP and activated Kraft pulps as earlier, with the addition of refined and homogenised MFC. (800 bar, one pass)

In order to address the first question, it was decided to conduct tests on paper sheets. This was because the paper sheets provide a test method specialised for testing these types of fibres and should provide more accurate results than moulded samples since that process was still experimental. In addition, the purpose of the moulded samples was to provide samples that can be compared to plastics, which was not necessary for this step as the goal was only to compare the tensile strength of fibre samples with and without MFC. Moreover, previous testing and theory suggested that adding MFC to the moulded pulp would most likely complicate the process and make it more difficult to create samples without defects. This is why it was decided to do the tensile testing on paper sheets and rather mould the MFC-reinforced pulp as

an experiment to see what effect MFC has on the moulding process without having to output full tensile test series. That experiment is covered in Section 3.8. The assessment of the dewatering of the pulp with MFC added would be conducted during both the making of the sheets for tensile testing and during the filtering for the MFC-reinforced moulding. This would be done by timing the drainage time for each sheet or sample.

The first step in planning the tensile test was to find a starting point for the amount of MFC that were to be added. Previous studies had shown that once you start adding more than 4-5% of MFC to a pulp, the increase in dewatering time is much greater than the increase in strength. [13] This is why an MFC amount around 4% had been established as a sort of compromise. For this thesis, it was decided to do three test series with 2%, 4%, and 6% of MFC added to both TMP and Kraft pulp for a total of six test series. The process of making MFC-reinforced paper sheets was the same as the process covered in Section 3.4 both in the process and the batch sizes. There was, however, one important difference since the correct amount of MFC has to be added to the pulp. This was done as a separate step after the disintegrated pulp had been mixed to a concentration of roughly $3\,\mathrm{g/L}$. The exact concentration was then measured and by multiplying this concentration with the total amount of pulp in the batch, the amount of TMP or Kraft present in the pulp was found. This could then be used to calculate how much MFC was needed to get the correct concentration by using the following formula:

$$\frac{x}{\text{Amount of TMP/Kraft fibres in the pulp} + x} = \frac{\text{Desired percentage of MFC}}{100} \tag{3.1}$$

This equation would, when solved for x, output the correct amount of MFC, in grammes, needed to achieve the correct concentration of MFC in the pulp. This amount of MFC was then measured and mixed into the pulp. The pulp was then ready to be poured into the sheet maker. There was, however, one important consideration to this process. Knowing the concentration of the pulp and the desired sheet weight, you usually calculate the amount of pulp needed to make one sheet. The same was done here but the initial concentration measured was for the pulp before MFC was added. With the addition of MFC, more fibres have been added to the pulp without increasing the volume. To adjust for this the following equation was used:

$$\frac{\text{Measured concentration without MFC}}{1 - \frac{\text{Desired percentage of MFC}}{100}} = \text{Final concentration with MFC added} \tag{3.2}$$

The final concentration was then used to calculate the amount of pulp needed for creating a sheet of paper. The rest of the paper process and tensile testing was then done in the same manner as the non-reinforced sheets. During the heated pressing of the sheets, there were however once again some defined rough spots that emerged on the TMP sheets, similar to what was seen during the initial calibration covered in Section 3.4. The spots were however easily avoidable when punching out the tensile test samples so the tensile tests of the reinforced sheets were conducted without any further issues. The spots and their implications are covered in detail together with the tensile results in Section 4.3.

Table 3.1: MFC-reinforced sample distribution

# of samples	% TMP	% Kraft	% MFC	Total amount of fibres [g]
2	100	0	0	7.1
2	98	0	2	7.1
2	0	100	0	6.5
2	0	98	2	6.5

3.8 MFC-reinforced moulding

The next step after conducting the MFC-reinforced tensile tests was to run a couple of moulding experiments to see what effect MFC had, if any, on the moulding process itself. The draining process for the moulding was already a somewhat time-consuming method so it was expected that any addition of MFC would increase the drainage time significantly. Because of this only 2% of MFC was added to the batches used for this experiment. In total this experiment consisted of four separate batches, two with TMP and two with Kraft. 2% MFC was then added to one TMP batch and one Kraft batch in a manner similar to what was done when making the MFC-reinforced paper sheets in Section 3.7. Each batch had enough pulp for moulding two samples and Table 3.1 shows the total amount of planned samples with their respective fibre amounts. The total fibre amount for each sample was kept the same as the tensile sample moulding in Section 3.6 to ensure that the sample thickness would be within the ASTM D638 standard.

The filtering and the moulding were conducted under the same parameters as the moulding done in Section 3.6 with the exception that the drainage time was monitored much more closely to be able to quantify the effect of the MFC. The results and discoveries from this experiment are covered in detail in Section 4.4.

3.9 Laser treatment and inherent water resistance

In order to assess the effect laser engraving or cutting could have on the properties of the moulded samples, a probing experiment was conducted in order to see if the laser treated samples were more resistant to water than the untreated ones. This experiment involved cutting out small $20\,\mathrm{mm}$ x $10\,\mathrm{mm}$ pieces from the moulded samples and weighing them before and after being submerged in water. This experiment did, however, have one major issue. The only proper samples available at this time were the samples from the MFC-reinforced moulding experiment, meaning that there were eight samples divided evenly between TMP and Kraft with half of them having a 2% MFC content. The problem was that the TMP samples had not been marked properly and it was unknown which of them the MFC had been added to. The experiment was still conducted but due to the uncertainties of the samples and the fairly inconclusive results the experiment will not be covered in detail here but can be found in its entirety Appendix C. This experiment did however also include a separate test to assess the difference in the inherent water resistance of the full size TMP and Kraft samples. This was done simply by weighing each sample and then submerging it in water for $30\,\mathrm{s}$ and then leaving it on a piece

of paper towel for $10\,\mathrm{s}$ before it was weighed again. The paper towel was there to ensure that there was no excess water on the surface that affects the final weight. These numbers were then used to calculate how much water had been absorbed by the sample. This test yielded some interesting results which are covered in Section 4.5 together with a short summary of the potential of the laser.

Chapter 4

Results and discussions

4.1 Initial tensile test results

The initial tensile tests of the paper sheets provided quite a lot of information to work with. The hypothesis was that the Kraft fibres would come out as the strongest with TMP in second and the recycled fibres would be the weakest of the three. This was however not the case. Figure 4.1 shows a plot of all 27 samples that were tested, nine of each fibre type. There was a clear separation between the three fibre types with TMP at the top, then recycled fibres in the middle and the Kraft fibres at the bottom. Table 4.1 shows the data printed from the tensile test rig. The data presented in this table were calculated from the peak values of the nine samples tested for each fibre type. Moreover, Table 4.2 shows the average thickness of the different sheets tested.

For the TMP and recycled fibres, the process of estimating the elongation along the plot was a fairly easy process due to the samples showing very clear tensile strength peaks and a linear estimation of the elongation from 0 to the maximum elongation reached was accurate within 0.4%. For the Kraft fibres, this was unfortunately not the case. Due to the flatter curve of the Kraft fibres, it was almost impossible to match the peak elongation values to their corresponding force since there were multiple possible matches along the curve. The TMP and recycled samples had very clear peak force values that allowed force and elongation to be matched up. Because of this, Figure 4.1 does not have any specific numbers for the strain on the x-axis due to how inaccurate those numbers would be. The x-axis still provides a reasonable visual representation of the difference in strain between the different samples tested.

Both the values in Table 4.1 and the plots in Figure 4.1 showed surprisingly low numbers for the Kraft samples. Not only was the peak force applied to the Kraft samples significantly lower but this difference was amplified when the greater thickness of the Kraft samples was taken into account, resulting in a very low peak stress. The Kraft samples did, however, have a much higher degree of elongation compared to the TMP in particular. The TMP samples were able to withstand a higher peak force but did not elongate a lot and snapped off completely. The Kraft samples, on the other hand, had a much flatter curve where the samples were able to be exposed to the peak pressure for a longer time before breaking completely. This flat curve characteristic is known as a perfectly plastic behaviour, meaning that the strain is increasing without any change in the stress. This implies that despite the low peak values, the Kraft fibres showed a potential of being quite resilient to stress and should be able to maintain more of their

Table 4.1: Initial tensile test values

Parameter	TMP	Recycled	Kraft
Force (avg) [N]	146.5	92.1	42.4
Force (min) [N]	122.1	77.3	38.1
Force (max) [N]	189.2	101.9	46.3
Elongation (avg) [mm]	1.54	2.49	2.40
Elongation (min) [mm]	1.26	1.45	1.70
Elongation (max) [mm]	1.87	3.61	3.23
Tensile Strength (avg)[kN/m]	9.77	6.14	2.82
Tensile Stiffness (avg) [kN/m]	1228.2	829.2	413.4

Table 4.2: Sheet thickness

Sheet	Sheet 1-4 average thickness [µm]	Average of sheet series [µm]
TMP	335, 370, 373, 358	359
Recycled	328, 297, 336, 364	331
Kraft	368, 434, 459, 427	422

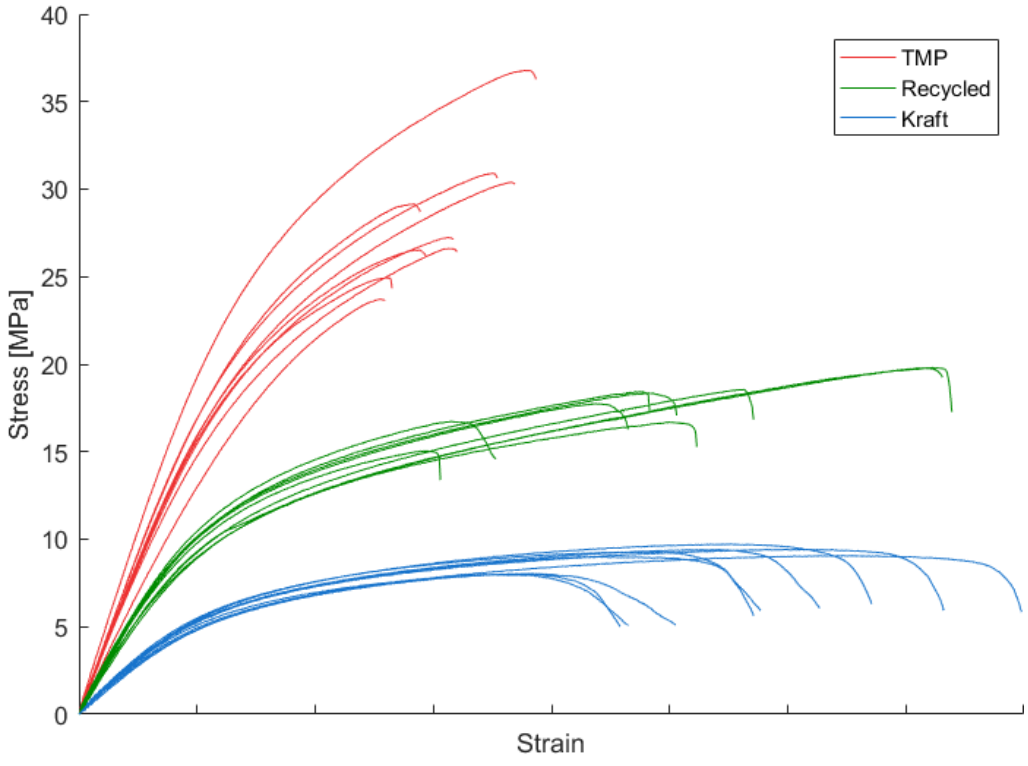


Figure 4.1: TMP, Recycled and Kraft stress-strain curves.

mechanical properties when deformed. The recycled samples were a bit of a middle point here showing a blend of the characteristics of both TMP and Kraft.

It was assumed that these underwhelming results from the Kraft samples could be the result of a lack of fibre activation. Fibre activation is the process of beating fibres in order to increase the flexibility and thus the inter-fibre bonding. This is often done by putting the fibres through a PFI mill as a part of the preparation process. This would activate the fibres and the hypothesis was that this would increase the strength of the Kraft samples significantly. It was decided to create some new Kraft paper sheets but with the PFI mill activation process added to the fibre preparation step. The PFI Mill requires 300g of total mass for each run with 10 wt% of dry fibres. The raw Kraft sheets were 90% dry fibres and thus 33.3g of Kraft sheet fibres were measured and disintegrated. After disintegration, the water was drained off and the remaining pulp was weighed. If the total weight was less than 300g, water was added until the 300g point was reached. The pulp was then spread inside the PFI Mill chamber and the mill was run for a total of 3500 revolutions with a load of 3.33 ± 0.10 $\mathrm{N/mm}$. Once the activation was finished the fibres were disintegrated again before they were ready to use. As mentioned in section 3.4, it was desirable to create at least four sheets from each batch. 30g of dry fibres was however not enough to create four sheets at a sheet weight of 7.6g so a second batch of activated fibres was prepared for a total of 60g, which was enough to create seven 7.6g sheets of paper.

Already during the sheeting process, it became clear that the activation in the PFI mill had made a difference to the fibres. The surface of the sheets was significantly smoother with less loose fibres on the surface. After heat-pressing, the thickness and roughness were measured for all seven sheets and compared to the first round of Kraft sheets that were made. Table 4.3 shows the comparison between the average thickness and roughness of the activated and non-activated Kraft sheets. These numbers showed some very clear differences between the activated and the non-activated sheets. The activated sheets were noticeably thinner and rougher on average. The impact roughness has on the mechanical properties is unknown but thinner sheets could imply a more compact and interlocked fibre structure. None of the seven sheets showed any significant flaws or weaknesses compared to the others, so sheet 1, 2 and 3 were chosen to be tested and compared to the initial Kraft tensile samples. As with the previous tensile test, three samples were stamped from each sheet for a total of nine samples. Figure 4.2 shows the resulting plots comparing the activated and non-activated Kraft samples. The activated fibres showed greatly increased mechanical properties compared to their non-activated counterparts. Not only was the peak stress increased by an impressive amount but the elongation was also significantly better. This was more in line with the hypothesis regarding the mechanical properties of Kraft fibres and showed the importance of proper fibre preparation. To ensure that the low numbers from the non-activated fibres were not just caused by a mistake or inaccuracy in the sheeting process, a new batch of non-activated Kraft sheets were made and tested to confirm whether the activation process was the cause of the increase in strength. These new non-activated sheets showed very similar numbers to the initial set both in terms of general properties and tensile strength. All batches tested came from the same raw material with the same preparation process except for the batch that went through the activation step. This led to the conclusion that activation of the Kraft fibres is a very important step to ensure that the Kraft fibres reach their potential in terms of mechanical properties.

Figure 4.3 shows a new comparison between TMP, recycled and activated Kraft samples. This graph shows a very different picture than what Figure 4.1 projects. The test data for all the samples can be seen in Table 4.4 which is also in line with the conclusions made about the

Table 4.3: Kraft sheet property comparison

Sheets	**Avg Thickness** [µm]	**Avg Roughness** [µm]
Kraft	422	9.2
Activated Kraft	313	11.5

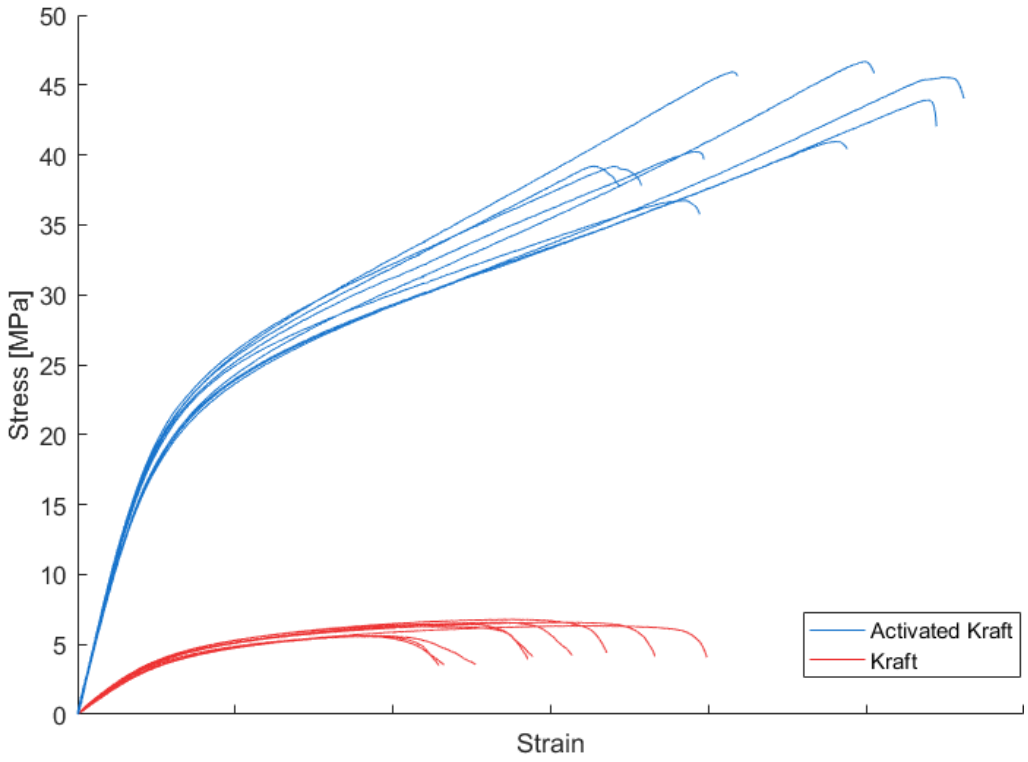


Figure 4.2: Activated and non-activated Kraft stress-strain curves.

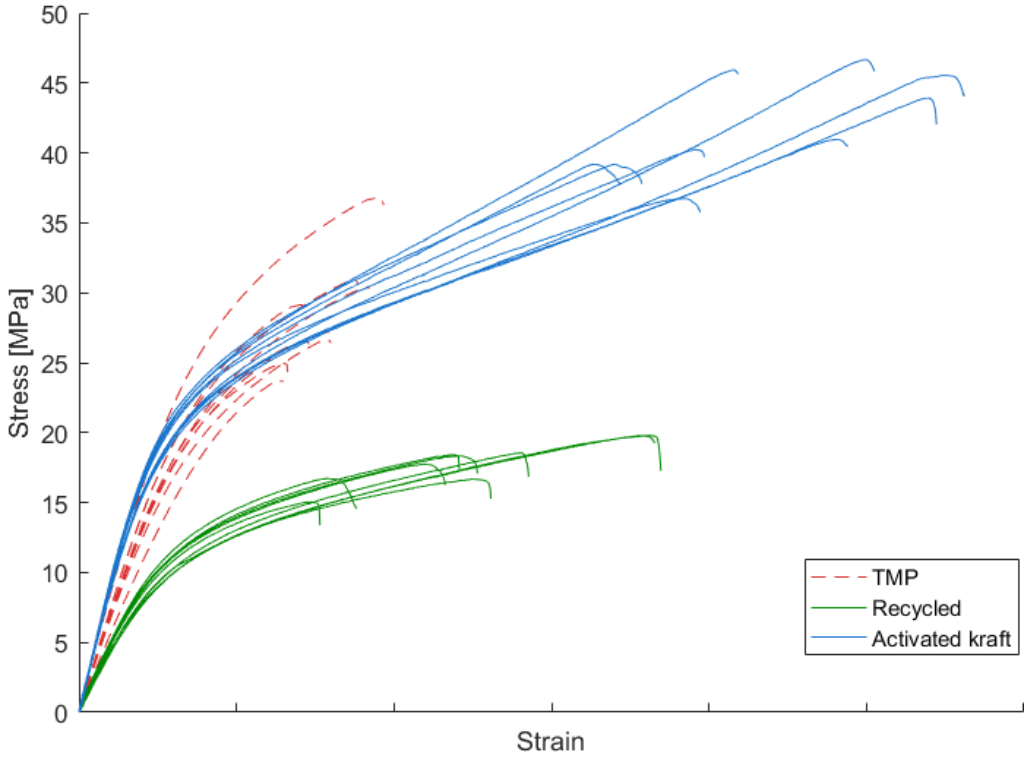


Figure 4.3: TMP, Recycled and Activated kraft stress-strain curves.

importance of the fibre activation for Kraft fibres. The force applied to the Kraft fibres was not only greatly exceeding that of the TMP but the elongation was much greater. The TMP and Kraft fibres show very similar elastic phases in the beginning but when the TMP broke, the Kraft samples continued to elongate and withstand the increased force. Looking at the nature of the fibres once they broke there were clear signs of behaviours in line with the tensile test data. The TMP samples had a very dense and brittle breaking point compared to the Kraft fibres which had a much more ductile behaviour with more fibres that seemed to have been intertwined before the break. This can be seen in Figure 4.4.

Table 4.4: Activated Kraft tensile test values

Parameter	TMP	Recycled	Activated kraft
Force (avg) [N]	146.5	92.1	200.6
Force (min) [N]	122.1	77.3	175.3
Force (max) [N]	189.2	101.9	222.7
Elongation (avg) [mm]	1.54	2.49	4.34
Elongation (min) [mm]	1.26	1.45	3.26
Elongation (max) [mm]	1.87	3.61	5.46
Tensile Strength (avg)[kN/m]	9.77	6.14	13.37
Tensile Stiffness (avg) [kN/m]	1228.2	829.2	1513.5

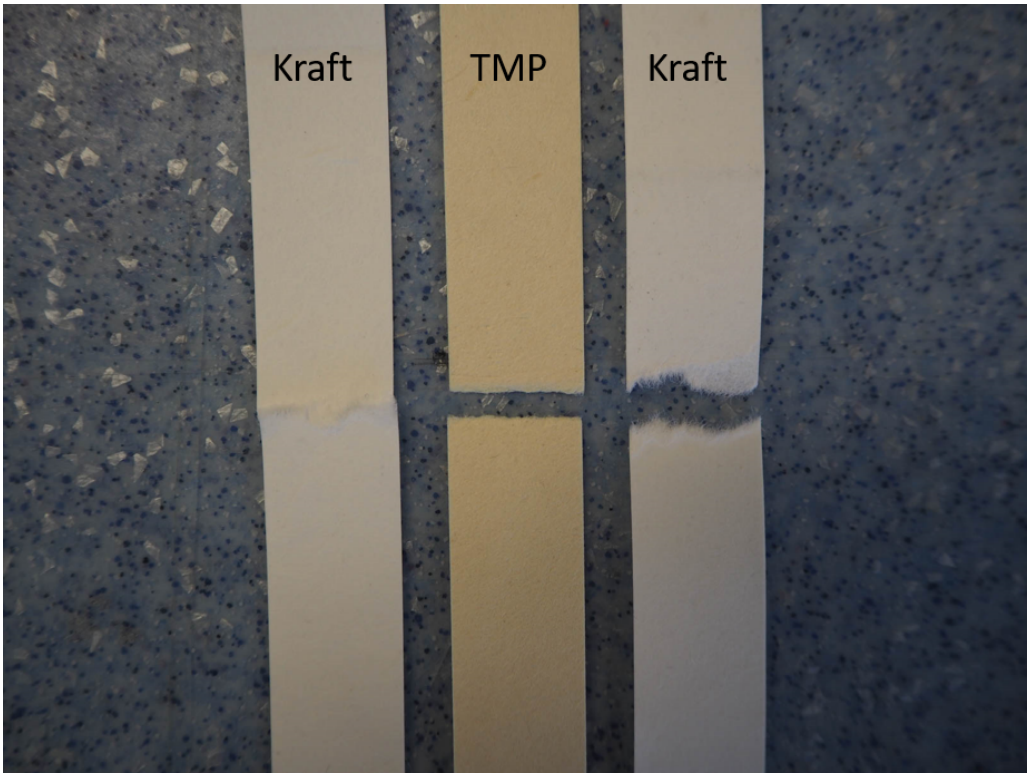


Figure 4.4: Visual comparison of tensile tested TMP and Kraft sheets.

4.2 Moulded sample results

The moulding of the tensile samples had two main goals. First, to explore the possibilities and limitations of this type of thermomoulding. Second, to obtain tensile test data to evaluate the behaviour and properties of the moulded material and hopefully be able to evaluate it in comparison to plastic samples. Section 3.6 already covered most of the issues that were encountered with the moulding of the pulp. The greatest issue was the pockets of water that appeared in some of the samples. The implications these pockets had on the strength of the material is unknown at this time but they had a detrimental impact on the visual quality of the sample. Depending on the future application of the material, this could be an issue. Testing and calibration did, however, show that these issues could be fixed by adjusting the moulding parameters and a future process should be able to adjust both moulding parameters and fibre configurations in order to achieve a satisfactory product.

N.B. It was discovered that the data-acquisition frequency during the tensile tests of the moulded samples was lower for one of the series due to an operator error. This should however not have any implications on the results as the data-acquisition frequency was still high enough to draw the curves properly.

The tensile test data showed some quite interesting characteristics. Figure 4.5 and 4.6 show the stress-strain curves of the moulded samples in comparison to their paper sheet counterparts. In addition, Figure 4.7 and 4.8 are boxplots based on the peak tensile strength of the samples tested. The ultimate tensile strength for the Kraft samples only showed a slight increase in the moulded samples while the TMP samples saw quite a significant increase compared to their paper sheet counterparts. The fact that TMP samples had been breaking in a more brittle fashion than the Kraft samples could be a reason why the thicker moulded samples showed a much greater strength than the sheets. The sheets were very thin and with a brittle sample like the TMP it could perhaps be assumed that such a thin and brittle sample could not reach the full potential and that an increase in thickness would thus have a greater impact than a similar thickness increase in a sample that even in sheet form showed quite high numbers. It is worth noting that the paper sheets and the moulded samples were tested according to very different standards, especially in terms of test speed. The sheets were tested at a rate of $100\,\mathrm{mm/min}$ while the moulded samples were tested as a plastic sample at $5\,\mathrm{mm/min}$. The implications of this were that the different characteristics of moulded samples and paper sheets could be an effect of the speed of which the force was applied and not necessarily of a difference in the fibres. The answer to this will lie in future research.

