

## Department of Materials and Environmental

Technology

# EFFECT OF HEMP FIBRE LENGTH ON THE PROPERTIES OF POLYPROPYLENE COMPOSITES

## KANEPIKIU PIKKUSE MÕJU POLÜPROPÜLEENIST KOMPOSIITIDE OMADUSTELE

## MASTER THESIS

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## **AUTHOR'S DECLARATION**

Hereby I declare, that I have written this thesis independently.

No academic degree has been applied for based on this material. All works, major viewpoints and data of the other authors used in this thesis have been referenced.

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## Department of Materials and Environmental Technology THESIS TASK

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Study programme: KVEM12/15 – Technology of Wood, Plastic and Textiles Main speciality: Wood technology Supervisor(s): Early Stage Researcher, Heikko Kallakas, +3726202910

#### Thesis topic:

(in English) Effect of hemp fibre length on the properties of polypropylene composites

(in Estonian) Kanepikiu pikkuse mõju polüpropüleenist komposiitide omadustele

#### Thesis main objectives:

1. Determine how fibre length affects the hemp fibre reinforced polypropylene composites

2. Determine if the hemp fibre amount is efficient to produce functional fibre reinforced polypropylene (FRP) composites

3. Determine the treatment method and properties of the produced hemp fibre reinforced polypropylene composites

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9	Preparation of pre-test samples	7 <sup>th</sup> April, 2017
10	Testing of pre-test samples	5 <sup>th</sup> May, 2017
11	Determination of best method/process to produce composite samples from hemp fibre and polypropylene powder	26 <sup>th</sup> May, 2017
12	Production of unmodified test specimens	20 <sup>th</sup> Sept.
13	Further preparation of hemp fibres and production of more unmodified composites	24 <sup>th</sup> Nov. 2018
14	Preparation of fibres for modification	22 <sup>nd</sup> Dec, 2018
15	Modification of hemp fibres Production of test samples from modified hemp fibres	2 <sup>nd</sup> Feb. 2018
16	Cutting of samples to standard test sizes and preparation of other complimentary materials (cutting of veneers, gluing of veneers to test specimens, notching of samples).	23 <sup>rd</sup> Feb. 2018
17	Testing of samples of water absorption and thickness swelling and mechanical testing and analysis of result	23 <sup>rd</sup> March, 2018
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## PREFACE

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Also appreciated is the support by Hempson OÜ especially in supplying the hemp bundles and for their profound interest in this research of hemp fibre as a reinforcement for plastic composites and type of products that can be produced when such can perform effectively.

While this study aims to evaluate the influence of fibre lengths on unmodified and modified fibre composite from hemp fibre and polypropylene powder, focus was on evaluating performance based on three main lengths. Samples were produced using the compression molding method. Unmodified fibre composites were first produced using this method, while modification was attained by chemically treating the fibres with alkaline and silane. The results from mechanical properties of these composites were evaluated showed that strength properties increased with increase in the length of the fibres, and conclusion and recommendations were drawn from this performance. Overall, the main objective was achieved in this master's thesis.

**Key words:** Hemp, fibre, polypropylene, modification, mechanical properties, physical properties, master thesis.

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## INTRODUCTION

Over the years, there has been increasing research to develop environmentally friendly, sustainable and reusable composite materials such as hemp, flax and sisal as alternatives to glass fibres and other carbon materials used as reinforcements for plastic polymers. (Placet, 2009).

These materials are not sustainable and very unrecyclable. They also constitute air pollution, especially when burnt after end use and are mainly obtained from fossil fuel which currently have high depletion than replacement rates. (Masuelli, 2013).

In determining the suitability of these natural fibres as reinforcement for thermoplastic composites, their mechanical properties were analyzed and compared with that of their glass fibre counterpart. The results obtained, showed comparable strength properties between these fibres and that of glass. (Wambua et al., 2003).

It is however, very important to fully analyse the factors affecting these natural fibres in the production of thermoplastics. Most importantly, the temperature and humidity that ensures the maintenance of fibre integrity. (Davies & Bruce, 1998). The right temperature is chosen with regards to the viscosity of the polymer matrix. (Gassan & Bledzki, 2001).

The use of hemp fibre which is obtained from a variety of cannabis sativa plant species is the focus of this project. A comparison between known natural fibres has shown that industrial hemp is the strongest and stiffest natural fibre with the capability for reinforcing polymer. The fibre is mainly crystalline cellulose (55 - 72 wt. %) while, hemicellulose and lignin are present in the range of (8 - 19 wt. %) and (2 - 5 wt. %) respectively. (Islam et al., 2010).

According to (Pickering et. al. 2016), the following should be considered for mechanical performance of RFP produced from natural fibres:

- 1) aspect ratio; treatment and fibre content;
- 2) matrix selection;
- 3) interfacial strength & Porosity;
- 4) fibre orientation & fibre dispersion;
- 6) composite manufacturing process; whether intrusion or extrusion.

It is important to state that, the studied research already showed how some of these factors affect the strength properties of the composites and how they can be influenced to improve the strength properties of the obtained composite product.

The aim of the master thesis is to investigate the effect of hemp fibre length on the mechanical and physical properties of polypropylene composites. In view of this, fibre length will be varied while mechanical performance is measured to help ascertain the most suitable fibre length in relation to fibre proportion for optimal performance. However, as opposed to previous research where injection moulding was identified as the most suitable method for production of fibre reinforced composites, the aim also in this thesis will be to evaluate performance of composites produced from compression moulding. To achieve this, this research objectives are the following:

1. Determine how fibre length affects the hemp fibre reinforced polypropylene composites

2. Determine if the hemp fibre amount is efficient to produce functional fibre reinforced polypropylene (FRP) composites

3. Determine the treatment method and properties of the produced hemp fibre reinforced polypropylene composites

In this thesis, chapter one addresses previous research about the subject, highlighting various points regarding natural fibre reinforced composites limitations, properties and advantages while also making comparison with composites from synthetic composites. Chapter two discusses the materials and methods taken to achieve the desired aim of this master's thesis