When considering the box plots there was, despite the difference in numbers, a clear similarity in the general size of the boxes between the moulded samples and the paper sheets. The implications of this were that it seemed like the Kraft samples were more inclined to have a greater spread in their peak values. Similarly, the TMP had peak values much closer to each other except for the single outlying value on the paper sheets. This spread can also be seen in the stress-strain plots where the moulded TMP samples have a much tighter and more defined curve clusters. In general, the moulded samples also portrayed a much more linear curve than the paper sheets. The Kraft curves were very linear with the TMP samples showing a bit more of a flattening at the top. The curvature at the beginning could be the result of the grips on the tensile test rig affecting the sample. Mounted strain gauges were not available for the tensile testing so the numbers used for these figures were taken directly from the test rig itself. This means that the initial part of the curves could most likely be disregarded and replaced with a

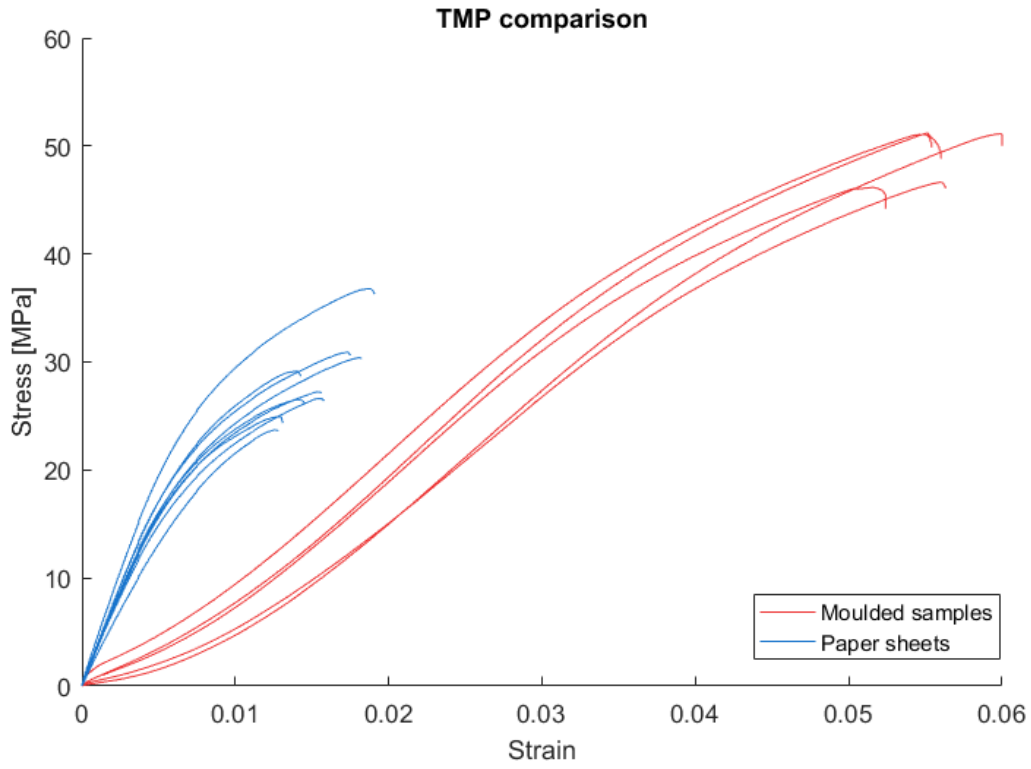


Figure 4.5: Stress-strain curves of paper sheets and moulded samples: TMP

curve regressing linearly from the rest of the curve. This would give a very linear curve with a slight curvature at the top. This curve characteristic could imply that the thicker moulded samples had a much more elastic behaviour compared to the paper sheets which showed a very standard elastic-plastic behaviour. This can, however, not be proved until further tensile tests with loading and unloading are done, which was not within the scope of this thesis. These findings still provide valuable information about the behaviour of moulded samples compared to the paper sheets. Another difference between these curves was that the Young's modulus for the moulded samples was significantly lower than the paper sheets. The paper sheet curves were overlapping heavily in the linear elastic region, meaning that for both the TMP and the Kraft samples there was little variation in modulus and they were around $4\,\mathrm{GPa}$ and $5\,\mathrm{GPa}$ respectively. The moulded samples, on the other hand, showed a greater degree of spread but in general the modulus was around $1\,\mathrm{GPa}$ for TMP and $0.5\,\mathrm{GPa}$ for Kraft. These values were all calculated from values taken from what appears to be the linear elastic region of the curves. The difference in modulus suggests that the paper sheets were stiffer than the moulded samples and thus that moulded samples are more inclined to deform elastically at lower loads. Whether this is a desirable material characteristic depends on the application of the material which has been unspecified during this thesis research. In addition, further improvements to the material will most likely affect this behaviour.

N.B. These curves were all plotted against engineering strain so a quick comparison with true strain was conducted to see if that would change any of the curve characteristics. This was not the case, meaning that these curves were essentially the same when plotted against both engineering strain and true strain.

In Section 4.1 the Kraft samples seemed to have a clear advantage in terms of strength and they boasted significantly longer elongation than the TMP samples. For the moulded samples things

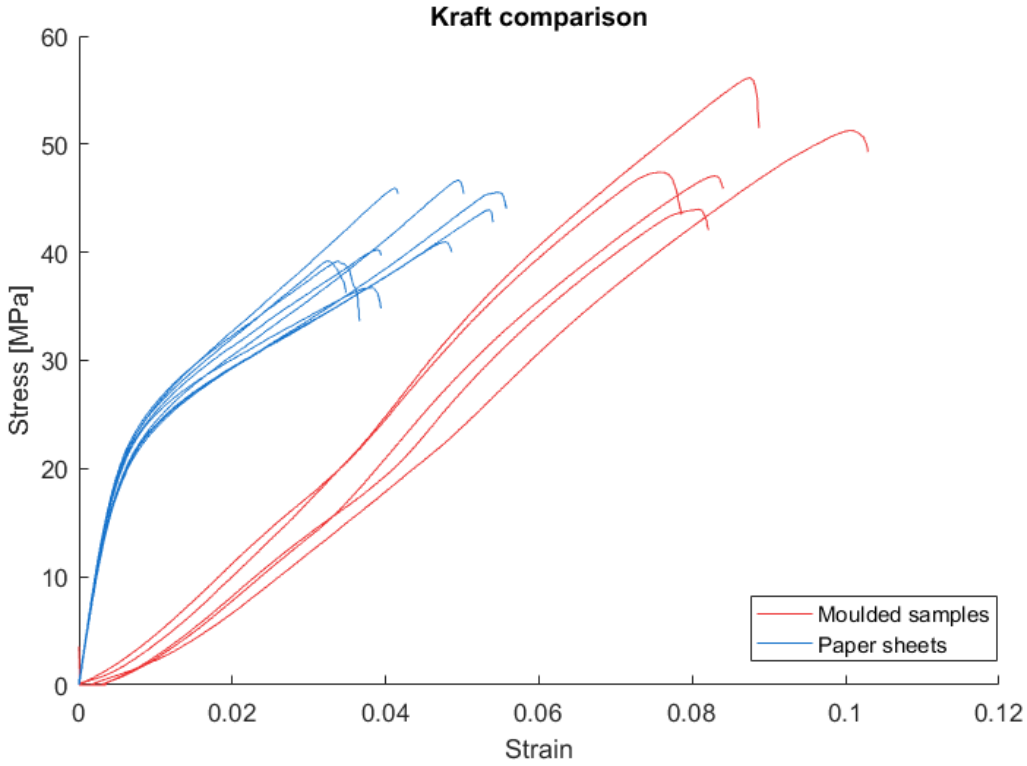


Figure 4.6: Stress-strain curves of paper sheets and moulded samples: Kraft

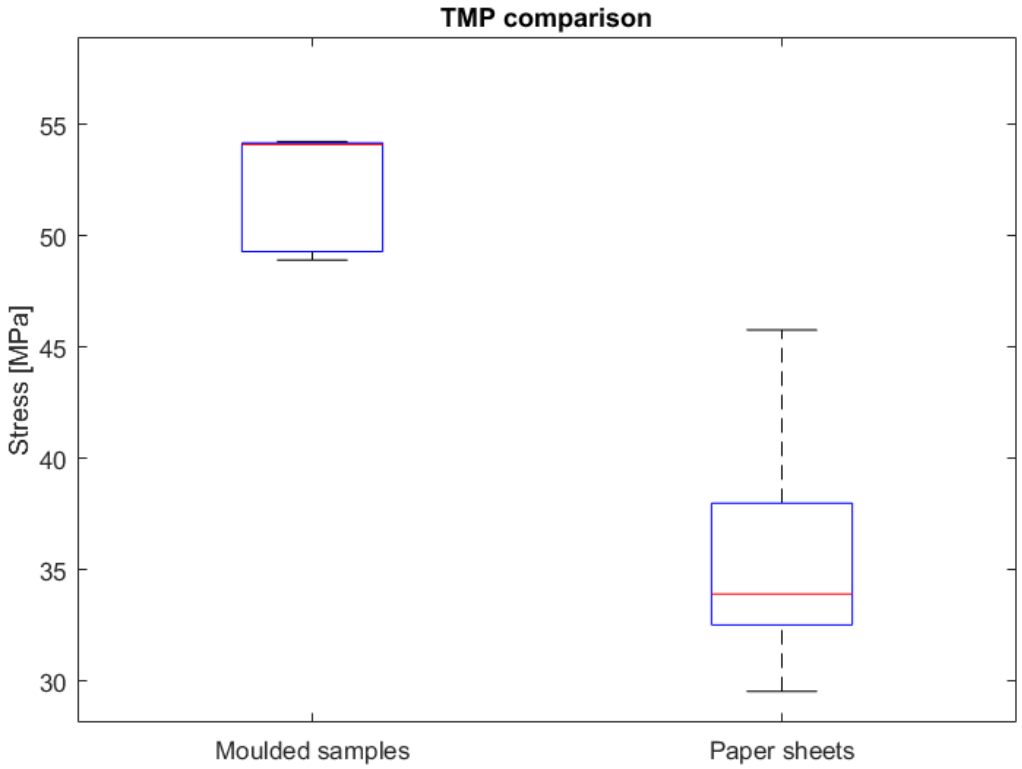


Figure 4.7: Box and whisker plot of paper sheets and moulded samples: TMP

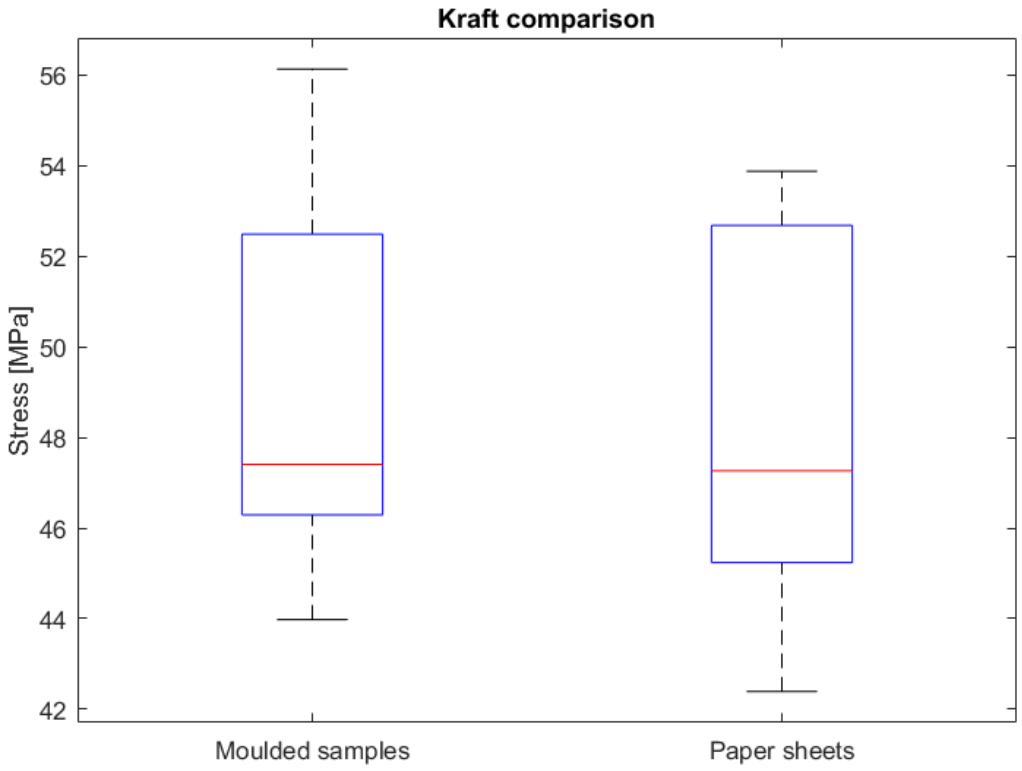


Figure 4.8: Box and whisker plot of paper sheets and moulded samples: Kraft

were looking a bit different. Figure 4.9 shows the stress-strain plots of the TMP and Kraft moulded samples. The elongation of the Kraft samples was still superior but not to the same extent as with the paper sheets. In addition to this, the strength of the TMP was now on a par with the Kraft strength. This could allow for a future material to be made out of either TMP or Kraft depending on the needs of specific applications. The ability to vary the fibre component of the material could prove to be valuable for the future of this material. Furthermore, prior to the proper sample testing, an interesting discovery was made. After the calibration of the mould, there were a number of samples that had been deemed not good enough, mostly due to extensive wet areas and insufficient drying. They had poor surface finishes and were assumed to be weaker structurally. They were used to test the MTS tensile rig to calibrate the settings before testing the proper sample series. Unfortunately, the data from these samples were not saved in the process due to experimentation with the operation software settings. However, the peak tensile forces that were observed during the testing of these ”defect samples” turned out to be similar to what was later seen in the proper sample series. This could imply that water pockets in the samples, when dried out eventually, did not actually decrease the strength but only affected the visual quality and the probability of post-forming instabilities.

One of the most interesting aspects of this testing was to compare the results of these moulded fibre samples to different types of plastic. The plastics chosen for this comparison were the six defined plastics of the common plastic system [66] in addition to ABS and PLA, which fall under the seventh ”other” category of the common plastics. Table 4.5 shows the minimum and maximum Young’s modulus, tensile strength and elongation of these plastics. These numbers were taken from the CES EduPack 2016 database of materials [67]. For some of these plastics, there were several possible production methods and structures that have quite different mechanical properties. Because of this, some of the numbers in this table have quite a large span, especially in terms of elongation. All the numbers were selected from unfilled plastics with no

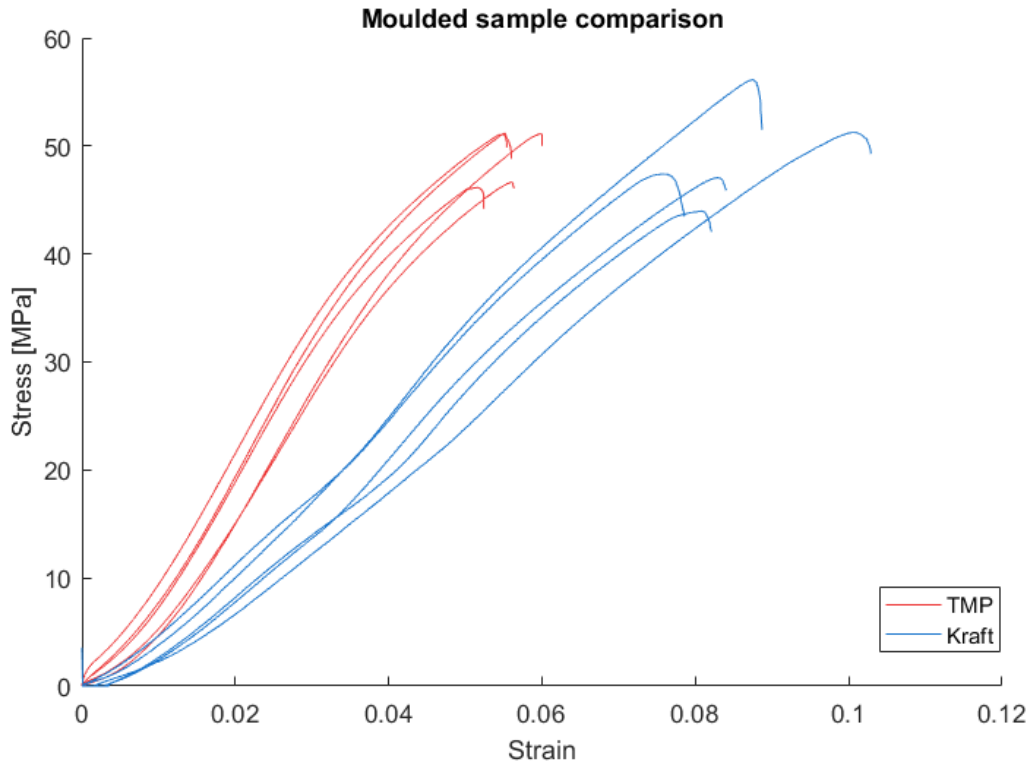


Figure 4.9: Moulded TMP and Kraft stress-strain curves

fibreglass or other additives apart from the polymer itself. Without any specific application in my mind, it was of course quite difficult to make any strong claims about whether or not the moulded pulp material could replace some of these plastics, especially considering the great variety of plastics. These numbers do, however, show quite clearly that despite some great differences, especially in terms of potential for elongation, the strength of the moulded pulp was on a par or above many of these plastics. Comparing this to something like MDF, which has a potential of up to $25.1\,\mathrm{MPa}$ in terms of tensile strength [67], this moulded material seemed to have the properties needed to replace certain plastic in some applications. With further research, the properties could also be tweaked and adapted to different applications.

4.3 MFC-reinforced sheet results

This section will cover the results of the MFC-reinforced sheet experiments which will consist of three main parts. The effect the MFC had on the sheet making process, how MFC affected the tensile test results of the paper sheets and how MFC affected the dewatering of the pulp.

4.3.1 Hot-pressing of reinforced sheets

As mentioned in Section 3.7, adding MFC to the TMP sheets had induced the emergence of rough spots during the hot-pressing step of the process. Figure 4.10 shows a comparison between a TMP sheet and Kraft sheet with the same amount of MFC added. The spot was only present in the TMP sheet and there were no signs of similar issues with the Kraft sheet. As

Table 4.5: Mechanical properties of plastics and moulded paper samples.

Material	**Young's Modulus [**GPa]	**Tensile strength [**MPa**]**	**Elongation**
PET	2.76 - 3.10	65.0 - 75.0	65.0% - 320.0 %
HDPE	1.07 - 1.09	22.0 - 31.0	1120.0% - 1290.0%
PVC	0.26 - 3.41	25.9 - 58.0	4.0% - 258.0%
LDPE	0.17 - 0.28	13.3 - 26.4	100.0% - 650.0%
PP	0.82 - 2.08	16.8 - 49.0	26.0% - 865.0%
PS	1.16 - 3.34	18.3 - 65.0	1.2% - 65.0%
ABS	1.10 - 2.90	27.6 - 55.2	5.0% - 100.0%
PLA	2.40 - 3.60	29.0 - 70.0	2.0% - 20.0%
TMP	1	48.9 - 54.1	5.2% - 6.0%
Kraft	0.5	44.0 - 56.1	7.9% - 10.3%

with the earlier observations of these types of spots, it was believed to be caused by moisture and water vapour not being able to escape due to the density of the fibre structure. Figure 4.11 shows three sheets with 2%, 4% and 6% MFC added respectively and the spots had a similar size and shape for the most part. It was, however, observed that the 6% sheets developed spots that were somewhat darker. The cause of this is still unknown but it could be caused by a change in the opacity of the material due to a denser fibre configuration within the spot owing to the higher MFC-content. These spots could possibly be countered by increasing the temperature during pressing even further, which was the solution to these spots when they were first encountered during the calibration of the pressing process in Section 3.4. However, this could not be done for this particular experiment as it was important to keep the parameters the same as the initial non-reinforced testing. Fortunately, these spots were easy to avoid when punching out the tensile samples and did not have any significant impact on the test results. However, they appeared to be the symptom of a much greater issue, namely that the press used for the hot-pressing did not distribute the force evenly.

This round of sheet-pressing consisted of a total of 12 TMP sheets, meaning that it did not take long until a certain pattern seemed to be emerging. The spots were not only similar in size but they also seemed to appear in the same area of the sheet every single time. The sheets were layered between two metal plates before being inserted into the press and changing the orientation of these plates did nothing to affect the position of the spot. This led to the conclusion that the press itself could possibly be applying more pressure on the upper right corner of its surface. This hypothesis was verified further when the thickness of all the sheets was measured. Similar to earlier sheets, the thickness was measured at five different points: upper left, centre, upper right, lower right, lower left. The numbers obtained from this showed a clear picture. There was quite a lot of variation between the five different points on a single sheet but there was less variation on a specific point across the four different sheets.

Table 4.6 shows the standard deviations of the measured thickness of the TMP sheets with 2% MFC added. The first section of the Table 4.6 lists the deviation between the five data points for

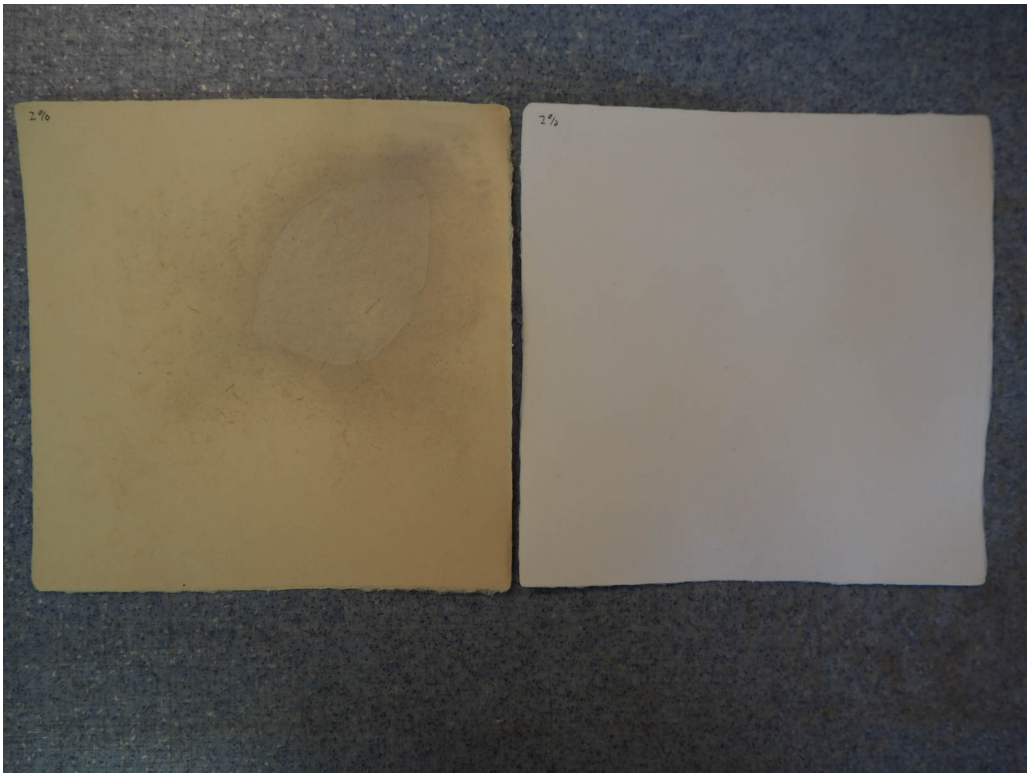


Figure 4.10: MFC-reinforced TMP and Kraft sheets. The TMP sheet has a very clear rough spot that is not present at all in the Kraft sheet.

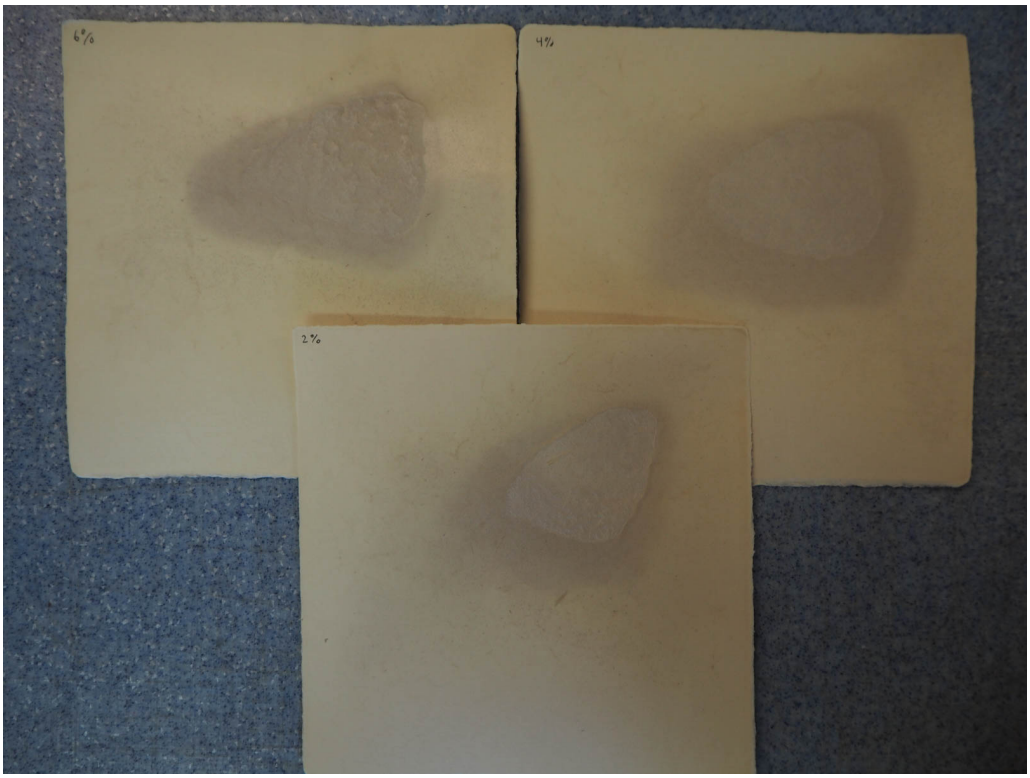


Figure 4.11: MFC-reinforced TMP sheets with 2%, 4% and 6% MFC added.

Table 4.6: Standard deviation of the sheet thickness for TMP sheets with 2% MFC

Standard deviation, in micrometer, of the thickness for a single sheet	
TMP sheet 1	41.0
TMP sheet 2	44.3
TMP sheet 3	37.5
TMP sheet 4	22.7

Standard deviation, in micrometer, of the thickness at specific measured points	
Upper left	6.8
Middle	26.6
Upper right	5.3
Lower right	7.8
lower left	20.7

each sheet, while the second section lists the deviation of each particular measuring point across the four different sheets. As it can be seen, the greatest deviation for a specific measuring point was comparable to the smallest deviation within a single sheet. This supported the theory that the press had an uneven pressure distribution, meaning that it would produce sheets with big variations in thickness across the sheet. The positive side was that even though it has uneven pressure, the pressure was at least consistent from one sheet to the next. This uneven pressure also seemed to be the cause, or at least one of the causes, of the spots seen on the sheets.

Table 4.7 shows the average thickness of the different sheet areas and shows that the thickness was significantly lower in the upper right corner of the sheets where the spots also appeared. Lower thickness most likely means higher pressure, which would explain why the spots started reappearing. The spots were not a problem for the non-reinforced TMP-sheets at the temperature used but when MFC was added, the combination of the more dense structure of the sheets and the higher pressure in that corner once again caused these spots to reappear. When looking at the other thicknesses, the highest average is in the lower left corner, diagonally opposite of the thinnest corner with the spots. The two other corners are fairly similar and the centre point is somewhere in the middle between the thinnest and thickest corners. The numbers seem to indicate that the press was applying a higher pressure on the upper right corner with a resulting lower pressure on the lower left while keeping the two other corners fairly even. This could be adjusted for future research but during this thesis research, the issue was discovered at a stage when it was too late to make any modifications. Fortunately, the implications of this issue were manageable and did not affect the tensile results in any significant way since every tensile sample was individually measured in terms of thickness to allow for proper calculations.

Table 4.7: Average sheet thickness for TMP sheets with 2% MFC

Average thickness, in micrometer, of each measured point across four sheets	
Upper left	344.5
Middle	374.5
Upper right	302.3
Lower right	351.3
lower left	396.0

4.3.2 Tensile test results

Figure 4.12 and 4.13 show the stress strain curves of TMP and Kraft sheets respectively with increasing amounts of MFC added. These curves do have a significant amount of overlap in the top end, making it difficult to draw any conclusions from them. Still, these plots provide some data regarding the elastic behaviour of these samples. The greatest degree of overlap was observed in the linear elastic region. The significance of this region is that it governs the Young's modulus of the material. In other words, the addition of MFC seems to have no significant effect on the modulus. There seems to be a slight increase from the samples with 0% MFC, especially in the Kraft samples, but this difference is quite small and the difference seen between e.g. 2% and 4% samples does not seem to be greater than the variation you would expect to see from a series of nine samples of the same type.

In terms of general strength, the boxplots in Figure 4.14 and 4.15 provide a picture that is easier to evaluate. These plots have been plotted from the ultimate tensile strength of each series, meaning that each box has a total of nine data points. Although there are some variations between TMP and Kraft, the general spread of the boxes show a high degree of similarity and similar conclusions may be drawn. Namely that the addition of MFC improves the strength of the material, especially in the case of the Kraft sheets. The greater strength increase for the Kraft sheets could be caused by the fact that the MFC used has been extracted from Kraft pulp which might cause the MFC to bind more easily to Kraft pulp.

Despite the clear increase in strength from no MFC to some MFC added, the variations based on the amount of MFC added was not as clear. There was somewhat of a trend towards higher strength in 6% samples compared to 2%, but 4% was underperforming in both cases compared to what was expected. This could be due to errors made in the production of the sheets or other issues with the process. However, the data still point to the fact that MFC will help increase the strength but that the effect of an increased MFC content would most likely not provide significant gains. This is especially true when considering the issues of dewatering related to MFC. The issues of dewatering the MFC-reinforced sheets is covered in Section 4.3.3.

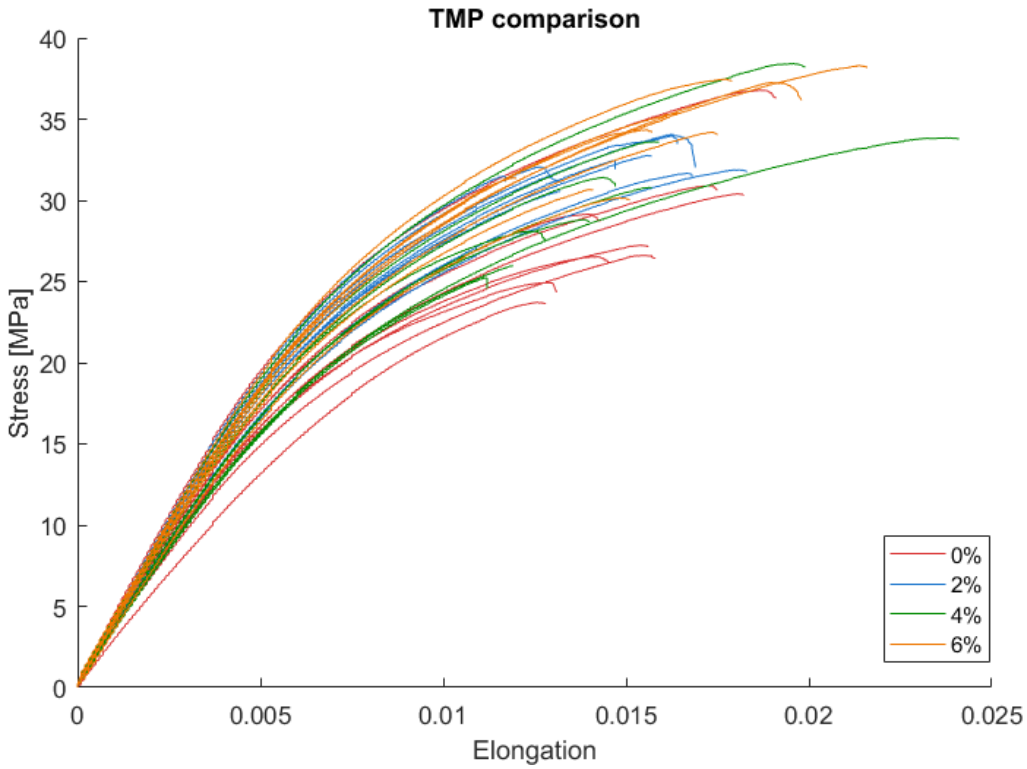


Figure 4.12: Stress-strain curves of TMP paper sheets with varying degrees of MFC-content.

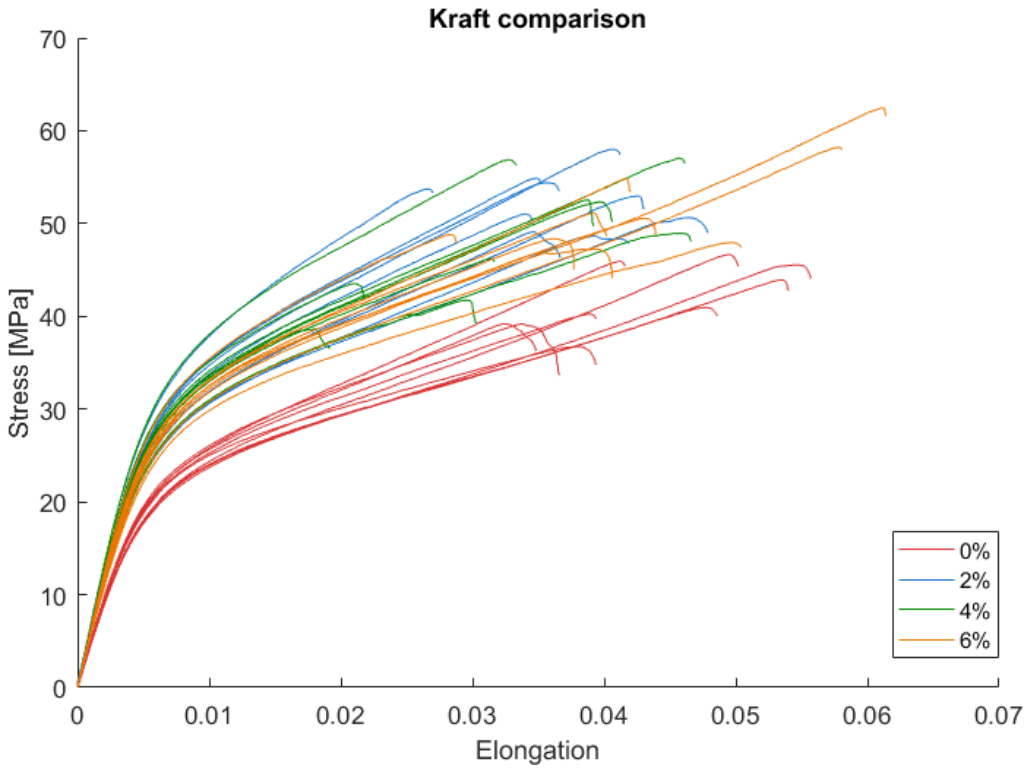


Figure 4.13: Stress-strain curves of Kraft paper sheets with varying degrees of MFC-content.

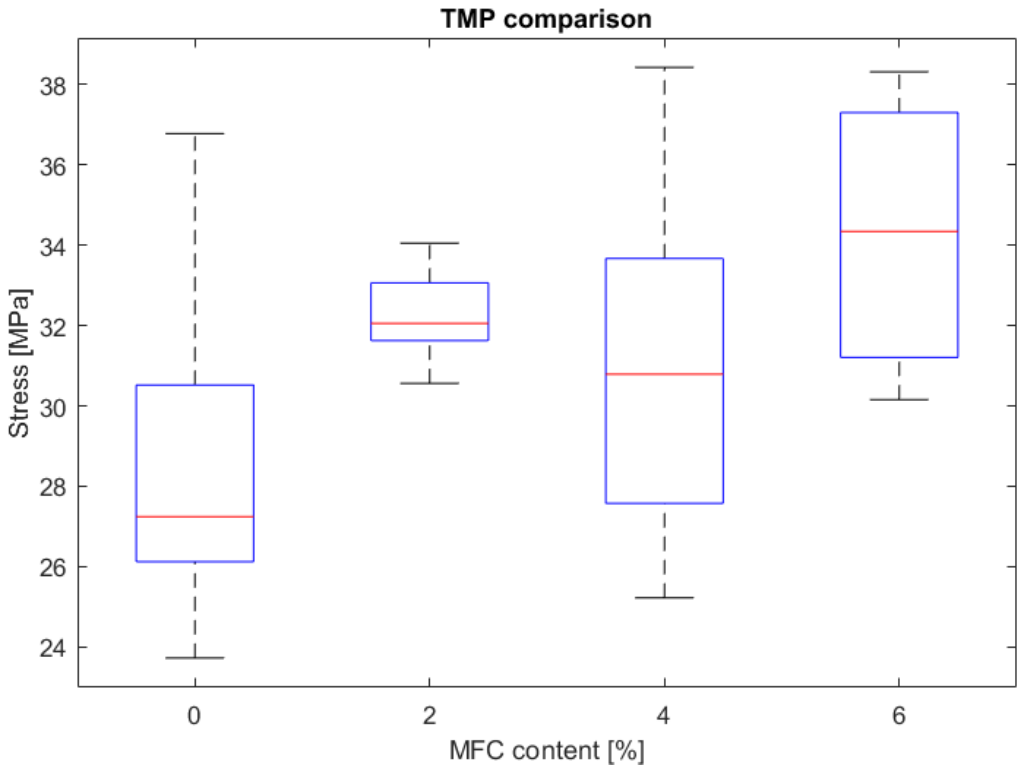


Figure 4.14: Box and whisker plot of TMP paper sheets with varying degrees of MFC-content.

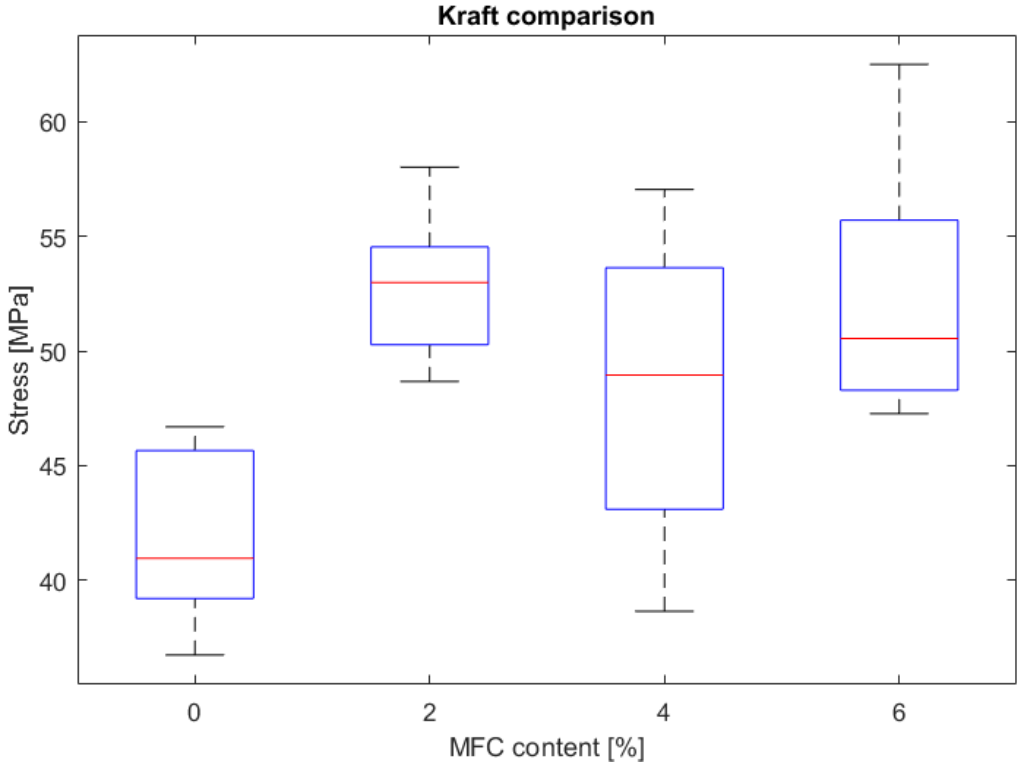


Figure 4.15: Box and whisker plot of Kraft paper sheets with varying degrees of MFC-content.

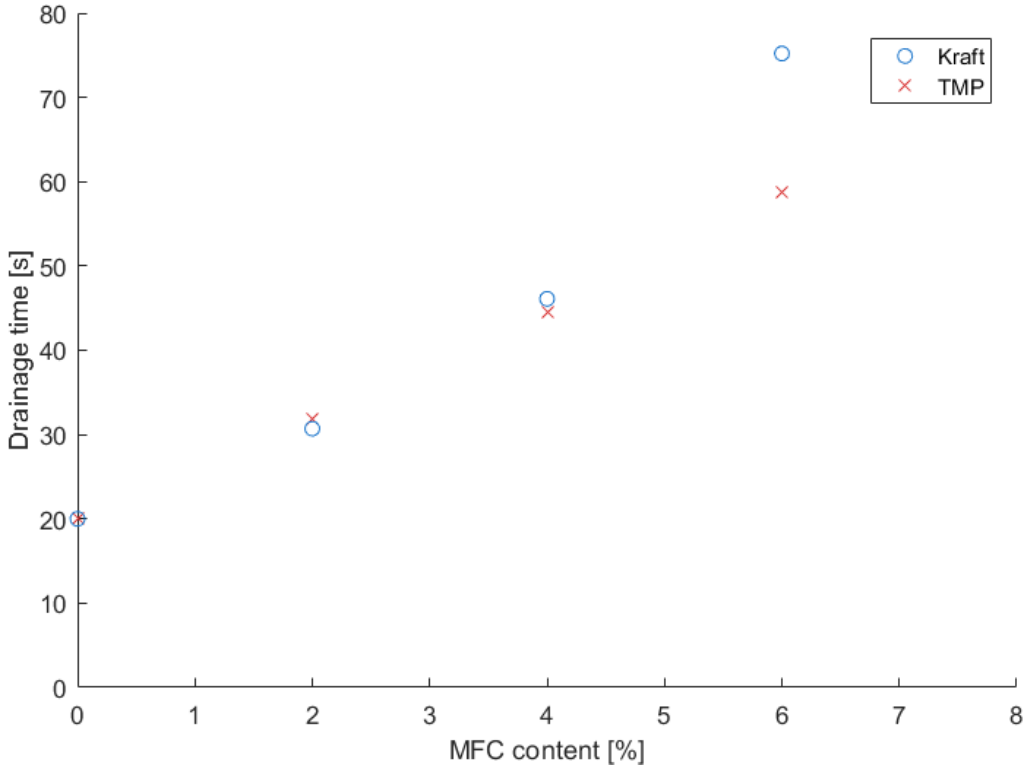


Figure 4.16: Paper sheet drainage times plotted against MFC content

4.3.3 Dewatering

As it was pointed out in Section 2.3.3, research shows that when MFC is added to a pulp, it could interfere with the ability to dewater or drain the pulp. This is of great concern to mass production processes as it directly affects the lead time on production. In this thesis research, the sheet former used for making the paper sheets had a built in timer that showed the exact time used to drain each sheet. Therefore, data could be collected effortlessly to assess the effect of the MFC. Figure 4.16 shows the measured drainage time of the paper sheets plotted against MFC content. This data shows a clear increase in drainage time as the MFC content was increased. This is especially true for the Kraft pulp which sees an almost exponential increase past 4% MFC. These numbers are in line with what was expected and are part of the reasons why high MFC content pulp has been difficult to make viable for mass production. The strength gained is desirable but the implication it has on the production process is just too great. For this particular material, it is difficult to envision a production process that would work for these higher MFC contents since filtering will be an important part of the process. Adding a smaller amount of MFC might be effective but would have to be balanced so that the strength gained is not overshadowed by dewatering issues.

4.4 MFC-reinforced moulding

This section will cover the results and observations of the attempts at moulding MFC-reinforced TMP and Kraft pulp. The focus of this was on the dewatering of the pulp and the individual moulding results of the TMP and Kraft samples. These samples were not to be tensile tested as the effect of MFC had already been established from the paper sheet tensile tests. The experiment was rather centred around observing the effect MFC had on the moulding of the samples. Already in Section 4.3 there were signs of changes in the behaviour of the sheets during hot pressing and similar changes were expected to be seen in the moulding as well. Especially considering the high-pressure environment that develops inside the mould. The hypothesis was that the addition of MFC would cause new issues with wet spots due to a denser sample and that there would be a greater chance of samples with defects.

4.4.1 Dewatering

Just as with the reinforced sheets, the filtering times of the moulded samples were monitored in order to assess the effect MFC had on dewatering. The sheet-tests have already shown numbers that support the hypothesis that MFC increases dewatering times. The moulding-filtering was, however, a somewhat different dewatering process, during which the thickness of the samples played an important role. Figure 4.17 shows a scatter plot of the time, in seconds, that it took for each sample to be fully drained out. Unlike the sheet-maker, this process did not have an automatic stop and built in timer so the timer was stopped manually when a visual inspection of the filtered sample deemed the filtering to be completed. This was determined by looking at the surface of the sample and assessing the degree of moisture still present. Due to this, the plotted times have a margin of error of $\pm$ 20 s which is relatively low considering that the longest filtering time observed was 980 s. This plot shows very much the same trend as Figure 4.16, namely that the MFC has a clear impact on the filtering times. One difference, however, is that these numbers show a much greater difference between TMP and Kraft even before any MFC was added. For the paper sheets, the drainage times are quite similar up until 6% MFC content but the filtering time nearly doubled from TMP to Kraft at 0% MFC. This suggests that the greater thickness of these samples could be causing greater issues for the drainage of Kraft pulp. Based on observations of the behaviour of the Kraft pulp during filtering, it seems like the Kraft pulp absorbs considerably more water than the TMP and creates a much denser filtered sample which in turn increases the filtering time. This could always be alleviated by creating a more efficient filtering process with a properly sealed vacuum for instance. The setup used for the filtering for this experiment was a simple prototype with much room for improvement. In addition to this, the Kraft pulp also seemed to be more inconsistent in regards to filtering times. The TMP samples were very similar both with and without MFC while the Kraft samples showed some variation already at 0% and considerably more at 2%.

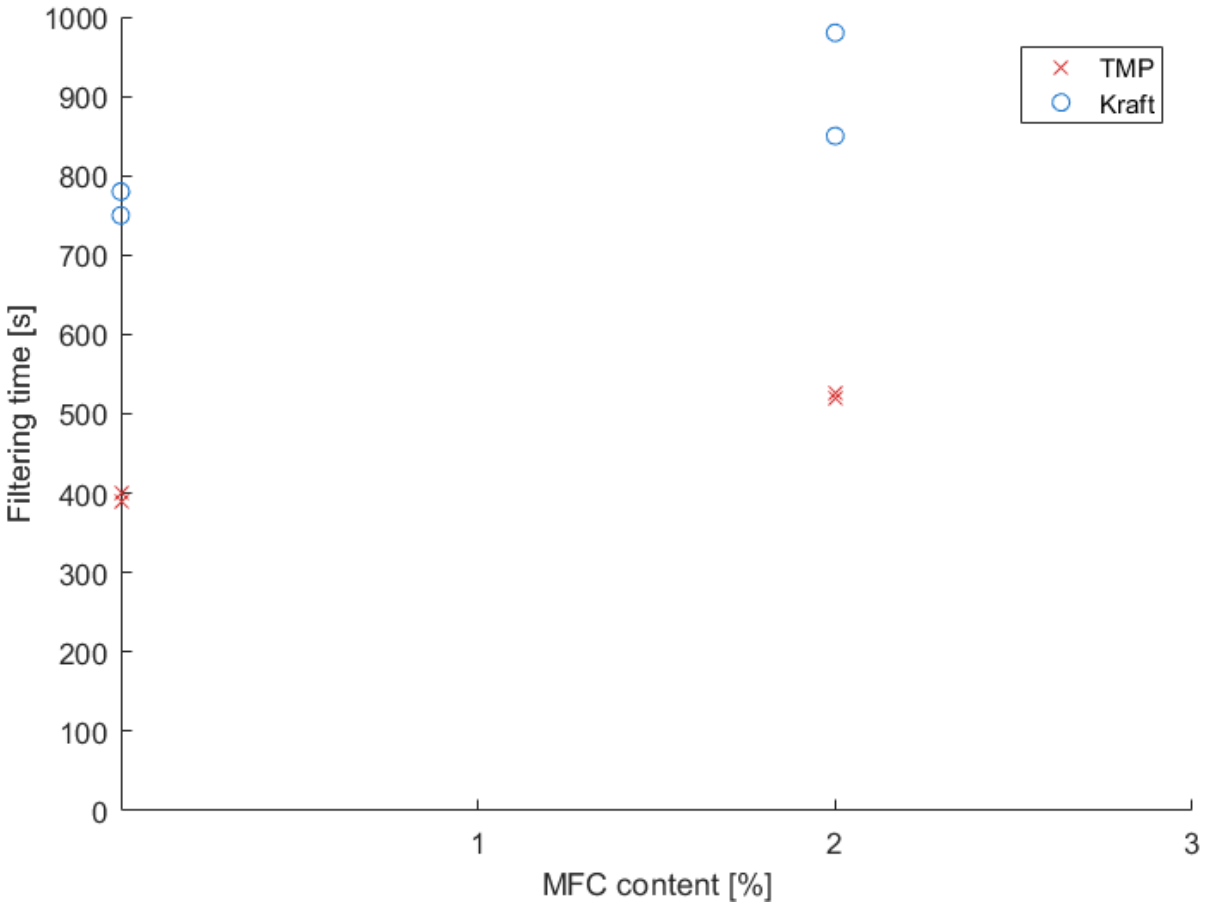


Figure 4.17: Mould filtering times plotted against MFC content

4.4.2 TMP

The TMP samples were the first to be moulded and it was expected that the samples would show some signs of wet spots or other defects that had been seen during the calibration of the TMP moulding. Surprisingly, this was not the case and the samples with 2% MFC added came out of the mould with the same surface finish and thickness as the samples with no added MFC. This was quite surprising and when looking at the samples there was no visual way of telling which one had MFC added. This could be due to the drainage process leaving the TMP in a state in which there was a certain headroom for adding smaller fibres to the structure without compromising the density. In general, the TMP showed signs of absorbing quite a bit less water than the Kraft pulp during filtering and moulding so when considering everything it was understandable how adding 2% MFC had no significant impact on the moulding. The MFC did still affect the dewatering ability of the pulp, meaning that pushing the MFC content any higher would not necessarily lead to a desirable outcome.

4.4.3 Kraft

Unlike the TMP samples, the Kraft samples did show some clear signs of the MFC affecting the moulding process as well. Figure 4.18 shows the two samples with 2% MFC added. They showed very clear signs of not only smaller wet pockets, but a bigger affected area that covered the entire narrow section. Figure 4.19 shows signs of even further wet pockets inside this already affected area. These sections were the result of water not being able to fully escape during drying and the samples showed clear signs of a lot of retained moisture when they were taken out of the mould. They had to air dry after moulding to get rid of the excess moisture which in turn created a very dry and wrinkled surface that even further deteriorated the visual quality of the sample. These samples also showed a tendency that the centre of the sample would be thinner than the edges. This was seen in some of the previous Kraft samples but it was present to a much greater extent in the MFC-reinforced Kraft samples. This created a concave cross section which is less desirable and it makes the calculation of mechanical properties more difficult. The retention of water inside the sample seemed to be the cause of all of these issues and once again this could perhaps be alleviated with a more efficient filtering process. If most of the water could be drained off before moulding is done, it would most likely be easier to compress the sample and prevent defects like the ones that were observed in these experiments. The bottom line at this point is still that adding MFC to the Kraft pulp increases the probability of these undesirable defects significantly.

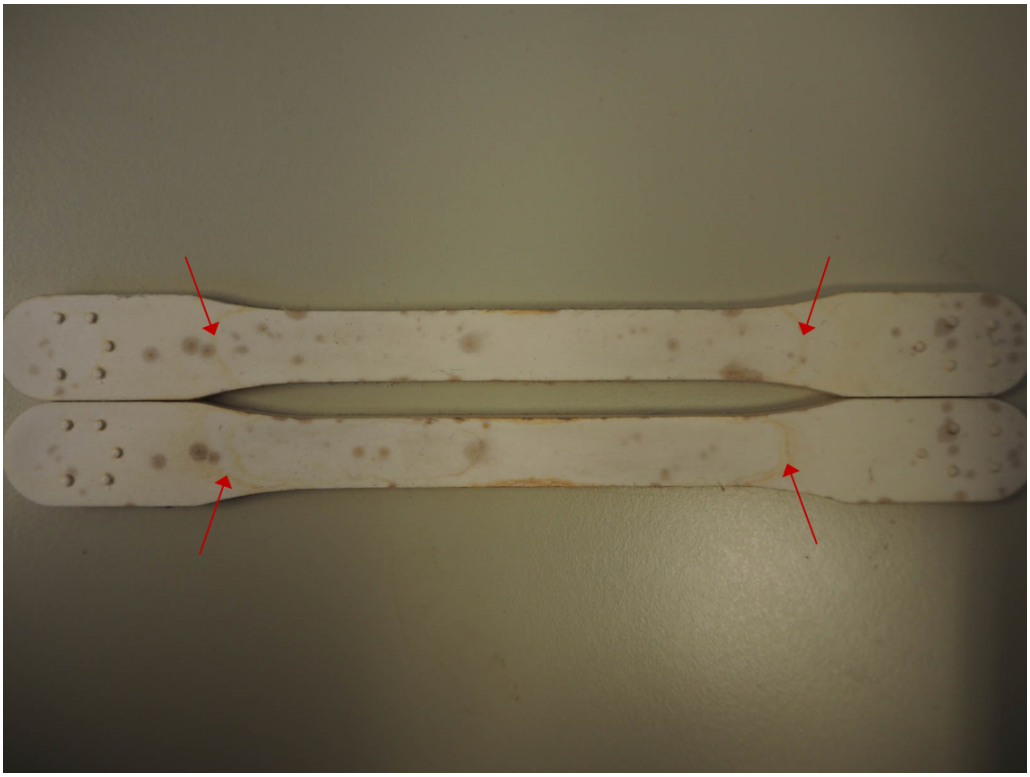


Figure 4.18: Moulded Kraft samples with 2% MFC content. The arrows point to the border between the gripping sections and the water-affected middle section.

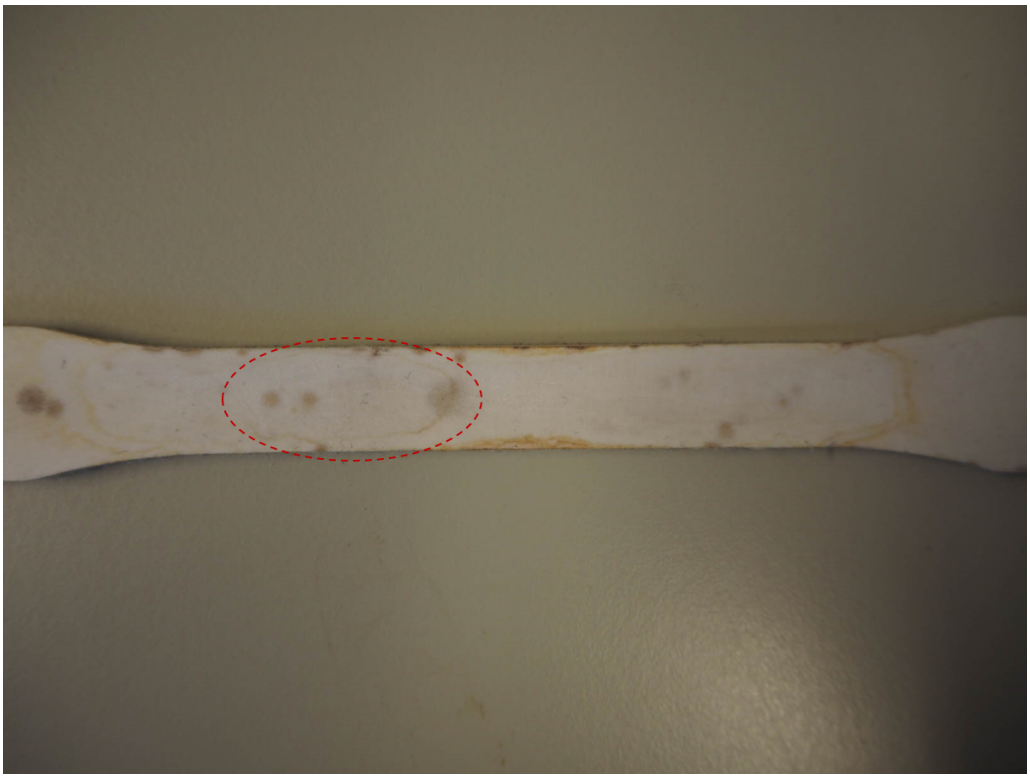


Figure 4.19: Detail picture of moulded Kraft sample with 2% MFC content. The dotted circle shows the wet pocket that has emerged inside the already affected narrow section.

4.4.4 Summary

For this experiment, it was only the Kraft samples that showed an increase in defects when MFC was added to the pulp. It was, however, safe to assume that with an increased MFC content in the TMP samples, a similar increase in defects could have been seen as well, considering the history of defects in the TMP during calibration of the moulding. The dewatering issues were on the other hand still very present for both pulp types, which was similar to what was seen with the MFC-reinforced paper sheets. These issues with dewatering and the increased presence of defects were the reasons why it was deemed unnecessary to do more moulding tests with more MFC added. It was already clear that for the current state of this moulding process the addition of 2% MFC would have enough of an impact for both better and for worse. The TMP did show the potential to handle a higher MFC-content but the benefit from this was greatly outweighed by the increase in dewatering times.

4.5 Laser treatment and water resistance results

Although the probing experiment with the laser treatment had some sample issues and some fairly inconclusive results, the laser treated samples did show a very clear change in the properties of the edge once it was cut. It did not seem like this had any implications for water resistance but it did seem like the edge had hardened somewhat. This could have implications on the mechanical properties of the sample and further research might be able to provide more conclusive results. The complete results of this experiment can be found in Appendix C. In terms of the inherent water resistance, the experiment conducted with the full-size samples showed a couple of interesting things. Firstly, the TMP and Kraft samples in question saw an increase in weight by $23\,\%$ and $34\,\%$ respectively, meaning that the Kraft sample did absorb quite a bit more water than the TMP. In addition to this, the observed behaviour of the samples was also quite different. The Kraft sample became significantly softer and seemed to use more time to dry and regain its properties. After a few hours of drying, the TMP sample was seemingly well on its way back to regaining most of its stiffness while the Kraft sample was still quite soft and wet. This is of course purely anecdotal evidence and further testing is needed to properly evaluate the difference between the inherent water resistance of TMP and Kraft. Overall, this test confirmed that there seems to be a difference there that could have implications on the applications of the fibres that should be worth exploring in the future.

4.6 Further Research

In terms of further research, there is quite a lot that can be done. One of the biggest areas of this would be the exploration and testing of natural additives or coatings that could improve mechanical properties or provide waterproofing without compromising the sustainability or biodegradability of the material. There are already a number of ideas for this which is covered in Section 4.6.1. The need for further mechanical testing is also present, e.g. tensile tests during which the sample is unloaded to assess the elastic properties or other types of tests such as bend tests or fatigue tests. The moulding process itself is also an area that will need to see further improvements. The current iteration of the mould showed a lot of promise for this type of in-mould drying and filtering but it has a long way to go in terms of being fully viable. Some

thoughts on the future of thermomoulding and the possible applications of this material are covered in Section 4.6.2.

4.6.1 Additives, treatments and waterproofing

In terms of waterproofing and modifications of the mechanical properties, there are existing ideas that could be worth exploring in the future. The need for some sort of coating or waterproofing is prominent as a pure fibre material does not have much inherent water resistance and the properties of the material may change drastically when subjected to water or high humidity. The following is a list of possible methods for waterproofing:

- $\mathbf{SiO_2}$ has shown potential for waterproofing paper [68] and could potentially be viable, despite some apparent issues with biodegradability [69].
- **Hexamethyldisilazane (HMDS)** has been proven to increase the hydrophobic properties of MFC [70] and could potentially be used as a post moulding process for making the moulded products more hydrophobic. HMDS is applied in gas form, meaning that it should have less of an impact, if any, on the structural properties of the product.
- **Tung oil** or China wood oil is a drying oil extracted from the Tung tree that has become quite popular as an environmentally friendly wood finish.
- **Shellac Resin** is a resin made from the female lac bug and a quick test conducted in Jacobsen (2016) confirmed that this resin did, in fact, seem to be affecting the surface and the properties of the moulded paper, implying that it could potentially be used for waterproofing.
- **Laser engraving and cutting** did, despite no conclusive evidence from the probing experiment, show signs of possibly being able to alter the properties of the moulded samples and could thus be something to explore further.

Other biodegradable coatings or additives could also prove to be viable and with the societal shift towards environmentally friendly solutions, it would not be far-fetched to assume that new inventions and solutions will emerge in the near future that could be used for a material like this.

4.6.2 Possible applications and the future of thermomoulding

This thesis did not have any specific application or industry specified when conducting these tests and experiments but was more focused on assessing the properties and the potential of a material like this. That is why this section will cover some of the author's personal thought about possible applications and the future development of the moulding process itself.

Possible applications

This type of thin-walled thermoformed pulp could possibly be applied to a wide array of applications in the future. This could be something like thin covers, shells or enclosures for products like routers or other electronics, or different types of packaging or containers where

a stronger material is wanted. The thin-walled moulded pulp itself has some limitations but if multiple thinner samples were to be laminated together, similarly to glued laminated timber, the applications could be expanded by quite a bit and include anything from structural components in an office chair to the headband of a pair of headphones. Moulding thicker components make things more complicated both in terms of dewatering, moulding and drying, so a laminate product could prove to be the solution to widening the possibilities of this material. These experiments have shown that the potential is there but all of this rests on the development of efficient production methods and further research.

The moulding process

The moulding done in this thesis was quite simple but the process worked quite well both in terms of the filtering and the moulding itself once the parameters were tuned. This process could possibly be expanded and perfected to decrease filtering times significantly and allow for more complex geometries to be moulded. Thermomoulding of pulp in general is an area that has proven to be very efficient and mould-dried pulp is showing a lot of promise and with further research, new moulding processes could be developed that could expand the applications of moulded pulp significantly. Injection moulding of pulp is one possibility that although difficult, could be possible if the right solution is found.

Chapter 5

Conclusion

This thesis aimed to evaluate the mechanical properties and behaviour of mould dried thermo-moulded pulp through the means of tensile testing of moulded samples and paper sheets. In addition, the thesis sought to explore the possibilities of using microfibrillated cellulose (MFC) to reinforce such moulded materials. The initial tensile testing in the form of paper sheets revealed some significantly different behaviours between the TMP and Kraft pulps used in this thesis, especially in terms of the strain the samples were capable of achieving. When it came to the tensile testing of moulded samples, similar behaviours were observed. In addition, the moulded samples showed a tensile behaviour that seemed to be a lot more linear elastic than the paper sheets. Both the moulded TMP and Kraft proved that they were able to achieve the same level of strength, which in turn was on a par with many of the current common plastics. This suggests that it should be possible to further develop this material for specific applications and hopefully achieve properties, such as strength or modulus, similar to the current plastic alternatives. The differences in behaviour between TMP and Kraft in terms of strain, water resistance or general mechanical properties could also prove to be useful assets as the different fibre types could be picked for different applications depending on the need. The Kraft fibres showed a significantly greater ability to handle strain but also had a generally lower modulus than the TMP. Overall, they both showed promise and there was no evidence of one fibre type being superior.

The addition of MFC contributed to an increase in strength for the samples in question but unfortunately, it also brought with it a heavy toll on the dewatering characteristics of the pulp. This means that although a small amount of MFC could be added to improve the strength, it would have to be balanced so that the dewatering or filtering of the pulp would not be hindered too much. Moreover, the moulding process itself proved to be quite promising and confirmed that mould-dried thermoforming is capable of creating moulded paper with good surface finish and density. The moulding had some issues with water pockets and other defects but with further development, there could be a lot of potential in this type of plastic-like moulding processes.

Concerning the specific objectives of this thesis, they have been fulfilled accordingly:

- Thermoformed sheet production: Based on the standardised processes for making paper sheets, a hot-pressing process was explored and parameters were quantified to produce hot-pressed paper sheets with high density and adequate finish.
- Thermoforming: A process for mould-dried thermoforming of pulp was implemented,

including a pre-moulding filtering process to ensure a homogeneous and strong sample structure. The devices proved to be adequate for forming samples for mechanical assessment in accordance with the ASTM D638 standard for tensile testing of plastic.

- Mechanical assessment: Standardised tensile samples were tested and mechanical properties of the samples were quantified based on the data obtained. The mechanical assessment revealed that the moulded samples had performance similar to some plastics.

In general, the moulded samples showed a lot of promise in terms of mechanical properties and also showed some interesting differences when compared to hot-pressed paper sheets. With the right research and the continuous development of new moulding processes, the world might see sustainable moulded paper materials used in new applications in many industries in the future.

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Appendix A - Filtering System

Figure 5.1: Pre-filtering setup: The female part of the mould, with the bottom detached, is inserted into the socket of the acrylic vacuum chamber which is connected to the steel chamber. The acrylic funnel is then placed on top of the mould and the pulp is poured into the funnel and the vacuum is turned on.

Figure 5.2: Pulp filtering: This is what the setup looks like after the pulp has been poured into the funnel and the filtering has begun.

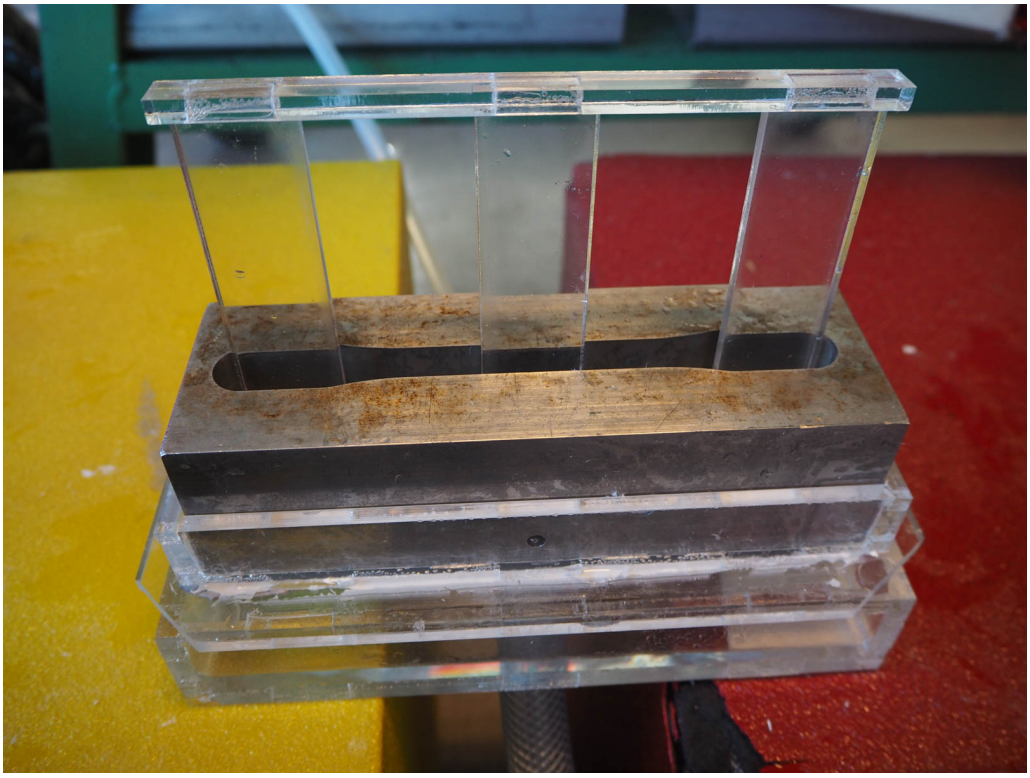

Figure 5.3: Filtering compression: Once all the pulp has been filtered through the bottom of the funnel, the funnel is removed and an acrylic piston is inserted into the mould in order to gently compress the filtered pulp. This allows for more water to be removed and prevents any loose fibres from creeping up against the walls when the mould is subjected to the hydraulic press later.

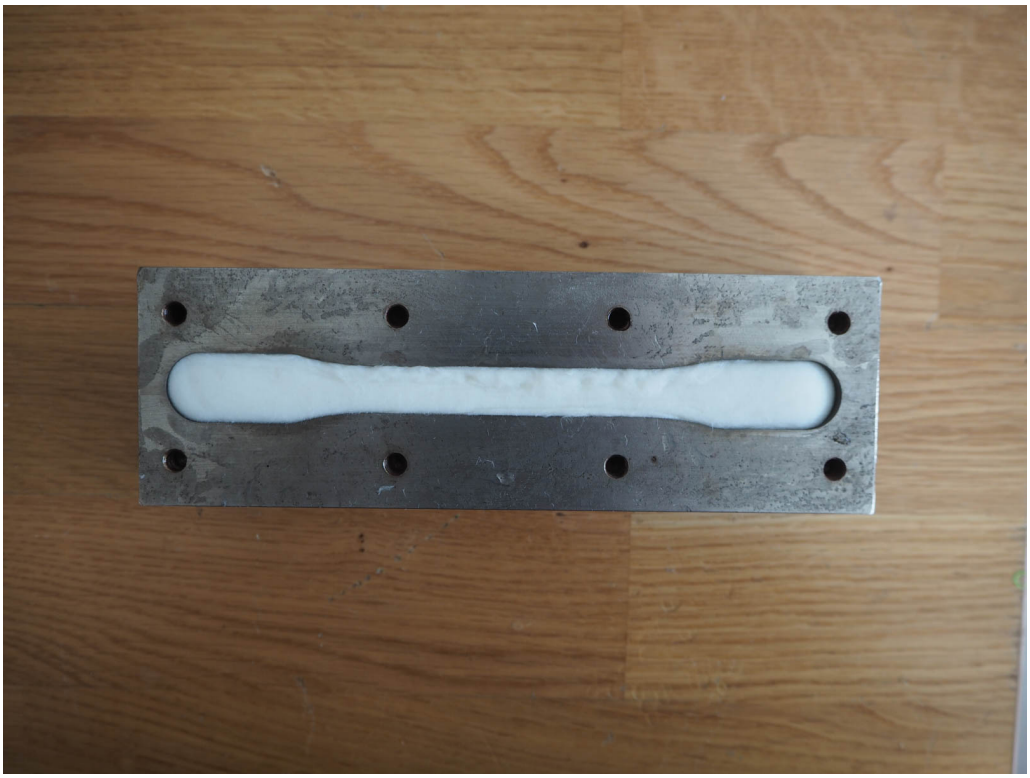

Figure 5.4: Filtered sample: This is the bottom view of a properly filtered sample. The sample is now ready to be moulded

Appendix B - Paper sheet making

The sheet-making process is done in accordance with ISO 5269-1 and the process is as follows. After a sheet weight has been chosen, the amount of fibres needed for each sheet is calculated based on the sheet weight and the sheet former surface area. This is then multiplied according to the amount of sheets you wish to make and the correct amount of fibres is measured and then disintegrated in accordance with ISO 5263-1, 2 and 3. [71–73] It is usually a good idea to disintegrate a bit more fibres than you need since some of the pulp will be removed to be used to measure the concentration of the pulp. After disintegration, the pulp is watered out with enough water to reach a concentration of $3\,\mathrm{g/L}$. This is however usually not accurate enough so a concentration test is done where $200\,\mathrm{ml}$ of pulp is taken from the suspension and then filtered and dried to calculate the exact concentration of fibres in the pulp. This concentration is then used to calculate how many litres of pulp is needed to create a single sheet at the desired sheet weight. This amount is then poured into the sheet former, seen in Figure 5.5. The pulp is poured in as the sheet former starts filling with water until the entire chamber is full. The sheet former will then mix the suspension with streams of bubbles before the water is drained off, leaving a freshly formed sheet at the bottom. The sheet is then transferred to an absorbent sheet of paper before it is sandwiched between absorbent paper, a smooth laminate plate and two thicker plastic plates. This sandwich is then transferred to a sheet press, seen in Figure 5.6. The sheet is then pressed in accordance with the ISO standard at 4 bar for 5+2 minutes. After the first five minutes, the sandwich is taken out and the absorbent paper sheets are replaced to allow for more water to be absorbed during the final two minutes. After this the sheet is stored in a plastic ziplock bag until it is taken to the hot-pressing, which is covered in Section 3.4

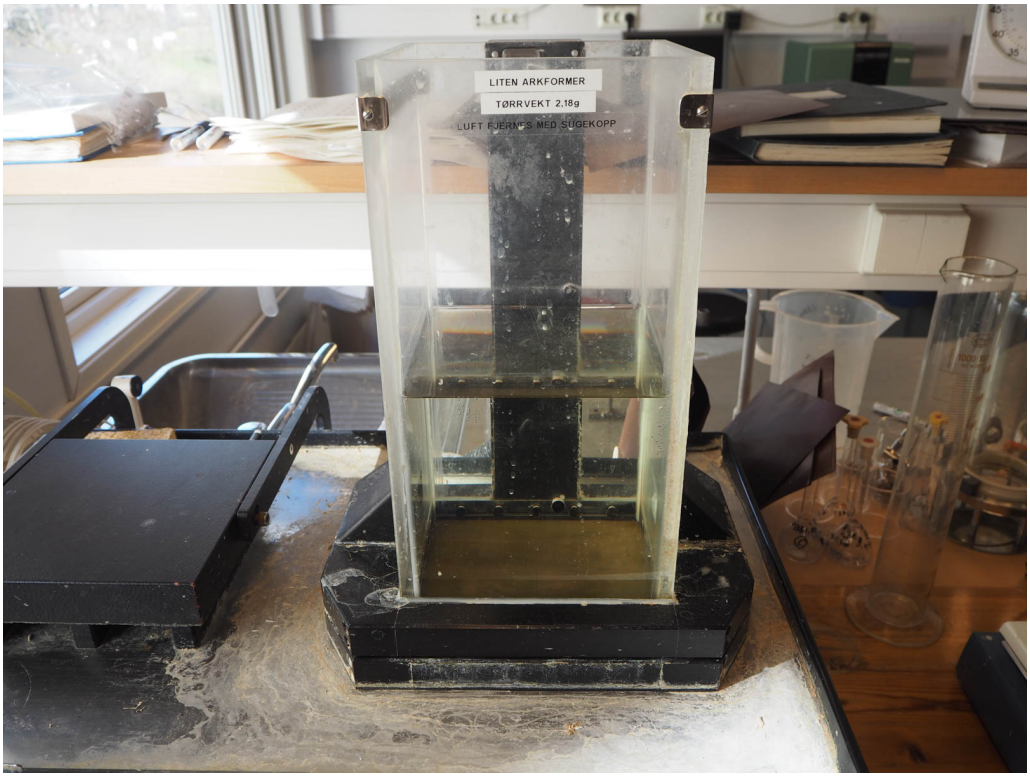

Figure 5.5: A fairly standard sheet former capable of creating $19.5\,\mathrm{cm} \times 19.5\,\mathrm{cm}$ paper sheets.

Figure 5.6: Sheet press: This press is used to remove most of the excess water still left in the formed sheet.

Appendix C - Laser treatment

This appendix covers in detail the probing experiment done to assess the properties of the moulded samples after being cut and engraved by a laser. Due to issues with the samples available and the inconclusiveness of the final results, this experiment was omitted from the main part of this thesis.

5.1 Test setup

During the work on this thesis, two new questions emerged:

- How much difference is there in the inherent water resistance of the moulded TMP and Kraft samples?
- What effect does laser cutting and etching have on the samples?

The answers to these questions were not important for the goals of this thesis but were still important enough for the potential of this material that some quick experiments were deemed to be an interesting addition.

The discovery that led to the idea of exploring laser treating the samples is covered in Section 3.2 and the goal of this experiment was essentially a non-extensive exploration of the effect a laser could have on a moulded paper material when the material is cut or etched with the laser. This could possibly be a way of adding additional waterproofing or strength to the material without compromising sustainability or biodegradability. This was essentially combined with trying to answer the question of the inherent water resistance of the moulded TMP and Kraft samples. The question of the inherent water resistance came to be as a result of curiosity. At the beginning of this thesis, it was assumed that both the moulded TMP and Kraft samples would most likely start disintegrating when subjected to large amounts of water. After the first couple of calibration rounds, a growing curiosity led to a TMP sample and a Kraft sample being subjected to the stream under a water tap for a few seconds in a very non-scientific test. To the author's surprise, there was quite a difference in how the samples reacted to the water. This lead to the idea of doing a slightly more scientific test of actually measuring and documenting this difference.

This experiment would be a simple setup where a piece of a TMP or Kraft sample would be weighed before and after being submerged in water in an attempt to measure how much water is absorbed. It was decided to cut $20\,\mathrm{mm}$ x $10\,\mathrm{mm}$ pieces in three different ways: Laser cut with engraved top and bottom surface, laser cut with no engraving and manually cut with scissors or a knife. The first step of this process was using some of the old samples in order to calibrate the engraving of the samples. Figure 5.7 shows the progress of the engraving calibration in chronological order from top to bottom. The numbers on the sample designate the dpi, power and speed settings. I.E. the first sample was engraved at 1200 dpi at 19% power and 100%

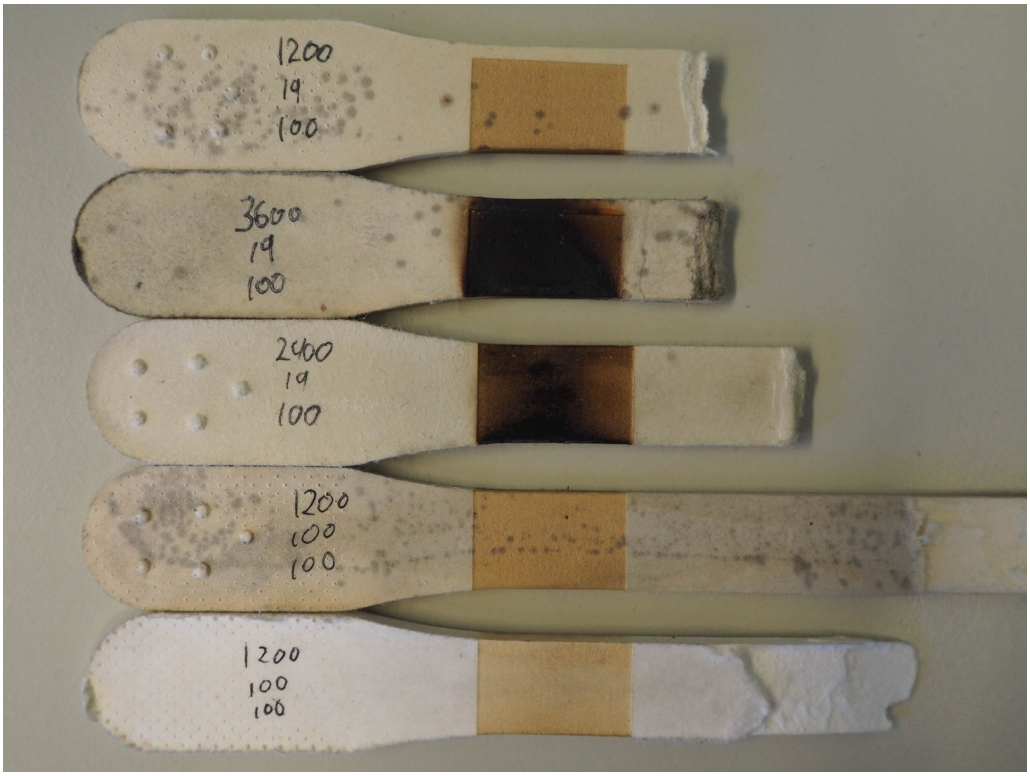

Figure 5.7: Sample engraving calibration

speed. As these samples clearly show, higher DPI resulted in severe burning of the fibres and thus it was decided to do the engraving at 1200 dpi and 100% power and speed. Three pieces of each form of cutting were then cut out from both TMP and Kraft samples. Figure 5.8 Shows the final pieces that were to be tested with TMP at the top and Kraft at the bottom. There are however a couple of sources of error here. First of all, cutting the non-treated samples pieces by hand was a quite difficult and as the picture shows those pieces are a bit more varied than the laser cut pieces. Due to a limited supply of moulded samples to cut these pieces from there are also some of these samples that have 2% of MFC added. This could possibly affect the results but moulding new samples only for this experiment would be too time-consuming so what was available had to be used. During the preparation of the samples, it was also noted that cutting the pieces manually seemed to affect the edge a lot and it was believed that this could possibly make them more absorbent. Moreover, the sharp corners present on these pieces could also lead to more water being absorbed. Because of this, it was decided to also do the same test for a full size moulded sample of TMP and Kraft. This would be without any laser treatment. The hypothesis was that the moulded samples would possibly have better water resistance before cutting them.

The rest of the experiment consisted simply of weighing each piece and then submerging it in water for $30\,\mathrm{s}$ and then leaving it on a piece of paper towel for $10\,\mathrm{s}$ before it is weighed again. The paper towel step was to ensure that there is no water on the surface that affects the final weight.

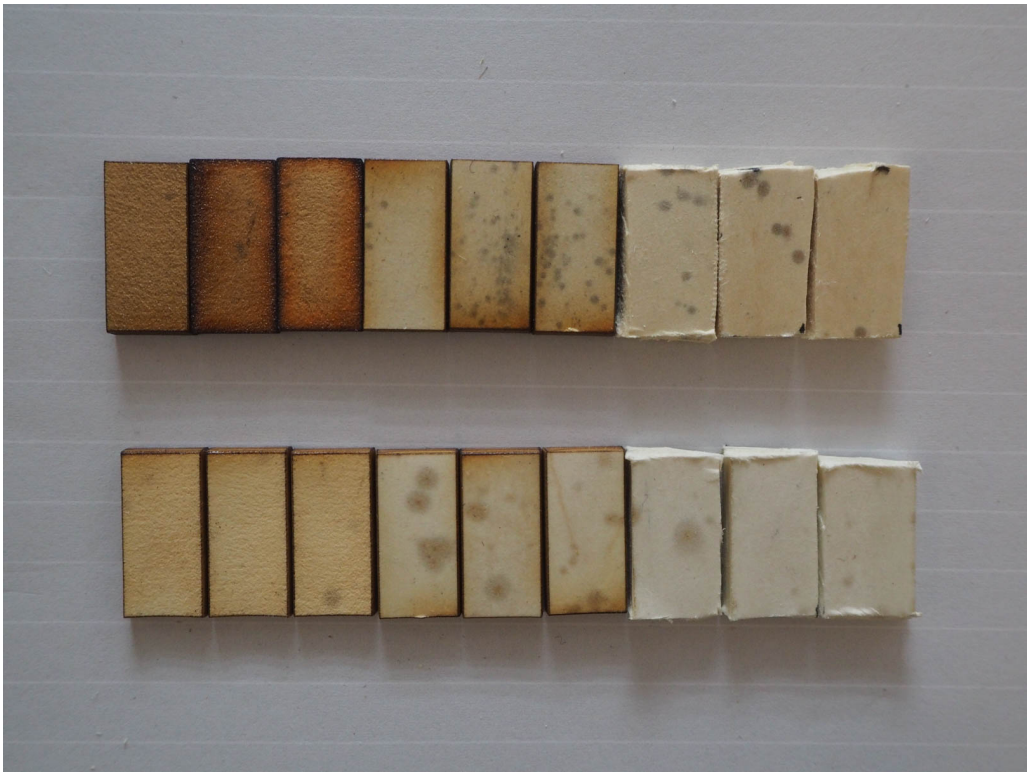

Figure 5.8: Water resistance test pieces with varying degrees of laser treatment.

5.2 Results

Figure 5.1 shows the percentage of increase in weight for the samples before and after being submerged in water.

In general, the data presented here is fairly inconclusive as there are both increases and decreases in the amount of water absorbed based on the laser treatment. The numbers for the full untreated samples do however suggest that the corners and edges of the smaller samples could very well be a cause for the higher numbers and more testing could perhaps provide a different picture.

Table 5.1: Weight of samples before and after being submerged in water

Fibre type	Etched surface	Laser cut edge	% weight increase after submergence
TMP	No	No	52
TMP	No	No	53
TMP	No	No	55
TMP	No	Yes	54
TMP	No	Yes	57
TMP	No	Yes	68
TMP	Yes	Yes	86
TMP	Yes	Yes	76
TMP	Yes	Yes	79
Kraft	No	No	46
Kraft	No	No	62
Kraft	No	No	60
Kraft	No	Yes	44
Kraft	No	Yes	36
Kraft	No	Yes	32
Kraft	Yes	Yes	53
Kraft	Yes	Yes	45
Kraft	Yes	Yes	51
Full samples			
TMP	NA	NA	23
Kraft	NA	NA	34

Appendix D - Initial sheet data

This appendix includes the measured sheet data for all the sheets in the initial tensile test.

5.3 Thickness

Table 5.2: TMP thickness

Sheet	**Thickness [µm]**	**Average [µm]**
TMP 1	345, 375, 362, 265, 328	335
TMP 2	344, 283, 392, 446, 386	370
TMP 3	368, 353, 452, 425, 266	373
TMP 4	347, 418, 388, 271, 368	358

Table 5.3: Recycled thickness

Sheet	**Thickness [µm]**	**Average [µm]**
Recycled 1	321, 360, 339, 264, 357	328
Recycled 2	258, 233, 323, 360, 309	297
Recycled 3	328, 330, 380, 351, 293	336
Recycled 4	362, 316, 377, 395, 370	364

Table 5.4: Kraft thickness

Sheet	**Thickness [µm]**	**Average [µm]**
Kraft 1	358, 444, 357, 292, 395	368
Kraft 2	444, 433, 403, 422, 468	434
Kraft 3	460, 480, 413, 448, 495	459
Kraft 4	432, 348, 433, 480, 444	427

5.4 Roughness

Table 5.5: TMP roughness

Sheet	**Roughness [µm]**					**Average [µm]**
TMP 1	3.98	4.07	4.84	4.10	4.42	4.28
TMP 2	4.48	4.25	5.60	6.35	4.96	5.13
TMP 3	5.02	6.40	11.94	11.28	5.05	7.94
TMP 4	4.48	5.34	5.20	5.78	5.11	5.18

Table 5.6: Recycled roughness

Sheet	**Roughness [µm]**					**Average [µm]**
Recycled 1	7.44	11.49	9.83	4.40	10.21	8.67
Recycled 2	6.82	10.19	11.85	11.40	5.80	9.21
Recycled 3	8.92	10.02	11.97	10.70	7.95	9.91
Recycled 4	9.66	8.04	12.02	12.86	10.27	10.57

Table 5.7: Kraft roughness

Sheet	**Roughness [µm]**					**Average [µm]**
Kraft 1	6.96	6.20	8.41	9.05	6.42	7.41
Kraft 2	9.43	10.27	10.21	10.26	9.18	9.87
Kraft 3	9.43	11.14	10.16	8.45	10.84	10.00
Kraft 4	8.91	7.28	8.88	11.60	9.93	9.32

Appendix E - Risk Assessment

This appendix includes the signed risk-assessment for this project.

NTNU HMS	Kartlegging av risikofylt aktivitet	Utarbeidet av	Nummer	Dato
		HMS-avd.	HMSRV2601	22.03.2011
		Godkjent av		Erstatter
		Rektor		01.12.2006

Enhet: **Dato: 2016.09.10**

Linjeleder:

Deltakere ved kartleggingen (m/ funksjon)**:** Martin Steinert, veileder/ Eirik Ulsaker Jacobsen, student
(Ansv. veileder, student, evt. medveiledere, evt. andre m. kompetanse)

Kort beskrivelse av hovedaktivitet/hovedprosess: Masterstudent Eirik U. Jacobsen. Sustainable fibre materials for replacing plastics in 3D-forming applications.

Er oppgaven rent teoretisk? (JA/NEI)**:** NEI

«JA» betyr at veileder innestår for at oppgaven ikke inneholder noen aktiviteter som krever risikovurdering.Dersom «JA»: Beskriv kort aktiviteteten i kartleggingskjemaet under. Risikovurdering trenger ikke å fylles ut.

Signaturer: *Ansvarlig veileder: Martin Steinert* *Student: Eirik U. Jacobsen* Eirik U. Jacobsen

ID nr.	Aktivitet/prosess	Ansvarlig	Eksisterende dokumentasjon	Eksisterende sikringstiltak	Lov, forskrift o.l.	Kommentar
1	Bruk av roterende maskineri	EUJ	Maskinens brukermanual	Ukjent	Ukjent	
2	Bruk av laserkutter	EUJ	Maskinens brukermanual	Ukjent	Ukjent	
3	Bruk av hydraulisk presse	EUJ	Maskinens brukermanual	Ukjent	Ukjent	
4	Bruk av varmeelementer	EUJ	Ukjent	Ukjent	Ukjent	
5	Bruk av manuelt verktøy	EUJ	Ukjent			

NTNU HMS	Risikovurdering	Utarbeidet av	Nummer	Dato
		HMS-avd.	HMSRV2601	22.03.2011
		Godkjent av		Erstatter
		Rektor		01.12.2006

ID nr	Aktivitet fra kartleggings-skjemaet	Mulig uønsket hendelse/ belastning	Vurdering av sannsyn-lighet	Vurdering av konsekvens:				Risiko-Verdi (menn-eske)	Kommentarer/status Forslag til tiltak
			(1-5)	Menneske (A-E)	Ytre miljø (A-E)	Øk/ materiell (A-E)	Om-dømme (A-E)		
1a	Bruk av roterende maskineri	Stor kuttskade	2	D	A	A	D	2D	Sørg for at roterende deler tilstrekkelig sikret/dekket. Vær nøye med opplæring i bruk av maskineri.
1b		Liten kuttskade	3	B	A	A	A	3B	Vær nøye med opplæring i bruk av maskineri. Ikke ha løse klær/tilbehør på kroppen.
1d		Flygende spon/gjenstander	3	C	A	A	B	3C	Bruk øyevern og tildekk hurtig roterende deler (Fres og lignende.)
2a	Bruk av laserkutter	Brannskade	3	B	A	A	A	3B	Vær nøye med opplæring i bruk av maskineri. Bruk hansker ved håndtering av varme materialer.
2b		Øyeskade-laser	2	D	A	A	C	2D	Bruk øyevern! Skru av laser når maskinen ved oppsett.
2c		Brann	2	B	A	D	C	2B	Vær nøye med opplæring i bruk av maskin. Ha slukkeutstur tilgjengelig
3	Bruk av hydraulisk presse.	Klemskade	3	B	A	A	A	2B	Holde hender o.l. unna maskinen under operasjon.
4	Bruk av varmeelementer	Brannskade	4	B	A	A	A	4B	Benytt vernehansker og vær forsiktig under håndtering av varme arbeidsstykker.
5a	Bruk av manuelt verktøy	Stor kuttskade	2	D	A	A	D	2D	Vis forsiktighet ved verktøybruk
5b		Liten kuttskade	3	B	A	A	A	3B	Vis forsiktighet ved verktøybruk

Sannsynlighet vurderes etter følgende kriterier:

Svært liten 1	Liten 2	Middels 3	Stor 4	Svært stor 5
1 gang pr 50 år eller sjeldnere	1 gang pr 10 år eller sjeldnere	1 gang pr år eller sjeldnere	1 gang pr måned eller sjeldnere	Skjer ukentlig

Konsekvens vurderes etter følgende kriterier:

Gradering	Menneske	Ytre miljø Vann, jord og luft	Øk/materiell	Omdømme
E **Svært Alvorlig**	Død	Svært langvarig og ikke reversibel skade	Drifts- eller aktivitetsstans >1 år.	Troverdighet og respekt betydelig og varig svekket
D **Alvorlig**	Alvorlig personskade. Mulig uførhet.	Langvarig skade. Lang restitusjonstid	Driftsstans > ½ år Aktivitetsstans i opp til 1 år	Troverdighet og respekt betydelig svekket
C **Moderat**	Alvorlig personskade.	Mindre skade og lang restitusjonstid	Drifts- eller aktivitetsstans < 1 mnd	Troverdighet og respekt svekket
B **Liten**	Skade som krever medisinsk behandling	Mindre skade og kort restitusjonstid	Drifts- eller aktivitetsstans < 1uke	Negativ påvirkning på troverdighet og respekt
A **Svært liten**	Skade som krever førstehjelp	Ubetydelig skade og kort restitusjonstid	Drifts- eller aktivitetsstans < 1dag	Liten påvirkning på troverdighet og respekt

Risikoverdi = Sannsynlighet x Konsekvens
Beregn risikoverdi for Menneske. Enheten vurderer selv om de i tillegg vil beregne risikoverdi for Ytre miljø, Økonomi/materiell og Omdømme. I så fall beregnes disse hver for seg.

Til kolonnen "Kommentarer/status, forslag til forebyggende og korrigerende tiltak":
Tiltak kan påvirke både sannsynlighet og konsekvens. Prioriter tiltak som kan forhindre at hendelsen inntreffer, dvs. sannsynlighetsreduserende tiltak foran skjerpet beredskap, dvs. konsekvensreduserende tiltak.

NTNU HMS/KS	Risikomatrise	utarbeidet av HMS-avd.	Nummer HMSRV2604	Dato 08.03.2010
		godkjent av Rektor		Erstatter 09.02.2010

MATRISE FOR RISIKOVURDERINGER ved NTNU

KONSEKVENS						
	Svært alvorlig	E1	E2	E3	E4	E5
	Alvorlig	D1	D2	D3	D4	D5
	Moderat	C1	C2	C3	C4	C5
	Liten	B1	B2	B3	B4	B5
	Svært liten	A1	A2	A3	A4	A5
		Svært liten	Liten	Middels	Stor	Svært stor
		SANNSYNLIGHET				

Prinsipp over akseptkriterium. Forklaring av fargene som er brukt i risikomatrisen.

Farge		Beskrivelse
Rød		Uakseptabel risiko. Tiltak skal gjennomføres for å redusere risikoen.
Gul		Vurderingsområde. Tiltak skal vurderes.
Grønn		Akseptabel risiko. Tiltak kan vurderes ut fra andre hensyn.

Appendix F - Jacobsen (2016)

This appendix includes the pre-master project in its entirety.

Zero Footprint Material Production

Eirik Ulsaker Jacobsen

Autumn 2016

Project Thesis in Mechanical Engineering

Norwegian University of Science and Technology
Faculty of Engineering Science and Technology
Department of Engineering Design and Materials

Supervisor: Martin Steinert

Co-supervisor: Jørgen Blindheim

NTNU – Trondheim
Norwegian University of
Science and Technology

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Abstract

Plastic is a very popular material for many industries due to the excellent mechanical properties and low weight plastic can achieve. However, there is a prominent issue concerning the heavy environmental impact of plastic. This is both due to the processing of crude oil and the lack of biodegradability which in turn impacts nature if the plastic is not processed in a proper waste-disposal stream. This project aims to explore alternatives to plastic that are biodegradable and can be made from wood-fibre pulp. This will be done by generating concepts, solutions and production methods with the goal of creating a material capable of replacing plastic in a mass production setting. Through the application of prototyping as a means of concept-evaluation some core concepts have been reached that will be the foundation of further research. The core concepts include means of filtering and preparing samples to be moulded under high pressure and temperature. This paper presents the work leading up to the point where tensile test specimens are to be made and tested. The testing will be covered in future research along with other potential directions and solutions.

Acknowledgements

This project originates from the initial work done by Jørgen Blindheim at TrollLabs at NTNU over the course of the summer 2016. This research is what made the foundation for the work done by myself on this project leading up to this paper. I would like to thank Martin Steinert for introducing me to this project and welcoming me as a part of TrollLabs. Being a part of TrollLabs has given me valuable insights and experience in the field of early product development. I would also like to thank my co-supervisor Jørgen Blindheim for his help in pushing me in the right direction and assisting me when needed through the course of the project. Special thanks go to Norske Skog, represented by Dag Molteberg, for supplying the project with the necessary raw materials. I would also like to thank Gary Chinga Carrasco, Øyvind Eriksen and Johnny K. Melbø at PFI for assisting with the theoretical and practical aspects of working with fibres and pulp.

Contents

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Abbreviations

TMP	=	Thermo-mechanical pulp
MFC	=	Microfribrilliated cellulose
DIP	=	De-inked pulp
CTMP	=	Chemi-thermo-mechanical pulp
PFI	=	Paper and Fibre Research institute
IPM	=	Department of Engineering Design and Materials
MDF	=	Medium Density Fiberboard
CNC	=	Computer Numerical Control
CAD	=	Computer-Aided Design
CAM	=	Computer-Aided Manufacturing
CAE	=	Computer-Aided Engineering
PID	=	Proportional-Integral-Derivative
SSR	=	Solid State Relay
ASTM	=	American Society for Testing and Materials
PLA	=	Polylactic Acid
WFRC	=	Wood-fibre-reinforced Composite
WPC	=	Wood-Plastic Composite
SEM	=	Scanning Electron Microscope

Chapter 1

Introduction and theory

This project, Zero Footprint Material Production, came to be as a result of the current state of material use in mass production. Plastic is a popular material for many applications but carries with it a heavy environmental footprint. With the current shift in the world toward cleaner energy and zero footprint solutions it is time to see if there is a better solution to all the products currently being produced in plastic. This project aims to explore concepts, solutions and production methods for a new material that would be able to replace plastic in mass production. Norske Skog, which have a long history of paper-production, is contributing with the necessary raw materials.

1.1 Introduction to fibres and pulp

Wood fibres have been used to make materials for thousands of years and the oldest archaeological fragments of the precursor of modern paper are from the 2nd century BC.[1] The process of pulp-based papermaking is said to have been developed as early as the 2nd century AD.[1] In modern days the applications of wood fibres have gone far beyond just paper and the production processes have been improved significantly. Pulp is now created by either chemically or mechanically separating cellulose fibres from wood, crops or waste paper. The most common methods for making pulp is thermo-mechanical pulping (TMP), chemi-thermo-mechanical pulping (CTMP) and purely chemical pulping.[2] There is also purely mechanical pulping but the mechanical process risks cutting fibres and thereby lowering their strength. **Table 1.1** shows the total world pulp production.[3] There is little doubt that the chemical kraft process (also known as the sulphate process) is the most popualar pulp-production method globally. According to Dag Molteberg, Development Manager at Norske Skog, they have the capability in their facilities to produce TMP and DIP (de-inked pulp, commonly know as recycled pulp) but they have to order any kraft-pulp they need for paper-making from other facilities. (Personal correspondence, 5th dec. 2016) In addition, they have a process for extracting something called microfibrillated cellulose (MFC) from kraft pulp and they are experimenting with extraction of MFC from TMP with help from the Paper and Fibre Research Insitute (PFI).

MFC is a form of nanocellulose extracted from wood fibre pulp which was first defined by Turbak et al. [4] and Herrick et al. [5] in 1983. These are tiny fibres in the structure of the cellulose that can be extracted from the pulp. **Figure 1.1** shows pictures from a scanning electron microscope (SEM) with some good examples of microfibrills in a fibrous structure.(Figure 1 from Chinga-Carrasco, G. 2011 [6]) In relation to the use of MFC, multiple studies have shown that MFC has a significant potential for increasing the strength of paper [7, 8, 9, 10].

These findings make MFC interesting for the application of a new fibrous material. Norske Skog is already producing a lot of TMP, which has been proven to be strengthened by MFC in paper-applications [9, 10]. The question, and the hypothesis for some of the work done on this project, is then if MFC could play a significant role in strengthening a material like the one this project aims to create.

Table 1.1: World pulp production

Pulping Method	**Production[M tonnes]**
Chemical	131.2
-Kraft	117
-Sulfite	7
-Semichemical	7.2
Mechanical	37.8
Nonwood	18
Total Virgin Fibres	187
Total Recovered Fibres	147
Total Pulp	**334**

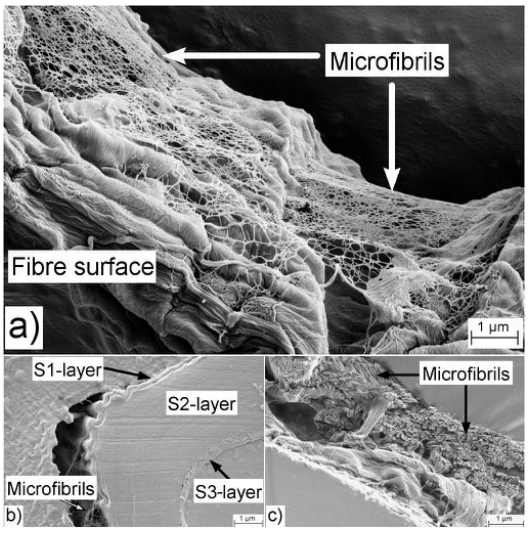


Figure 1.1: SEM-pictures of wood pulp fibres

1.2 Why this material is needed

Plastic has been a part of everyday life for many years. The first fully synthetic plastic, appropriately named Bakelite, was created by Leo Baekeland already in 1907. [11] Ever since then plastics have become an integral part of our society due to the excellent mechanical properties and low weight that can be achieved. Besides all the advantages, there is still a different aspect to the world of plastics which is not the brightest; namely the environmental impacts. In 2007, the world produced 260 million tonnes of plastic with 24.6 million tonnes of post-consumer waste in Europe. Half of this waste was deposited in landfills while $30\,\%$ was used for energy-recovery (the process of creating energy from burning waste) and only the remaining $20\,\%$ was recycled. [12] However, according to the 2016 report from the Association of European Plastic Manufacturers [13], their numbers are moving in the right direction. They claim that in 2014 their recovery rate was $29.7\,\%$ recycling, $39.5\,\%$ energy-recovery and only $30.8\,\%$ landfill. These percentages are based on the 25.8 million tonnes in the official European waste streams that year. These are positive results but there is still a prominent issue because these numbers are only based on official waste stream data that only cover Europe, a region that is actively trying to work towards better recycling and waste handling solutions. What about the data that is more difficult to find, for instance the amount of plastic dumped into nature that will never be processed, or waste-disposal rates in developing countries? In addition, there are issues regarding thermoplastics and thermosets as well. Thermoplastics are plastics capable of being reheated and remoulded and are highly suitable for recycling and reuse because of this. Thermosets on the other hand can not be softened by heat which makes them suitable for high-heat applications but in most cases this makes recycling more challenging. Moreover, the fact that most plastics are made from oil is also an issue.

Oil is a finite natural resource that is surrounded by a lot of controversy. The reintroduction of oil into our eco-system is believed to be a cause of environmental disturbances and the retrieval and refining of oil raises ethical and environmental questions. This is especially true for certain methods like hydraulic fracturing or oil-sand-retrieval. Even though his is not the main focus of this project it is safe to say that there is an issue with oil-based plastics. Fortunately, there has been an emergence of biodegradable polymers, like PLA, in recent years. These are plastics that provide the same mechanical properties as oil-plastics, but are created from natural and renewable resources like starch or soy. [14] They are, however, not entirely without flaws either and chapter 1.4 covers some of the issues surrounding biodegradability and compostability.

Wood fibres and pulp on the other hand is a material with a lot of potential. It is a renewable source of material with great properties in regards to biodegradability and compostability. The thought behind this project is that replacing plastic in mass production with a biodegradable and oil-free material could have significant long term effects on the environment. There is an issue with the energy consumption of pulp-production, and with TMP-production in particular [15], but this is an area where Norske Skog and other pulp-manufacturers are trying to improve. The issue of energy-consumption is also alleviated by the current shift towards renewable energy. Cleaner energy will help this type of energy-consuming production have less of an impact on the environment. Norske Skog already produces a lot of pulp and paper and with paper being replaced by digital alternatives in a lot of areas of society (newspapers, books, magazines etc.) they are very interested in the development of a product like this as it will allow them to maintain pulp production with a new focus.

1.3 Current alternatives

Utilising the mechanical properties of wood fibres is not a new idea by any measures. There are already a number of materials where fibres are used as reinforcement. The umbrella-term for a lot of these materials is wood-fibre-reinforced composites (WFRC). Examples of this would be MDF, which is a mix of fibres and glue, or what is referred to as wood-plastic composites (WPC). With the rise of bio-polymers there are now new materials entering the markets. Durapulp from Södra [16] is an example of this. Durapulp combines wood fibres with PLA to create a stronger plastic material. This is very similar to what this project is attempting to achieve but as mentioned in the previous section and covered in detail in section 1.4 there are issues concerning the level of biodegradability of bio-polymers and whether or not they truly satisfy the level of biodegradability this project needs. Reinforcing bio-polymers with fibres is good choice for a material like this but there are also possibilities that have not been explored yet.

As far as production methods go, moulding pulp has already been a popular production method for many years. Moulding pulp is a process used for making egg-cartons, trays etc. Examples of this can be seen in **Figure 1.2**. While these products serve their purpose quite well, most people who have ever handled an egg-carton or paper plate will know that the strength of the material is not excellent and neither is its water resistance. However, the success of these materials prove that the process of moulding pulp is capable of creating good materials and if there is a way to increase the strength and water resistance it might be a method viable for this project.

Figure 1.2: Two examples of pulp-moulded material

1.4 Biodegradability and compostability

In general, there is some confusion regarding what a material being biodegradable actually implies. According to the Merriam Webster dictionary, the term biodegradable is defined as follows;

Capable of being broken down especially into innocuous products by the action of living things (as microorganisms).

This definition could be applied to a compostable material as well which might be the cause of the confusion surrounding this. The problem is that while all compostable materials are biodegradable, not all biodegradable materials are compostable. This is why there is a need for defined parameters that separate these two categories. In the European Union there are regulations controlling the use of these terms for packaging. With these regulations there are other definitions set in place and in Pagga (1998) [17] we find the following definitions of biodegradability and compostability;

Biodegradation *is a degradation caused by biological activity, especially by enzymatic action, leading to a significant change in the chemical structure of a material.*

Compostability *is a property of a packaging to be biodegraded in a composting process. To claim compostability it must have been demonstrated that a packaging can be biodegraded in a composting system as can be shown by standard methods. The end-product must meet the relevant compost quality criteria.*

The key point here is that there are strict criteria that need to be met in order for a material to be claimed as compostable. This is covered in the *EN 13432* standard [18] which dictates the following;

- **Chemical composition:** The standard sets limits for volatile matter, heavy metals (Cu, Zn, Ni, Cd, Pb, Hg, Cr, Mo, Se, As) and fluorine
- **Biodegredation:** Chemical breakdown of materials into CO2, water and minerals. Pursuant to the standard at least $90\,\%$ of the materials have to be broken down by biological action within 6 months.
- **Disintegration:** The physical decomposition of a product into tiny pieces. After 12 weeks at least $90\,\%$ of the product should be able to pass through a 2 x 2 mm mesh.
- **Quality of the final compost and ecotoxicity:** The quality of the compost should not decline as a result of the added packaging material. The standard specifies checking this via ecotoxicity tests: this involves making an examination to see if the germination and biomass production of plants are not adversely affected by the influence of composted packaging.

The standard states that a material may be deemed compostable only if all of these requirements are met. The requirement for disintegration is what most biodegradable plastics might have issues fulfilling. While a lot of plastics may degrade over time and may even satisfy the 6 month biodegredation-criteria there is a good chance they will not be able to disintegrate $90\,\%$ of the material within 12 weeks. The reality is that most of the biodegradable plastics available will spend quite a lot of time in a landfill before they degrade. They might still be composted industrially where perfect conditions can be achieved but if plastics are to be taken through an industrial process to degrade them, the question then arises as to whether it would be better to recycle them instead. In addition to this, not all biodegradable plastics are created equal and

therefore one biodegradable plastic could have significantly different environmental impacts than another.

For this project in particular, the concept of compostability is a bit problematic. The preferred outcome would be a $100\,\%$ compostable material that would degrade naturally. This is however difficult to achieve since materials that are water-resistant generally have issues with being composted. There are however materials and concepts, which will be covered in future research, that hopefully will take this project as close to the goal of $100\,\%$ compostable as possible.

1.5 Initial research and testing

Before starting the chapter concerning the work done on this project there is a need to describe some of the initial work that established the foundation for the work done in the last few months. The author joined this project in early September but at that point his co-supervisor Jørgen Blindheim had already been assigned to this project over the course of the summer 2016 and did a lot of the initial research and testing. His approach to this problem quickly converged towards the concept of pressing pulp into a solid mass with a mould and a hydraulic press and then boiling off the water. His first attempts involved a mould designed for moulding carbon bike seats but that mould proved not to be optimal for this application, so he prototyped a smaller mould to test the concept. **Figure 1.3** shows the mould he made for this purpose. The mould has drainage holes drilled through the bottom and three holes in the top and bottom to fit a total of six heating elements. There is also a hole drilled in the side for a temperature sensor.

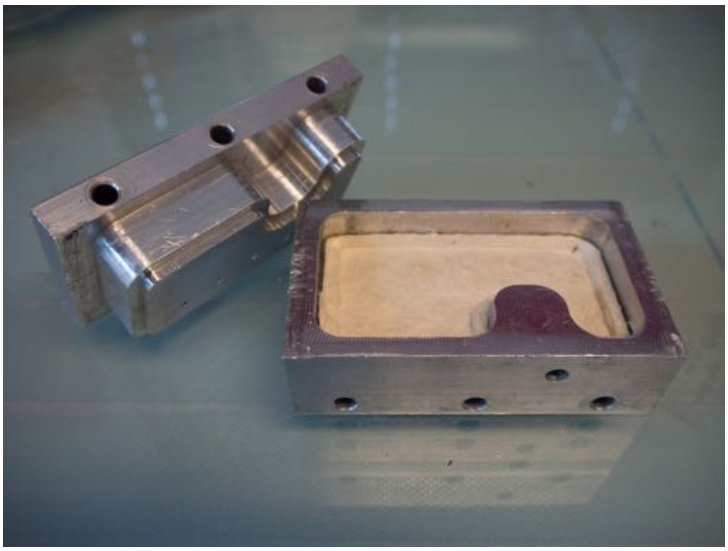


Figure 1.3: The first paper-mould

This mould proved to be quite capable of pressing the fibres and Mr. Blindheim managed to create a lot of samples from the fibres he had available. At the moment that was TMP, MFC, Kraft (in the form of paper tissues) and normal white A4 paper of unknown origin. **Figure 1.4**

show a selection of some of the samples he made with this mould. The best samples out of these were the samples pressed from paper tissues. The paper tissue samples had an excellent surface finish and showed decent mechanical properties despite being pressed from tissues and not from pulp. (The tissues create a layered structure that is quite weak to bending.) The TMP, which is one of the materials that Norske Skog produces, did unfortunately show some issues during moulding. The pulp was too dense which would cause the internal pressure in the mould to rise significantly and thus raise the boil-off point of the water. This led to some decent samples and some that were downright disastrous. The TMP samples are represented by the samples in the upper right corner of Figure 1.4 and some of the other samples with a tan/yellow colour. Pure MFC did not prove to be very mouldable and the fibres would simply creep out the side of the mould during pressing instead of compressing to a solid sample. This is however not a big issue since the future application of MFC in this project will most likely revolve around MFC being added to other fibres in smaller quantities to elevate the mechanical properties.

Figure 1.4: Some of the initial samples

The big take-away from this preliminary research revolves around two main discoveries.

- Moulding fibres with pressure and heat has the capability of producing materials with good mechanical properties
- TMP has an issue with the density during moulding which needs to be resolved

This is the point where the author entered the project which meant that there were two clear steps ahead. First figuring out how to solve the issues with the TMP and then proceeding to make a mould that can create samples for proper mechanical testing.

1.6 Prototypes and prototyping

In general, prototypes and prototyping are fairly well known concepts to most people. There may be different definitions based on what field we are considering but when considering the field of engineering design and product development we may define prototypes according to the work of Ulrich and Eppinger [19] which states the following;

> *We define prototype as 'an approximation of the product along one or more dimensions of interest'*
>
> *Prototyping is the process of developing such an approximation of the product*

Prototyping can be a very powerful tool in product development which is why it is one of the go-to steps of many projects. It is however important to assess how the prototyping should be done. Focusing on the wrong aspects of prototyping can not only waste your time but it may even prevent you from finding the best solution to your problem. There are many ways to prototype, ranging from a simple cardboard mock-up to a steel creation with full functionality. In some projects you may even want to do some experience-based prototyping which is a concept Buchenau [20] defined in 2000 as the following;

> *We can say an Experience Prototype is any kind of representation, in any medium, that is designed to understand, explore or communicate what it might be like to engage with the product, space or system we are designing.*

This could be very useful in many projects, especially in those where design thinking methodology is applied since design thinking applies a lot of human interaction to the projects through empathising and other tools.

For this project the focus of the prototyping is mainly validation and exploration of ideas. The project has been in a highly practical stage of concept testing for the last few months. Chapter 2 goes into detail about a lot of the prototypes made in the last months and the takeaways from those processes. The ability to prototype is very crucial and all the physical work done here is a prototype in some shape or another. The mould that was created, covered in chapter 2.3, is essentially an early prototype of what a factory-process would do in the final stages of this product. Prototyping allows for the testing of a hypothesis or the verification of a concept. We live in a physical world which means that despite our best efforts to theorise and calculate a response the only way to really know if something functions the way it should is to build it. The filtering system covered in chapter 2.5 is a great example where there was initially a good idea of what was likely to happen if it was built but until it was actually built there was nothing really supporting the theory. Prototyping allows you to take your theories and concepts and validate or verify them, which can be crucial to the success of your project. The next chapter will cover all the prototyping done so far on this project.

Chapter 2

Experiments and prototyping

2.1 TMP filtering

After the initial testing was done by Jørgen Blindheim and the author of this paper, it was clear that there was an issue with the density of the TMP. The working hypothesis at this point was that this was caused by the fine fibres of the pulp and that if it was possible to filter out some of the finer fibres it would allow the TMP to be pressed. Initially, it was considered that the issues might have been caused by the lignin in the pulp but after consulting with the people working at PFI it seemed more likely that it was the fine fibres that were the cause based on their previous experience with this type of fibres. The solution to this problem was a process called pressure filtering. Pressure filtering uses a rotational filter to separate the fine fibres from the long fibres and requires very specific equipment. PFI had a lot of experience in this type of work so it was agreed that a test run would be done to see if pressure filtering would help with making the TMP less dense. If the results from this initial test run were satisfactory, PFI would do a larger scale filtering job of the TMP.

For this test run it was decided to do two batches in parallel. For each batch $50\,\mathrm{g}$ of dry fibres were needed. The reasoning behind this number is that the pressure filter runs very well with a fibre concentration of around $0.5\,\%$ and a total volume of $10\,\mathrm{l}$. This makes a batch of $50\,\mathrm{g}$ very suitable and will give a good picture of the effects of the filtering. The TMP had a 30 wt% of dry fibres which meant that each batch consisted of roughly $166\,\mathrm{g}$ of pulp. The next step was to break the fibres up in a disintegrator. A disintegrator is a machine designed specifically to separate wood fibres. This is paramount before attempting to filter them because the fibres will be much easier to filter if they are as separated as possible. The temperature of the water is also very important since higher temperatures helps the fibres break loose from each other. Two disintegrators from *Lorentzen & Wettre* (Model: Code 003, Type 9340) were used for this. Each disintegrator handles around $25\,\mathrm{g}$ of dry fibres at a time so each batch was split in two disintegration-batches of $83\,\mathrm{g}$ with about $2.5\,\mathrm{l}$ of water at $90\,^{\circ}\mathrm{C}$ per disintegrator. The disintegrators run at roughly $3000\,\mathrm{rpm}$ for a total of 10 minutes, or 30000 revolutions. After disintegration, the batch is thinned out with water until there is approximately $0.5\,\%$ of dry fibres in each batch. Then a small sample is taken out to check the concentration. The final pre-filtering batch numbers are listed in **Table 2.1**

Batch B had a slightly higher concentration to begin with but it was thinned out to match the $0.585\,\%$ of batch A. This is higher than the $0.5\,\%$ target but as long as the batches are similar the higher concentration will not make much of a difference.

After all the concentrations were dialled the next step was the actual pressure filtering. A *Valmet*

Table 2.1: Pre-filtering concentrations

Batch	Measured concentration [%]	Total mass[g]	Dry mass[g]
Batch A	0,585	9722	56,9
Batch B	0,605	9617	58,2
Batch B thinned	0,585	9950	58,2

Laboratory pressure screen (Model: TAP031) was used for this with a filter of the following type, *Metso MF03-5F020G00-2 0,2 O.A 10% 803059*. The machine was set up with a 35 degree accept opening and 30 degree reject opening. These are the angles of the valves letting the fine and course fibres out of the pressure filterer where accept is the fine fibres and reject is the coarse fibres. Each batch was added to the machine together with a total of 6 litres of water through the course of the filtering process. This is to prevent clogging and help make the filtering run smoothly. For batch A, which was the first batch filtered, there were some issues with fibre-build-up in the filter. This was alleviated by more careful control of the added water but it still meant that the batch had to be taken back to the lab for disintegration before attempting to filter again. Seeing how batch A now had been filtered two times already, it was decided that a third round of filtering would be done as an attempt to figure out if multiple rounds of filtering would make the pulp better for the moulding process. Batch B did not have any issues with build-up and was done as planned with a single round of pressure filtering.

After the filtering all the excess water was drained off and a new concentration test was conducted. The final batch numbers after the filtering are shown in **Table 2.2**

Table 2.2: Final concentrations

Batch	Measured concentration [%]	Total mass[g]	Dry mass[g]
Batch A	5,6	738	41,3
Batch B	4,9	921	45,1

At this point it was noticed that the excess water that was drained off seemed to have a certain amount of fine fibres in it. In other words, the draining process also removed fine fibres from the TMP. Thus it was decided to create a batch C that was only disintegrated and drained. If batch C proved to be satisfactory for moulding then this would mean that the whole process of pressure filtering would not be necessary and a simple gravity-based filtering could be done instead. This would save a lot of time and make the preparation step before moulding a lot simpler.

The final step was to use a machine called *Fibre Tester Plus*, from the company *Lorentzen & Wettre*, to measure out the amount of fine and long fibres left in each batch. This machine tests a small sample of fibres from a batch and outputs a full rundown of the fibre count. This was done with two parallels from each batch. The general fibre counts from this test are listed in **Table 2.3**.

It is clear from the fibre count that filtering three times (batch A) removed a lot more of the fine fibres than a single filtering (batch B). There is however a clear difference in the amount of fine fibres before and after filtering. The complete rundown of all the numbers from the fibre count can be found in **Appendix C** along with graphs as well. Unfortunately, there was no time to

Table 2.3: Fibre count

Batch	Fines before filtering [%]	Fines after filtering[%]
Batch A1	43,5	28,9
Batch A2	42,5	24,2
Batch B1	48,8	41,8
Batch B2	49,9	37,3

run the same tests on batch C which could have given some interesting, although not necessary, data.

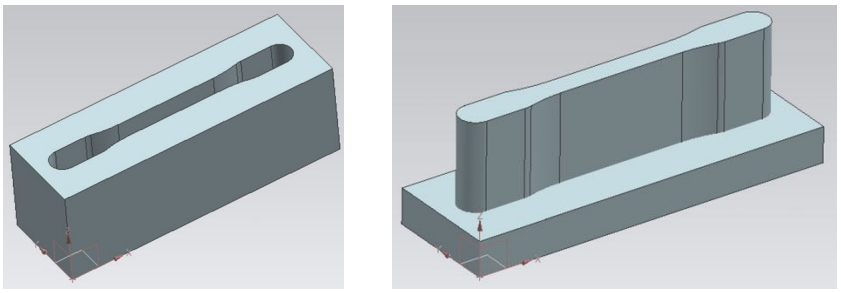
2.2 TMP filtering aftermath

After the pressure filtering at PFI it was decided to test batch C in the current mould to see how it performed. Surprisingly, it worked flawlessly. The samples came out with a great surface finish and no issues with pressure build-up in the mould. This meant that pressure filtering would not be necessary for any subsequent samples and it would be sufficient with gravity-filtering. The samples did however show some issues with cracks when put under stress. The reason for this is that the fibres tend to clump together when stored and processed and even when subjected to high pressure in the mould these clumps will not unify properly and thus create weak spots in the final samples. In order to alleviate this problem, work was started on a method for doing the gravity-filtering straight down into the mould. The hypothesis was that by first disintegrating the fibres in a large amount of water and then filtering it straight into the mould, it would create a much more homogeneous mass in which the fibres would filter down uniformly. The process of creating this filtering-prototype is covered in section 2.5

2.3 The mould

After finishing the initial pressure-filtering and getting an idea of the process needed to prepare the fibres work began on preparing a new mould in order to create samples that could be tested properly. A standardised tensile test specimen with dimensions according to ASTM D638 – 14 [21] was chosen for this purpose. This is the standard for tensile testing of plastics which is the type of material this project is aiming to replace. The ASTM standard for fibrous composites does not specify very strict sample dimensions and thus the ASTM D638 – 14 will generate a sample that will be within the standards for both plastic and fibrous composites. Doing a three-point bend test could also have been suitable but the pressure point applied by a three-point test could have an unwanted impact on the material behaviour. A three-point bend is without a doubt a very relevant test for a material like this, but at this point the goal was to see how different fibre-configurations would compare to each other and for that purpose a tensile test seemed more appropriate. In addition, the mould for the tensile specimens could also be used to create samples for three-point bend tests if the thickness of the specimen is increased. The same can not be said for getting a tensile specimen from a mould designed for three-point specimens.

Figure 2.1 shows the female (a) and male (b) CAD-models created for the mould. The software used for this was Siemens NX 11

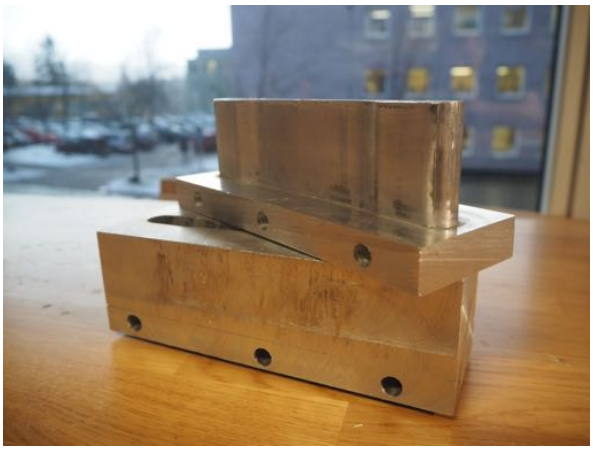


(a) Female **(b)** Male

Figure 2.1: CAD model of the mould

The base dimensions are 205x65 mm and the total height of the models are 70 mm which is the thickness of the aluminium material available at the time. The hole and the piston has been extruded to 50 mm, leaving 20 mm for the bottom and top of the mould. This allows for enough room to create thicker samples while leaving enough solid material in the bottom and top to drill holes for the heating elements that will be used. In order for a CNC mill to create this shape a rounded part has been added at each end of the specimen shape. This is not covered by ASTM but this is extra material and will thus not interfere with the other dimensions set by the ASTM standard. In addition, the transition from the grip to the gauge had to be slightly adjusted to accommodate the tools on the mill as well. The CAM work was done in Simens NX and the files were then handed over to the responsible machine operator. In order to mill the mould out with a satisfactory result, it was decided to use the *Mazak Vertical Center Smart 530C* CNC mill at the IPM workshop which meant that the job could not be done by the author but had to be done by an experienced machine operator. Unfortunately, the process of milling out the mould turned out to produce quite a few problems which is covered in chapter 3.1.

The mould was eventually finished and **Figure 2.2** shows the complete mould (a) and some test samples (b). The bottom of the mould was cut off and modified to be bolted on which provides an easy way of getting the samples out in addition to being an important feature for the mould to work with the filtering system (covered in section 2.4). There were drilled drainage holes in the bottom, six holes for the heating elements, and a hole for the temperature sensor that will monitor the mould temperature. The test samples shown are pressed from normal paper towels that were soaked in water. At this point the mould was working as intended and outputting samples with good surface finishes. More pictures of the mould can be found in **Appendix D**.



(a) Finished mould

(b) Test samples

Figure 2.2: The mould and test samples

2.4 Arduino heating module

In order to heat the mould and boil off the water a system of heating-elements capable of supplying enough power to effectively heat the mould was needed. The solution was six cylindrical (d=10mm) heating elements that were to be inserted into the mould. The control system for these elements was created by using an Arduino connected to a solid state relay that would supply power to the rods. The components used for this build was the following;

- Arduino UNO Microcontroller
- Fotek SSR-40AA Solid state relay
- MAX 6675 Thermocouple
- Type K Thermocouple probe
- 6x 250W Heating rods

The rods are all connected to a $230\,\mathrm{V}$ wall socket through the SSR which in turn is controlled by a signal from the Arduino. In the beginning it was attempted to limit the amount of cabling needed for the rods by pairing them up in a parallel configuration but it turned out that the cable connectors available at the time could not handle the current and would melt very quickly. This is why each element has its own separate cabling. The Arduino controls the SSR with a PID-controller which runs on three parameters; set-point, input and output. In this case, the set point is the desired temperature for the mould to reach, the input is the current temperature the sensor is measuring in the mould and the output is a variable that tells the Arduino whether the SSR should be open or closed in order to reach the set-point. Since the system is very slow and the elements retain a lot of heat even after being turned off a normal threshold based cut-off will cause a lot of overshooting of the set-temperature. A well-tuned PID does not overshoot and makes the temperature stable at the set-point for the duration of the process. To achieve this you need to tune the proportional, integral and derivative parameters of the PID to work with your specific system. When the work on this heater began the final mould was not ready yet so a placeholder cube was created to do the initial tuning of the controller. **Figure 2.3** shows the initial test setup.

Once the mould was finished, the work of fine tuning the PID further began so that it would perform with the proper mould and wet fibres. After a lot of testing, a final set of parameters that provided a steady aggressive ramp up to the set-temperature without overshooting was reached. **Figure 2.4** shows the completed heater boxed in. At this moment, the Arduino was powered by either a $9\,\mathrm{V}$ external power or through the USB. Future additions will include an internal $230\,\mathrm{V}$ to $5\,\mathrm{V}$ transformer to enable the unit to run on a single wall-socket power cord. In addition, a temperature-display and an LED showing the status of the SSR will be added. More pictures along with the Arduino code can be found in **Appendix E**

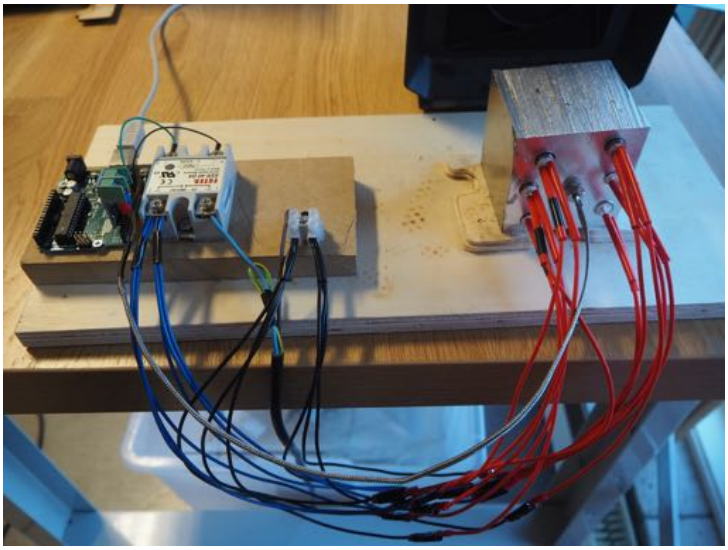


Figure 2.3: The initial heating-rig

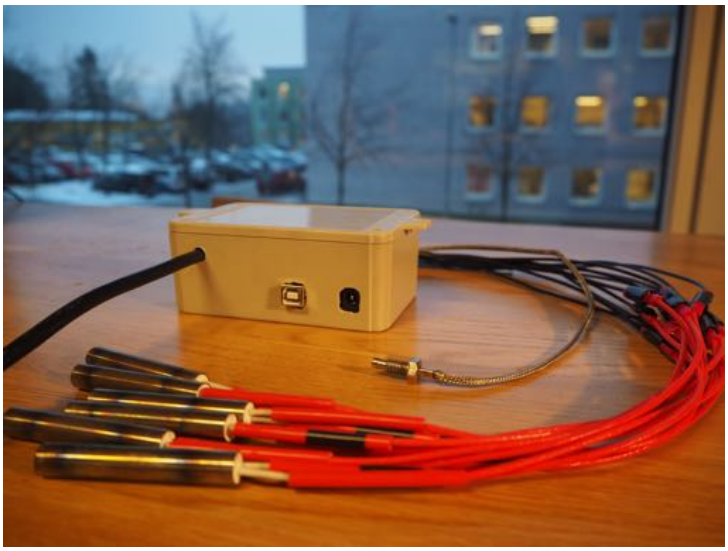
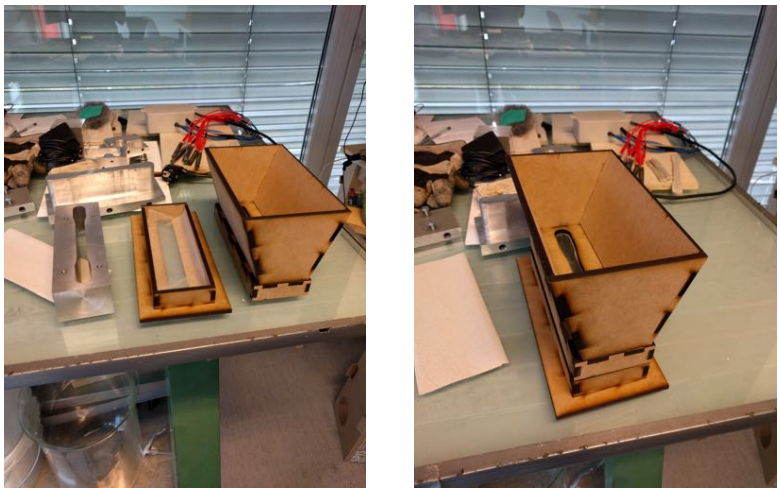


Figure 2.4: The finished heater

2.5 Filtering system

As mentioned in chapter 2.2, a conclusion had been made that there was a need for a way to filter the samples directly into the mould to get a solid and homogeneous mass. The initial ideas revolved around some sort of filtering tower to be placed above the mould and filled with the disintegrated fibres. However, after some ideating it was concluded that this process could be done in a much simpler and more compact way by utilising a small funnel instead. At this point the concept seemed very promising but to prove this, a prototype had to be made so the concept could be validate. Using Solidworks some sketches for the laser cutter were drawn and an initial test run with 6mm MDF was conducted. For the final functional prototype the idea was to use 6mm acrylic glass but acrylic requires a lot more time both for cutting and gluing the pieces together which is why MDF was chosen for the initial prototype as it would give much faster feedback on whether or not the system worked. In addition it was important to check if all the pieces fit together properly or if the sketches needed to be adjusted.



(a) Separated **(b)** Assembled

Figure 2.5: The MDF prototype

Figure 2.5 shows the initial MDF prototype in separated (a) and assembled (b) configuration. The idea is that the bottom socket has a filter that covers the bottom of the mould when the mould is inserted into the socket. The funnel is then placed on top and the system is ready. Disintegrated fibres are then poured into the funnel and all the water will be filtered out through the bottom while the fibres are left evenly inside the mould. When the filtering is done the funnel is removed and the piston is inserted before the bottom filtering-socket is removed and the bottom of the mould is attached.

It was clear at this point that all the pieces fit perfectly but it still had to be tested to see if the concept actually functioned like planned. MDF and wood glue does not handle water very well but will withstand it long enough to do one test before it breaks down. During the test there

was a lot of water leaking and the wood glue started coming loose but the filtering still worked exactly like planned. **Figure 2.6** shows the result of the test-filtering with a sample of freshly filtered TMP fibres deposited perfectly into the mould.

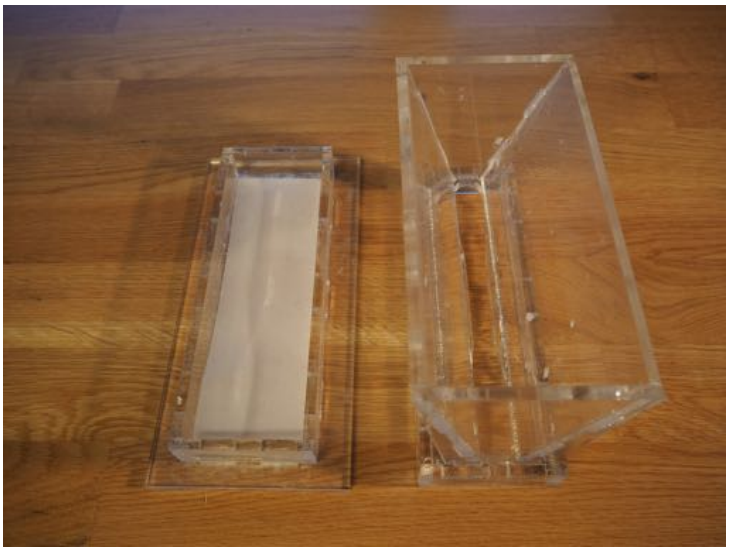
Figure 2.6: Test-filtering

All in all, this was a very successful test. The next step now was to cut the pieces out in acrylic in addition to doing a slight design adjustment to make the walls of the funnel narrower at the bottom. It also turned out that the filter used during the initial testing was a bit too fine and did not let a lot of fine fibres through. This was problematic since filtering out some of the fine fibres was the reason behind this process initially. Furthermore, the fine filter also made the filtering process quite slow. Luckily, the people working in the lab at PFI were kind enough to donate a piece of the same filter used for the draining process mentioned in chapter 2.1. This filter was not only coarser but it was also thicker and more rigid which helped keep the fibres inside the mould during filtering.

The next iteration of the prototype was thus cut from 6mm acrylic which was then glued together with *Evonik Acrifix 1S 0116* acrylic cement. At first epoxy was considered for gluing the pieces but after doing some research it was obvious that for a water tight connection between two pieces of acrylic there is no better alternative than acrylic cement since the result is comparable to welding where the two pieces are fused together. **Figure 2.7** shows the first filtering test with the new acrylic funnel (a) and the final pieces (b). This proved to be very efficient with no considerable water leaking.

This system proved to work very well and provided an excellent way to not only filter out the finer fibres of the TMP but also deposit the sample inside the mould. In addition, the filter is easily removed which allows for interchanging for a finer or coarser filter at will. More pictures of this system and the testing can be found in **Appendix F**.

(a) Filtering with acrylic funnel



(b) Acrylic pieces

Figure 2.7: Final prototype

2.6 Sample preparation

With a working mould and a filtering system in place the remaining step was to plan a process for preparing batches for filtering. In order to have complete control of the amount of fibres in each sample and the concentrations of the pre-filtering batches, every sample needs to be prepared separately. At this point the following fibre materials had been received from Norske Skog;

- TMP: 35% dry fibres
- MFC: 2,7% dry fibres
- Long fibred Sulphate: 90% dry fibres. (Cellulose with 70% spruce and 30% pine)

The plan was to create test samples of TMP and Sulphate pulp with varying concentrations of MFC added. The batch preparations consist of disintegrating the desired amount of fibres and adding water until a concentration suitable for filtering was reached. Since IPM does not have any disintegrators, PFI was contacted for assistance in the matter. It did however turn out that using the PFI labs to disintegrate and prepare the batches would be very costly, time-consuming and cumbersome. Instead, it was decided to do the preparations at IPM and build a simple disintegrator for this purpose. The disintegrator head is essentially a flat propeller that is designed to beat the fibres without cutting. The solution was to cut out the propeller shape in 6 mm acrylic glass, attach this head to a threaded rod and then attach the rod to a handheld power drill. **Figure 2.8** shows the finished disintegrator head.

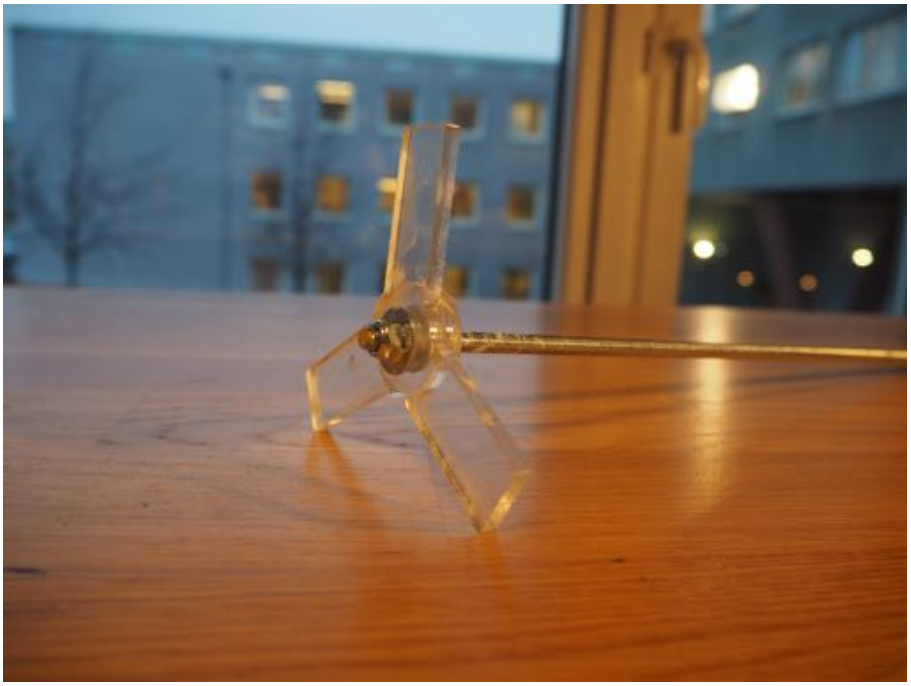


Figure 2.8: Disintegrator prototype

Finding the right concentrations of fibres for the batches would be done by simply calculating and measuring out the exact amounts of pulp needed to get the correct concentrations. This is easily done when all the dry fibre concentrations are known. In order to ensure that this process with the prototyped disintegrator would be sufficient, Johnny K. Melbø at the PFI lab was contacted to evaluate the process and the prototype. His feedback was that as long as the water used was hot enough and a sufficient amount of time was spent disintegrating the fibres there should be no problems at all. Initial testing also proved the same and the degree of disintegration was in line with what was needed for the filtering. This allowed a lot more control over each batch in addition to increasing the efficiency of the transaction from batch-preparation to filtering and pressing.

Chapter 3

Results and summary

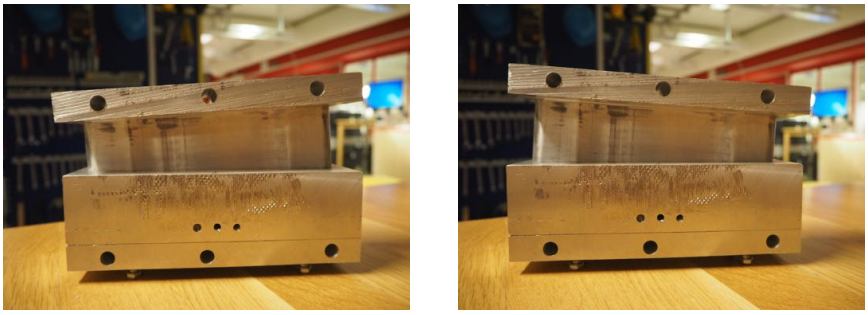
3.1 Problems with the mould

After dialling in the sample-preparation process and the filtering, the process of pressing samples could begin. This did, however, not go exactly as expected. It turned out that there were a number of issues with the mould that did not become clear until it was attempted to press the TMP.

The first issue is related to the gap between the female and male shape of the mould. The mould was designed with a clearance of 0.1mm between the two parts which would have been sufficient but during the process of milling the female shape, there was some form of malfunction with the mill. This was not something that could be seen during the milling process but when looking at the finished product there was clearly something that had gone wrong. The top of the hole was milled perfectly to the desired width but the bottom was about 0.2mm narrower than in the top. The CAM-files did not have this deviation and this was assumed to be caused by some issue with the tool of the mill. Since two solid aluminium blocks are quite expensive it was decided to try to work around the issue instead of making a new mould. It was at this point that the bottom of the mould was cut off and the work with sanding down the mould in order to widen the gap began. The only issue with this is that sanding evenly and precisely is a difficult task to do by hand and there were no tools available that could assist with this problem. However, after a few days of sanding the male and female shapes of the mould finally fit together. As seen in chapter 2.3 the mould was at this point capable of pressing excellent samples and multiple test runs were conducted without any issues. It was however noted that the mould seemed to jam slightly more after a few uses but it was assumed that this was because of the water in the mould and the tight fit. It did however become very clear when pressing the TMP that there were some problems with the mould itself.

The TMP was much more fluid than the pulp that had been pressed previously and because of this it was prone to creeping up in the gap between the male and female mould when it was compressed. The first couple TMP-samples showed very little material creeping up but after a couple more runs there were quite a lot of material creeping up and thereby jamming the mould completely. After the last run it took 30 minutes of brute force to get the piston out. This is where the real issue was discovered. The sanding process itself had made parts of the gap between the parts too big and thereby making the mould more prone to both the fibres creeping up and jamming in general. When the gap gets bigger the inner piston is given more room to angle itself which will cause jamming as well. **Figure 3.1** shows how angled the piston can get at this point. This is a big problem since the long travel of the piston makes it very prone to

jamming at even the slightest angling.



(a) (b)

Figure 3.1: Jamming issues

Because of the softer mechanical properties of aluminium this issue grew with every use of the mould. Every time the mould jammed a little it would deform or remove bit of material to make the gaps even larger. The extreme angles seen in Figure 3.1 were not possible to achieve when the mould was new. In addition to this, there is a recurring issue with the aluminium mould deforming under the hydraulic pressure in general. In retrospect, aluminium was probably not the optimal choice but seeing the success of the aluminium mould created by Jørgen Blindheim and the fact that a perfect solid aluminium block was available, it was assumed that the aluminium would be capable of doing this job especially considering the thickness of the aluminium. This did however not work out and it was clear that a new steel mould had to be made. The next day after the extreme jamming issues with the aluminium mould the engineers running the workshop at IPM were consulted and it turned out there was no way they would be able to create a satisfactory steel mould before Christmas. In other words, the pressing and testing of the tensile samples had to be postponed until January.

3.2 How to solve the problems with the mould

As stated in section 3.1, the plan is to attempt to make a new mould from steel as soon as possible after Christmas. There is however some other measures and considerations that need to be taken in order to achieve a satisfactory result with the new mould. The first step would be to ensure that the right type of steel is used. After discussing with some of the engineers in the workshop it was agreed that the best course of action would be to order some proper tooling-steel to ensure that the mould will handle the stress of multiple runs of specimen-making. It would also be preferable if new tools for the CNC mill would be ordered to prevent future tool issues causing deviations from the CAM-files. In addition to better materials and tools, changes to the actual model will also have to be considered. The clearance between the male and female part will probably be decreased to as little as 0.05mm to prevent any significant gap for the fibres to creep up in. This should also be paired with proper draining in the bottom of the mould. The decreased gap would also ensure that the piston is guided properly straight down and does not jam as easily. The use of a release agent will most likely help alleviate some of the previous issues as well. A release agent was not used for the aluminium mould since it seemed better to wait with the release-coating until the initial testing was done to prevent any damage to the coating during testing. The use of a release agent could have made a difference but it would

not have done anything with the fact that the gaps were too big and that the aluminium was deforming and making the gaps larger. All in all the issues with aluminium were too significant for the mould to work.

When remaking the mould with steel the female part will most likely be redesigned to be milled out without a solid bottom as well. This will allow the tool to mill all the way through which is an easier task for the mill to accomplish than making a 50 mm deep hole with a solid bottom. The bolt-on bottom is a very practical addition as well so cutting off the bottom of the workpiece before milling will help in many ways.

3.3 Discussion and further research

Throughout this project there has been a number of developments critical to the progress of this project. The filtering of TMP at the PFI-lab stands out as one of the early episodes that had a huge impact on the progression. At that point, the author and Jørgen Blindheim almost by chance discovered that doing a simple gravity-filtering themselves would prove sufficient to alleviate the density issues they were experiencing with the TMP. If this had not been discovered, all the TMP would have had to be pressure-filtered at PFI which would severely limit the ability to make changes and create samples rapidly.

Unfortunately the issues with the aluminium mould meant that no test-data were attained this semester. There were however a lot to take away from the the samples that were made despite not being able to get any numbers on the tensile strength. The surfaces and perceived mechanical properties of the samples that were created were very promising and there is no doubt that the pressing of pulp is a manufacturing method that would be very suitable for a material like this. Moulds can be created to accommodate a wide variety of products and moulding is already a well-established production method for many different materials. Although moulding pulp seems like a good path to take, the goal is still to generate other alternative concepts to be evaluated in the next steps of this project.

Concerning the future research for this project, there is still a lot to be done. There are two main issues that require a solution, namely the mechanical properties and the water resistance. There are other issues as well, but for this material these are the two most important aspects. The mechanical properties of the fibres are already acceptable but it is important to ensure that the production method, fibre composition or added matrix (if a composite material is deemed to be the best solution) provide mechanical properties that will allow this material to be used in mass production for applications where plastic is now being used. The water resistance is however one of the largest obstacles for this project. Paper is very susceptible to water. Any water touching the surface of a fibre-based material will instantly affect the surface and over time the water will penetrate the material and severely alter the strength. This is a well known characteristic of paper and other pure fibre materials and it is something this project needs to focus on. However, there is a lot of promising ideas on how to alleviate this. The most obvious is creating a composite material with some form of bio-polymer like PLA. The only issue, as previously discussed, is if the bio-polymer path is the best path to take in terms of biodegradability. There is however many different ways you can use bio-polymers for this, for instance through the use of some sort of coating. There is also an alternative which involves MFC that has been treated to create a thin water-repellent layer. This is at an experimental stage at PFI but could prove to be valuable and allow the development of a material without bio-polymers. Jørgen Blindheim also ordered something called shellac resin which is a resin created from the bug shells. A quick test was done by applying a small amount of resin to a

fibre sample and it already showed some improvement to the water-resistance of the sample. This is perhaps not the most convenient material to coat with but it is still worth looking into. In other words there is still a lot of research to be done on solutions for the water-resistance involving both bio-polymers and other non-polymer measures.

3.4 Conclusion

The work done this semester revolved mainly around the idea of trying to make samples from wood-fibre pulp that could be tested properly in a tensile rig to get some actual numbers on the mechanical properties of different types of fibres. The idea was that subjecting the pulp to high pressure by putting a mould in a hydraulic press and then heating the mould to boil off any excess water would create a good sample material. This concept had already been proven to work by Jørgen Blindheim but there was a need for a sample shape that could be properly tested. The process of making this happen involved a number of steps including filtering TMP to remove the fine fibres (chapter 2.1), creating a tensile test sample mould (chapter 2.3), making an Arduino-controlled heater module (chapter 2.4), prototyping a filtering system for the fibres (chapter 2.5) and creating a process for preparing the fibres for the mould. (chapter 2.6)

In the end, all the pieces of the puzzle were ready but it turned out the choice of aluminium as the material for the mould was not suitable. It became clear at this point that the mould would not be able to produce the needed samples due to a number of issues stemming from the mechanical properties of aluminium and issues during the process of milling the mould. These problems are covered in chapter 3.1. Chapter 3.2 covered some of the thoughts around solutions to this problem such as acquiring proper tooling steel and new tools for the CNC mill to ensure that the next iteration of the mould will be capable of handling any strain it is put through during moulding. Combining this with a slight redesign of the mould should result in a mould that will not have the same issues.

Despite not being able to produce any samples for testing, the work done over the course of this semester has created a solid foundation of equipment and processes for working with and processing pulp. This project is by no means finished and the next steps will involve a lot of concept-evaluation concerning solutions to water-resistance, the general mechanical properties and the production process. However, the foundation has been established and wood fibres and pulp is looking very promising in regards to creating the material that this project is trying to develop.

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Appendix A - Assignment Text

This appendix includes the signed assignment text and project description.

THE NORWEGIAN UNIVERSITY
OF SCIENCE AND TECHNOLOGY
DEPARTMENT OF ENGINEERING DESIGN
AND MATERIALS

PROJECT WORK FALL 2016
FOR
STUD.TECHN. Eirik Ulsaker Jacobsen

Zero footprint material production

Develop paper/fiber based zero footprint material and explore potential production methods based on potential requirements for furniture from SBS and raw materials from Norske Skog.

- generate concepts,
- build prototypes
- build test setups
- test and compare alternatives
- judge and evaluate concepts

Also, it is expected to contribute to one or more scientific publications during the project/master thesis.
Supporting coach is Jørgen Blindheim.

Formal requirements:

Students are required to submit an A3 page describing the planned work three weeks after the project start as a pdf-file via "IPM DropIT" (http://129.241.88.67:8080/Default.aspx). A template can be found on IPM's web-page (https://www.ntnu.edu/ipm/project-and-specialization).

Performing a risk assessment is mandatory for any experimental work. Known main activities must be risk assessed before they start, and the form must be handed in within 3 weeks after you receive the problem text. The form must be signed by your supervisor. Risk assessment is an ongoing activity, and must be carried out before starting any activity that might cause injuries or damage materials/equipment or the external environment. Copies of the signed risk assessments have to be put in the appendix of the project report.

No later than 1 week before the deadline of the final project report, you are required to submit an updated A3 page summarizing and illustrating the results obtained in the project work.

Official deadline for the delivery of the report is 13 December 2016 at 2 p.m. The final report has to be delivered at the Department's reception (1 paper version) and via "IPM DropIT".

When evaluating the project, we take into consideration how clearly the problem is presented, the thoroughness of the report, and to which extent the student gives an independent presentation of the topic using his/her own assessments.

The report must include the signed problem text, and be written as a scientific report with summary of important findings, conclusion, literature references, table of contents, etc. Specific problems to be addressed in the project are to be stated in the beginning of the report and briefly discussed. Generally the report should not exceed thirty pages including illustrations and sketches.

Additional tables, drawings, detailed sketches, photographs, etc. can be included in an appendix at the end of the thirty page report. References to the appendix must be specified. The report should be presented so that it can be fully understood without referencing the Appendix. Figures and tables must be presented with explanations. Literature references should be indicated by means of a number in brackets in the text, and each reference should be further specified at the end of the report in a reference list. References should be specified with name of author(s) and book, title and year of publication, and page number.

Contact persons:
At the department Martin Steinert, Jørgen Blindheim
From the industry DNA

Martin Steinert
Supervisor

NTNU
Norges teknisk-naturvitenskapelige universitet
Institutt for produktutvikling og materialer

Appendix B - Risk Assessment

This appendix includes the signed risk-assessment for this project.

NTNU HMS	Kartlegging av risikofylt aktivitet	Utarbeidet av	Nummer	Dato
		HMS-avd.	HMSRV2601	22.03.2011
		Godkjent av		Erstatter
		Rektor		01.12.2006

Enhet: **Dato: 2016.09.10**

Linjeleder:

Deltakere ved kartleggingen (m/ funksjon)**:** Martin Steinert, veileder/ Eirik Ulsaker Jacobsen, student/Jørgen Blindheim, medveileder
(Ansv. veileder, student, evt. medveiledere, evt. andre m. kompetanse)

Kort beskrivelse av hovedaktivitet/hovedprosess: Prosjektoppgave student Eirik U. Jacobsen. Zero footprint material production.

Er oppgaven rent teoretisk? (JA/NEI)**:** NEI *«JA» betyr at veileder innestår for at oppgaven ikke inneholder noen aktiviteter som krever risikovurdering. Dersom «JA»: Beskriv kort aktiviteteten i kartleggingsskjemaet under. Risikovurdering trenger ikke å fylles ut.*

Signaturer: *Ansvarlig veileder: Martin Steinert* *Student: Eirik U. Jacobsen* Eirik U. Jacobsen

ID nr.	Aktivitet/prosess	Ansvarlig	Eksisterende dokumentasjon	Eksisterende sikringstiltak	Lov, forskrift o.l.	Kommentar
1	Bruk av roterende maskineri	EUJ	Maskinens brukermanual	Ukjent	Ukjent	
2	Bruk av laserkutter	EUJ	Maskinens brukermanual	Ukjent	Ukjent	
3	Bruk av hydraulisk presse	EUJ	Maskinens brukermanual	Ukjent	Ukjent	
4	Bruk av 3D-printer	EUJ	Maskinens brukermanual	Ukjent	Ukjent	
5	Bruk av manuelt verktøy	EUJ	Ukjent			

NTNU | HMS

Risikovurdering

Utarbeidet av	Nummer	Dato
HMS-avd.	HMSRV2601	22.03.2011
Godkjent av		Erstatter
Rektor		01.12.2006

ID nr	Aktivitet fra kartleggings-skjemaet	Mulig uønsket hendelse/ belastning	Vurdering av sannsyn-lighet	Vurdering av konsekvens:				Risiko-Verdi (menn-eske)	Kommentarer/status Forslag til tiltak
			(1-5)	Menneske (A-E)	Ytre miljø (A-E)	Øk/ materiell (A-E)	Om-dømme (A-E)		
1a	Bruk av roterende maskineri	Stor kuttskade	2	D	A	A	D	2D	Sørg for at roterende deler tilstrekkelig sikret/dekket. Vær nøye med opplæring i bruk av maskineri.
1b		Liten kuttskade	3	B	A	A	A	3B	Vær nøye med opplæring i bruk av maskineri. Ikke ha løse klær/tilbehør på kroppen.
1d		Flygende spon/gjenstander	3	C	A	A	B	3C	Bruk øyevern og tildekk hurtig roterende deler (Fres og lignende.)
2a	Bruk av laserkutter	Brannskade	3	B	A	A	A	3B	Vær nøye med opplæring i bruk av maskineri. Bruk hansker ved håndtering av varme materialer.
2b		Øyeskade-laser	2	D	A	A	C	2D	Bruk øyevern! Skru av laser når maskinen ved oppsett.
2c		Brann	2	B	A	D	C	2B	Vær nøye med opplæring i bruk av maskin. Ha slukkeutstur tilgjengelig
3	Bruk av hydraulisk presse.	Klemskade	3	B	A	A	A	2B	Holde hender o.l. unna maskinen under operasjon.
4a	Bruk av 3D-printer	Brannskade	3	B	A	A	A	3B	Vær nøye med opplæring i bruk av maskin.
4b		Innhalering av plast/ printemateriale	5	A	A	A	A	5A	Bruk åndedretsvern/ vernebriller
5a	Bruk av manuelt verktøy	Stor kuttskade	2	D	A	A	D	2D	Vis forsiktighet ved verktøybruk
5b		Liten kuttskade	3	B	A	A	A	3B	Vis forsiktighet ved verktøybruk

NTNU HMS	Risikovurdering	Utarbeidet av	Nummer	Dato
		HMS-avd.	HMSRV2601	22.03.2011
		Godkjent av		Erstatter
		Rektor		01.12.2006

Sannsynlighet vurderes etter følgende kriterier:

Svært liten 1	Liten 2	Middels 3	Stor 4	Svært stor 5
1 gang pr 50 år eller sjeldnere	1 gang pr 10 år eller sjeldnere	1 gang pr år eller sjeldnere	1 gang pr måned eller sjeldnere	Skjer ukentlig

Konsekvens vurderes etter følgende kriterier:

Gradering	Menneske	Ytre miljø Vann, jord og luft	Øk/materiell	Omdømme
E **Svært Alvorlig**	Død	Svært langvarig og ikke reversibel skade	Drifts- eller aktivitetsstans >1 år.	Troverdighet og respekt betydelig og varig svekket
D **Alvorlig**	Alvorlig personskade. Mulig uførhet.	Langvarig skade. Lang restitusjonstid	Driftsstans > ½ år Aktivitetsstans i opp til 1 år	Troverdighet og respekt betydelig svekket
C **Moderat**	Alvorlig personskade.	Mindre skade og lang restitusjonstid	Drifts- eller aktivitetsstans < 1 mnd	Troverdighet og respekt svekket
B **Liten**	Skade som krever medisinsk behandling	Mindre skade og kort restitusjonstid	Drifts- eller aktivitetsstans < 1uke	Negativ påvirkning på troverdighet og respekt
A **Svært liten**	Skade som krever førstehjelp	Ubetydelig skade og kort restitusjonstid	Drifts- eller aktivitetsstans < 1dag	Liten påvirkning på troverdighet og respekt

Risikoverdi = Sannsynlighet x Konsekvens
Beregn risikoverdi for Menneske. Enheten vurderer selv om de i tillegg vil beregne risikoverdi for Ytre miljø, Økonomi/materiell og Omdømme. I så fall beregne disse hver for seg.

Til kolonnen "Kommentarer/status, forslag til forebyggende og korrigerende tiltak":
Tiltak kan påvirke både sannsynlighet og konsekvens. Prioriter tiltak som kan forhindre at hendelsen inntreffer, dvs. sannsynlighetsreduserende tiltak foran skjerpet beredskap, dvs. konsekvensreduserende tiltak.

NTNU	Risikomatrise	utarbeidet av	Nummer	Dato
HMS/KS		HMS-avd.	HMSRV2604	08.03.2010
		godkjent av		Erstatter
		Rektor		09.02.2010

MATRISE FOR RISIKOVURDERINGER ved NTNU

KONSEKVENS						
	Svært alvorlig	E1	E2	E3	E4	E5
	Alvorlig	D1	D2	D3	D4	D5
	Moderat	C1	C2	C3	C4	C5
	Liten	B1	B2	B3	B4	B5
	Svært liten	A1	A2	A3	A4	A5
		Svært liten	Liten	Middels	Stor	Svært stor
		SANNSYNLIGHET				

Prinsipp over akseptkriterium. Forklaring av fargene som er brukt i risikomatrisen.

Farge		Beskrivelse
Rød		Uakseptabel risiko. Tiltak skal gjennomføres for å redusere risikoen.
Gul		Vurderingsområde. Tiltak skal vurderes.
Grønn		Akseptabel risiko. Tiltak kan vurderes ut fra andre hensyn.

Appendix C - TMP-filtering

The first page of this appendix will show a selection of pictures from the filtering process done at PFI while the subsequent pages will include the fibre count data sheets that were retrieved from the fibre counting equipment.

The order of the fibre count data sheets is the following:

- Batch A, Parallel 1 before filtering
- Batch A, Parallel 1 after filtering
- Batch A, Parallel 2 before filtering
- Batch A, Parallel 2 after filtering
- Batch B, Parallel 1 before filtering
- Batch B, Parallel 2 after filtering
- Batch B, Parallel 1 before filtering
- Batch B, Parallel 2 after filtering

Appendix C

(a) One of the disintegrators

(b) Dy fibre concentration measuring

Figure 3.2: Pictures from the TMP-filtering

C2

(a) The draining process

(b) The fibre counter

Figure 3.3: Additional pictures from the TMP-filtering

Appendix C

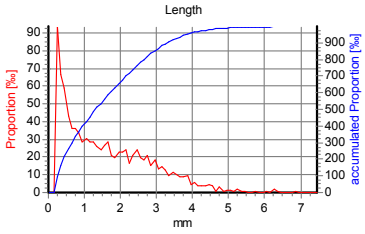
Lorentzen & Wettre
A MEMBER OF THE ABB GROUP

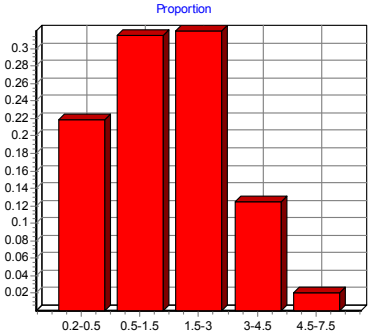
Sample name: 157400102.01
Sample type: Default
Time: 10/3/2016 1:13:45 PM
Comment: Masse A Før siling Parallell 1

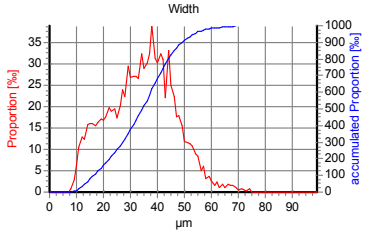
Number of fibers 9125 (236758)
Number of images 7875
Temperature 21.9 °C

Length weighted (ISO)

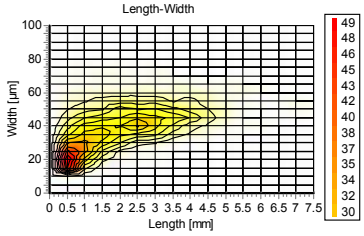
Variable	Value	Difference
Mean length	1.633 mm	0.079 mm
Mean width	34.7 µm	0.4 µm
Mean shape	83.0 %	0.0 %
Mean fibril area	11.5 %	-0.7 %
Mean fibril perimeter	37.3 %	-1.1 %
Mean fines	43.5 %	







Variable	Weighting	Value
Fines	Length	43.5 %
Number of fibers		9125 (236758)
Number of images		7875
Temperature		21.9 °C
Sample weight		0.100 g



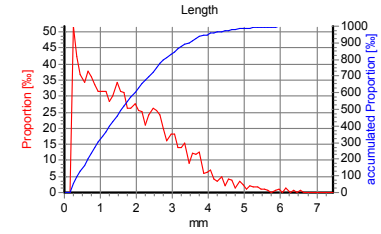
Date: 10/13/2016 9:31:06 AM Instrument number: 268

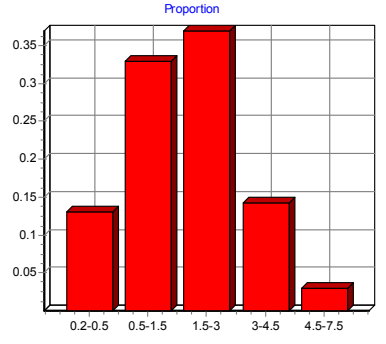
Lorentzen & Wettre
A MEMBER OF THE ABB GROUP

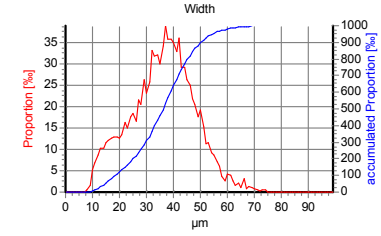
Sample name:	157400102.01	Number of fibers	9860 (211021)
Sample type:	Default	Number of images	7875
Time:	9/28/2016 1:10:26 PM	Temperature	25.4 °C
Comment	Masse A. Reject etter 3 runde siling		

Length weighted (ISO)

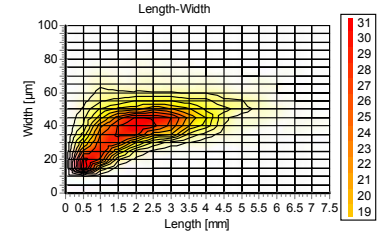
Variable	Value	Difference
Mean length	1.828 mm	0.060 mm
Mean width	36.4 µm	0.1 µm
Mean shape	83.2 %	0.0 %
Mean fibril area	10.4 %	0.2 %
Mean fibril perimeter	36.8 %	0.0 %
Mean fines	28.9 %	







Variable	Weighting	Value
Fines	Length	28.9 %
Number of fibers		9860 (211021)
Number of images		7875
Temperature		25.4 °C
Sample weight		0.100 g



Date: 10/3/2016 12:34:46 PM Instrument number: 268

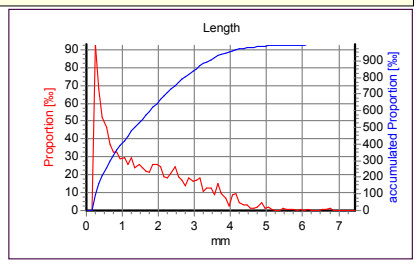
Appendix C

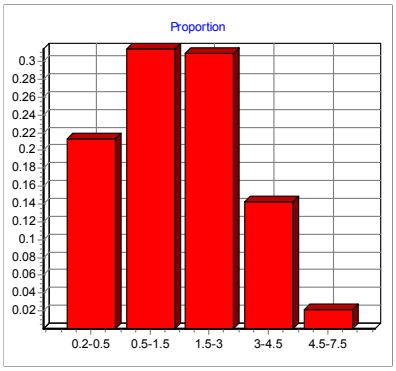
Lorentzen & Wettre
A MEMBER OF THE ABB GROUP

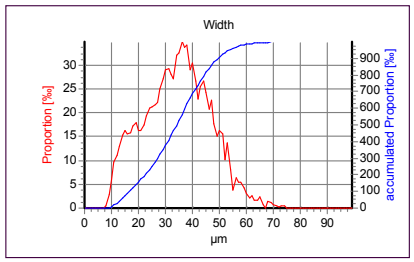
Sample name:	157400102.01	Number of fibers	8862 (221717)
Sample type:	Default	Number of images	7861
Time:	10/3/2016 1:28:07 PM	Temperature	27.7 °C
Comment	Masse A Før siling Parallell 2		

Length weighted (ISO)

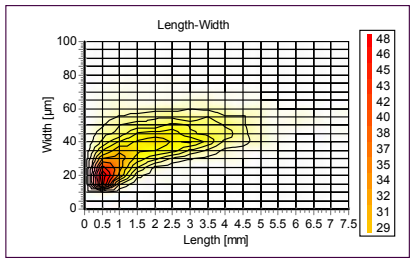
Variable	Value	Difference
Mean length	1.670 mm	-0.035 mm
Mean width	34.7 µm	-0.5 µm
Mean shape	83.1 %	0.2 %
Mean fibril area	10.7 %	-0.9 %
Mean fibril perimeter	35.5 %	-1.9 %
Mean fines	42.5 %	







Variable	Weighting	Value
Fines	Length	42.5 %
Number of fibers		8862 (221717)
Number of images		7861
Temperature		27.7 °C
Sample weight		0.100 g



Date: 10/13/2016 9:31:24 AM Instrument number: 268

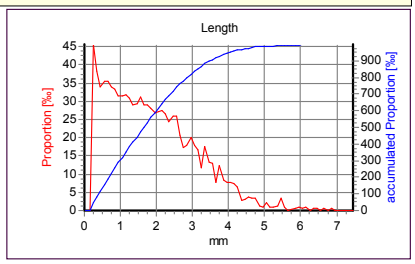
C6

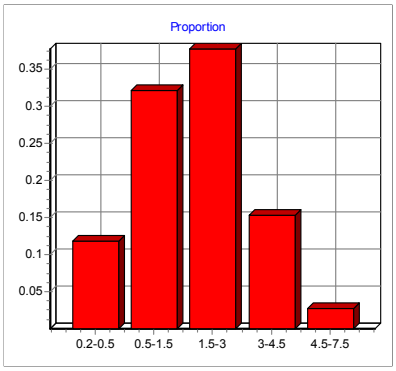
Lorentzen & Wettre
A MEMBER OF THE ABB GROUP

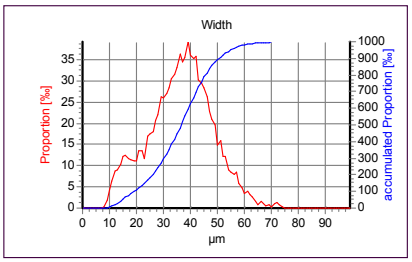
Sample name:	157400102.01	Number of fibers	11934 (194191)
Sample type:	Default	Number of images	7875
Time:	9/28/2016 1:24:51 PM	Temperature	28.2 °C
Comment	Masse A. Reject etter 3 siling		

Length weighted (ISO)

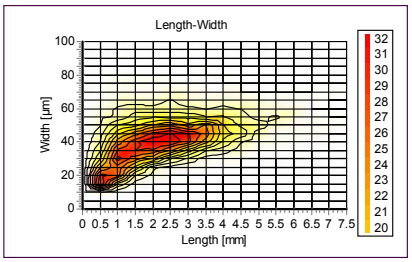
Variable	Value	Difference
Mean length	1.876 mm	0.111 mm
Mean width	36.9 µm	0.7 µm
Mean shape	83.4 %	-0.1 %
Mean fibril area	9.8 %	-0.5 %
Mean fibril perimeter	35.1 %	-0.8 %
Mean fines	24.2 %	







Variable	Weighting	Value
Fines	Length	24.2 %
Number of fibers		11934 (194191)
Number of images		7875
Temperature		28.2 °C
Sample weight		0.100 g



Date: 10/3/2016 12:35:12 PM Instrument number: 268

Appendix C

Lorentzen & Wettre
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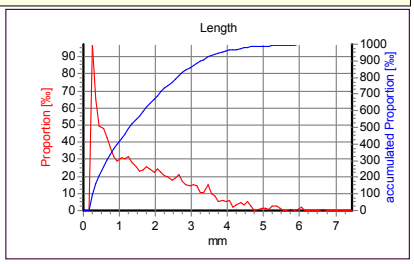
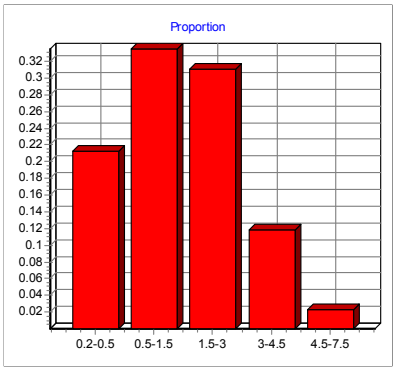
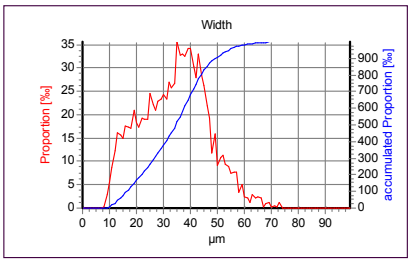
Sample name:	157400102.01 B utg	Number of fibers	8755 (307320)
Sample type:	Default	Number of images	7827
Time:	9/28/2016 12:27:12 PM	Temperature	21.8 °C
Comment	Masse B. Utg.matriale før siling		

Length weighted (ISO)

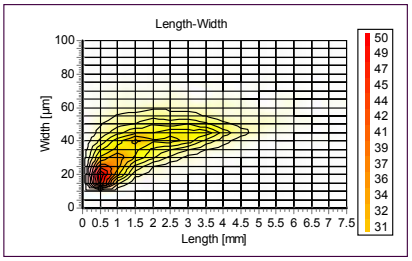
Variable	Value	Difference
Mean length	1.608 mm	-0.050 mm
Mean width	34.7 µm	0.1 µm
Mean shape	82.9 %	-0.1 %
Mean fibril area	12.3 %	0.8 %
Mean fibril perimeter	38.4 %	0.7 %
Mean fines	48.8 %	







Variable	Weighting	Value
Fines	Length	48.8 %
Number of fibers		8755 (307320)
Number of images		7827
Temperature		21.8 °C
Sample weight		0.100 g



Date: 10/3/2016 12:31:03 PM Instrument number: 268

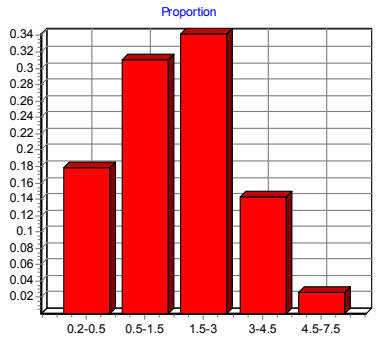
Lorentzen & Wettre
A MEMBER OF THE ABB GROUP

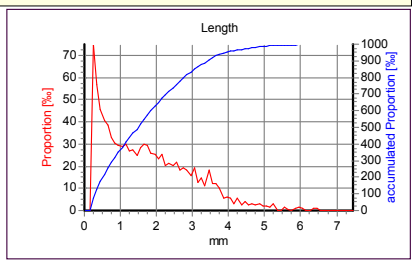
Sample name: 157400102.01
Sample type: Default
Time: 9/28/2016 12:55:58 PM
Comment: Masse B. Reject etter 1 siling

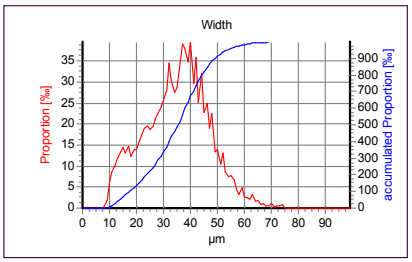
Number of fibers 8619 (284639)
Number of images 7850
Temperature 26.0 °C

Length weighted (ISO)

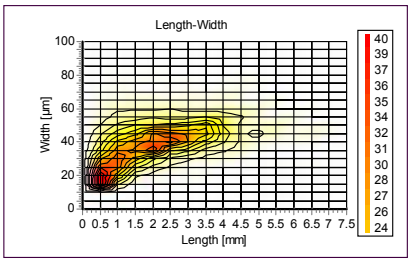
Variable	Value	Difference
Mean length	1.743 mm	0.008 mm
Mean width	35.5 µm	0.5 µm
Mean shape	83.0 %	0.0 %
Mean fibril area	10.2 %	-0.8 %
Mean fibril perimeter	35.3 %	-1.4 %
Mean fines	41.8 %	







Variable	Weighting	Value
Fines	Length	41.8 %
Number of fibers		8619 (284639)
Number of images		7850
Temperature		26.0 °C
Sample weight		0.100 g



Date: 10/3/2016 12:33:02 PM Instrument number: 268

Appendix C

Lorentzen & Wettre
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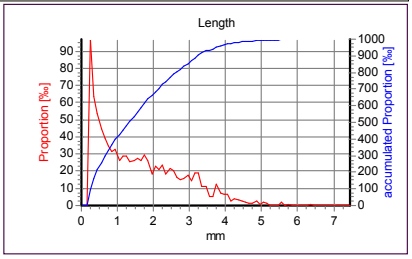
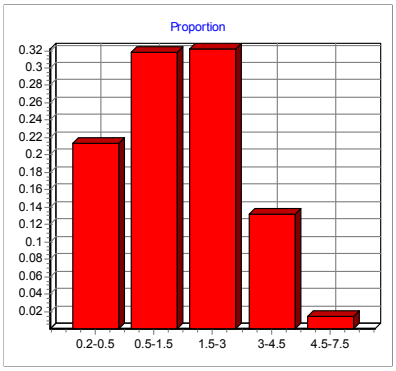
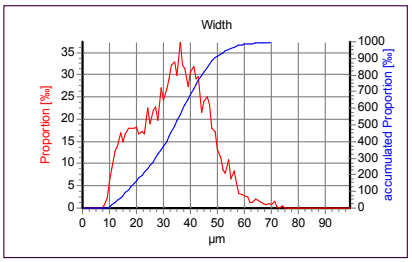
Sample name:	157400102.01	Number of fibers	9795 (398351)
Sample type:	Default	Number of images	7866
Time:	9/28/2016 12:41:36 PM	Temperature	20.3 °C
Comment	Masse B. Utg.matriale før siling		

Length weighted (ISO)

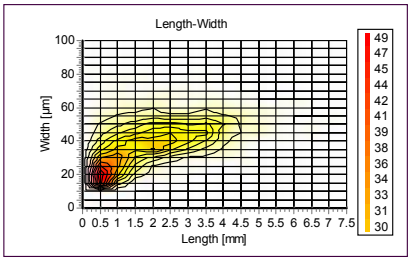
Variable	Value	Difference
Mean length	1.611 mm	-0.048 mm
Mean width	34.7 µm	-0.3 µm
Mean shape	82.9 %	0.4 %
Mean fibril area	12.3 %	0.5 %
Mean fibril perimeter	39.0 %	-0.5 %
Mean fines	49.9 %	







Variable	Weighting	Value
Fines	Length	49.9 %
Number of fibers		9795 (398351)
Number of images		7866
Temperature		20.3 °C
Sample weight		0.100 g



Date: 10/3/2016 12:32:42 PM Instrument number: 268

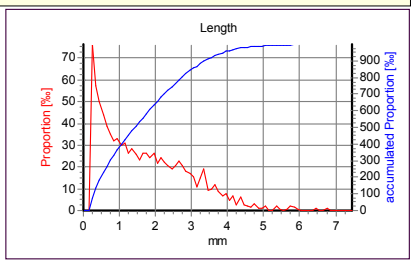
Lorentzen & Wettre
A MEMBER OF THE ABB GROUP

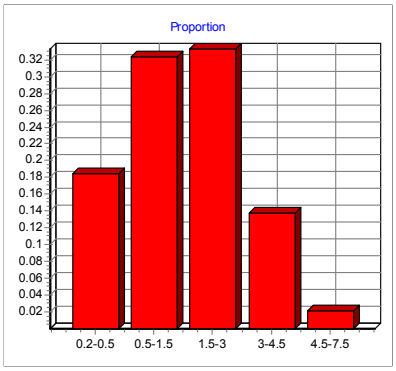
Sample name: 157400102.01
Sample type: Default
Time: 9/28/2016 1:39:22 PM
Comment: Masse B. Reject etter 1 siling

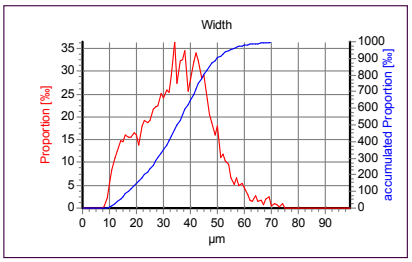
Number of fibers 9315 (220208)
Number of images 7869
Temperature 27.7 °C

Length weighted (ISO)

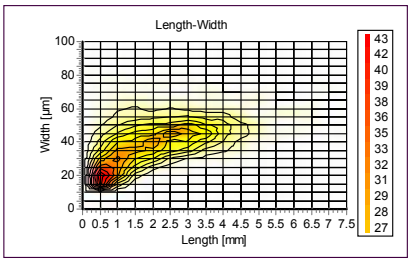
Variable	Value	Difference
Mean length	1.708 mm	0.019 mm
Mean width	35.5 µm	0.5 µm
Mean shape	82.9 %	0.1 %
Mean fibril area	11.2 %	-0.1 %
Mean fibril perimeter	36.9 %	0.0 %
Mean fines	37.3 %	







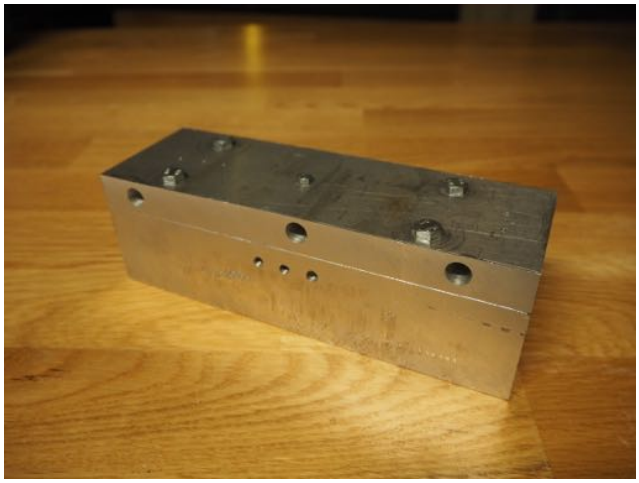
Variable	Weighting	Value
Fines	Length	37.3 %
Number of fibers		9315 (220208)
Number of images		7869
Temperature		27.7 °C
Sample weight		0.100 g



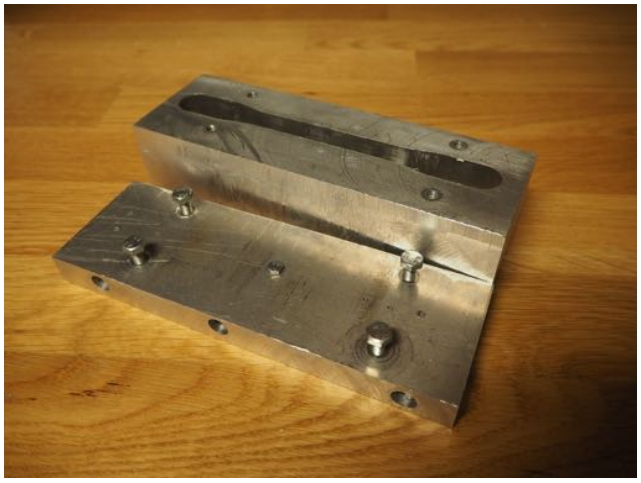
Date: 10/3/2016 12:33:40 PM Instrument number: 268

Appendix D - The Aluminium Mould

This appendix includes additional pictures of the finished aluminium mould.

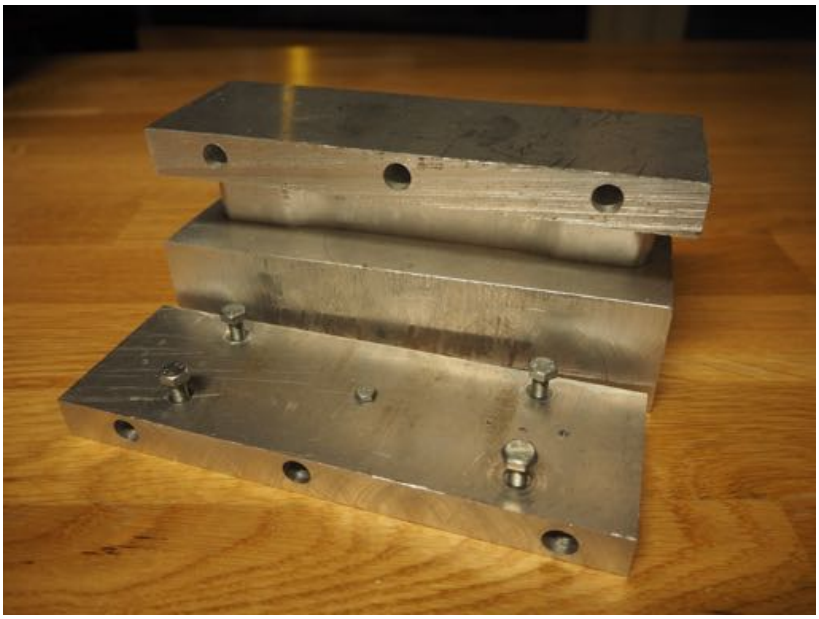


(a) View of detachable bottom

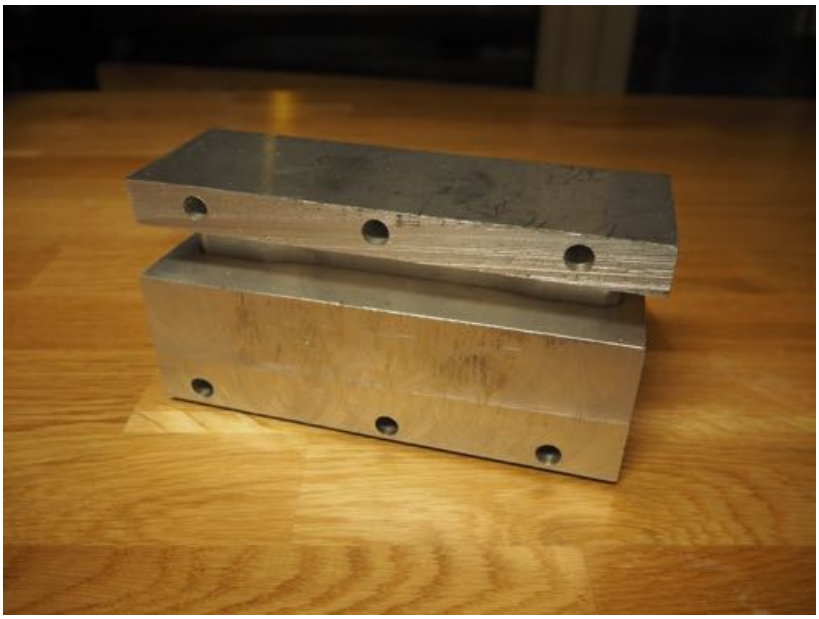


(b) Bottom detached

Figure 3.4: Pictures of the complete mould



(a) Complete mould with bottom detached



(b) Complete mould with attached bottom

Figure 3.5: Additonal pictures of the complete mould

Appendix E - The Arduino Heater

This appendix includes additional pictures of the heater module and the Arduino code used.

This first section will cover the code used in the setup of my Arduino heater box. The box utilizes a PID-controller and a solid-state relay to power and control six 250W heating elements for a total of 1500W.

Arduino-code

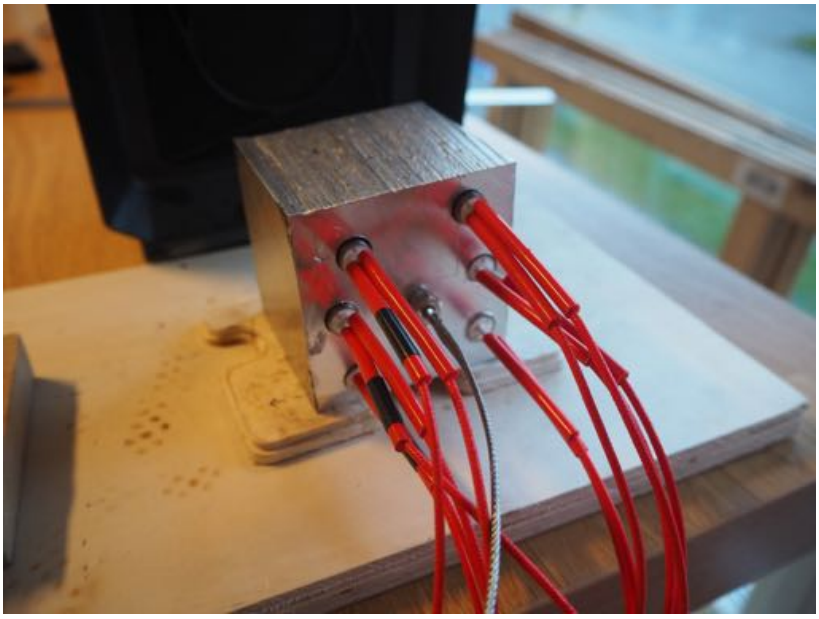
```
#include "max6675.h"
#include <PID_v1.h>
//#include <PID_AutoTune_v0.h> decided to for manual tuning

//Thermocouple start-----------------------------------------------------
int thermoDO = 6;  //Initating the thermocouple signal pins
int thermoCS = 5;
int thermoCLK = 4;

MAX6675 thermocouple(thermoCLK, thermoCS, thermoDO);
int vccPin = 3;  //Initiating thermocouple live and ground pin
int gndPin = 2;


//Controller start-------------------------------------------------------
#define RELAY_PIN 13 //Defining the pin which the relay connects to

//Define Variables we'll be connecting to
double Setpoint;
double Input;
double Output;

//Specify the links and initial tuning parameters
double Kp=100, Ki=0.10, Kd=0;

PID myPID(&Input, &Output, &Setpoint, Kp, Ki, Kd, DIRECT);

int WindowSize = 5000;
unsigned long windowStartTime;

void setup() {
  Serial.begin(9600);
  // Setup thermocouple ----------------------------------------------
  // Setting up the arduino to make the thermoucouple pins ground and 5V
```

Appendix E

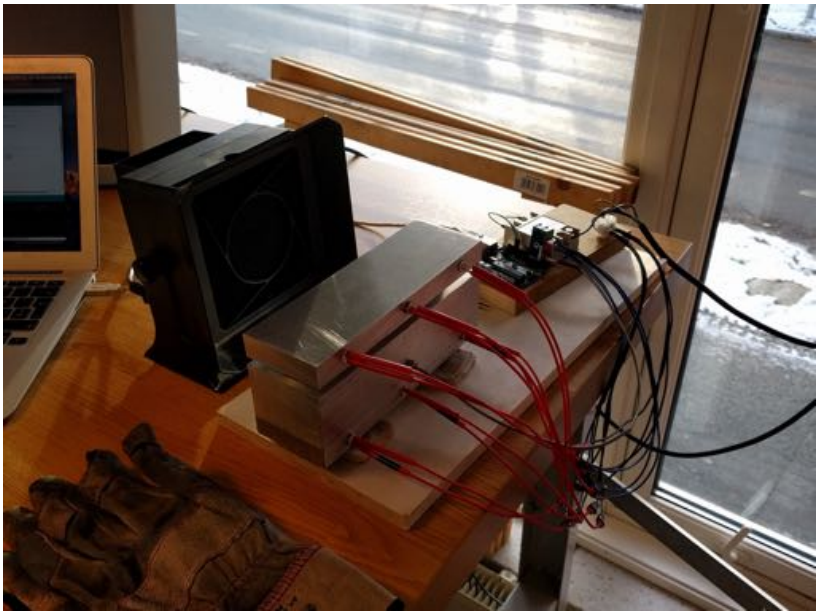
```
  pinMode(vccPin, OUTPUT); digitalWrite(vccPin, HIGH);
  pinMode(gndPin, OUTPUT); digitalWrite(gndPin, LOW);

  Serial.println("MAX6675 test");
  // wait for MAX chip to stabilize
  delay(500);

  // Setup controller ----------------------------------------------------
  windowStartTime = millis();

  Setpoint = 180; //degrees celcius, desired temperature

  //set pins as outputs
  //pinMode(INPUT_PIN, OUTPUT);digitalWrite(INPUT_PIN, LOW);
  pinMode(RELAY_PIN, OUTPUT);
  digitalWrite(RELAY_PIN, LOW);

  //tell the PID to range between 0 and the full window size
  myPID.SetOutputLimits(0, WindowSize);

  //turn the PID on
  myPID.SetMode(AUTOMATIC);

  // end setup ----------------------------------------------------------
  Serial.println("Ready");

}

void loop() {
  Input = thermocouple.readCelsius(); //Read the thermocouple value
  //Serial.println(Input);

  myPID.Compute();

  //Serial.print("INPUT:");
  //Serial.println(Input);
  //Serial.print("OUTPUT:");
  //Serial.print(Output);

  //Use the PID output to turn the relay on or off.
  if(millis() - windowStartTime > WindowSize)
  { //time to shift the Relay Window
    windowStartTime += WindowSize;
  }
  if(Output > millis() - windowStartTime) digitalWrite(RELAY_PIN, HIGH);
  else digitalWrite(RELAY_PIN, LOW);
  delay(300);
}
```

Arduino-code end

E2

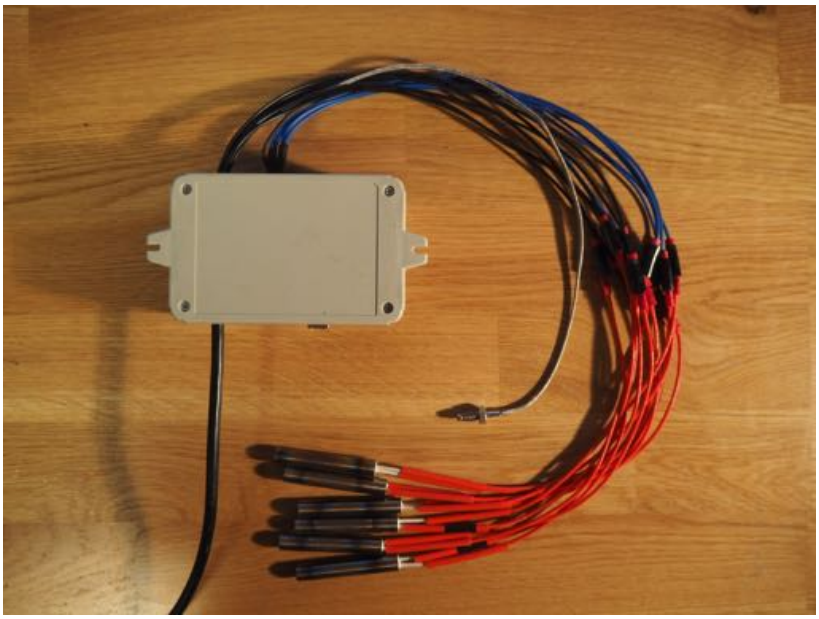


(a) The test cube

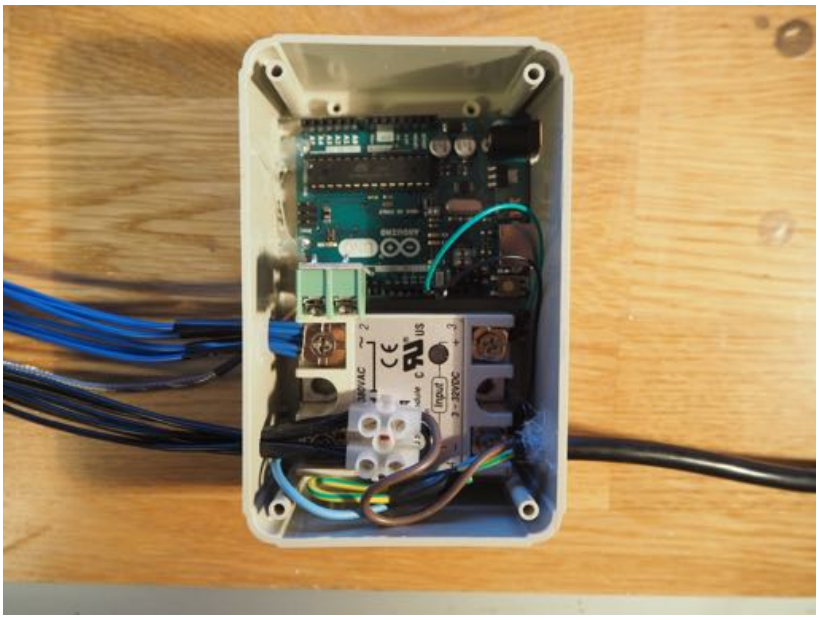


(b) Testing with the finished mould

Figure 3.6: Arduino module pictures



(a) Finished heater



(b) Inside of the box

Figure 3.7: Additional Arduino module pictures

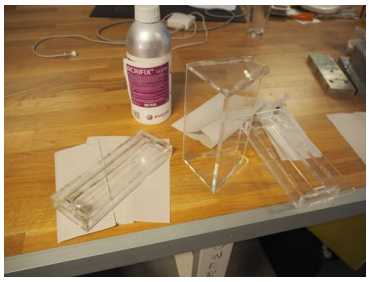
Appendix F

Appendix F - The Filtering Prototype

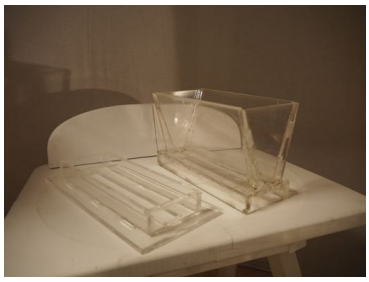
This appendix includes pictures of the filtering prorotype and the concept testing.

(a) Concept validation with MDF

(b) Acrylic gluing 1



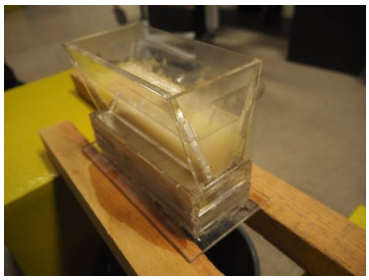
(c) Acrylic gluing 2



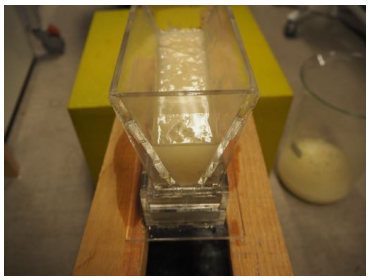
(d) Finished acrylic prototype

Figure 3.8: Filtering: Testing and prototype building

Appendix F

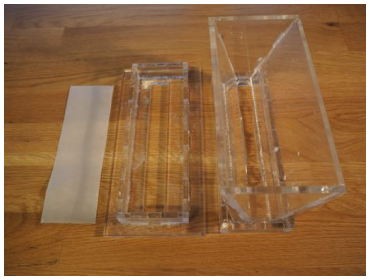


(a) Acrylic testing 1

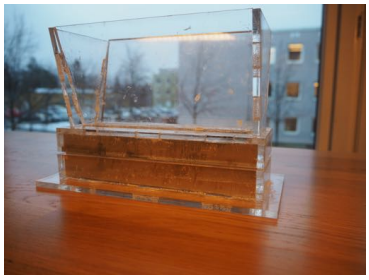


(b) Acrylic testing 2

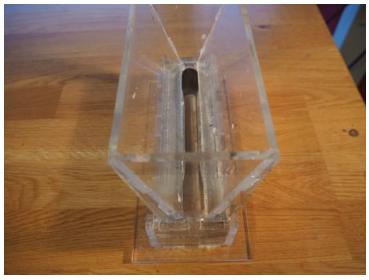
(c) Filter in



(d) Filter out



(e) With the mould



(f) With the mould 2

Figure 3.9: Filtering: Testing and finished prototype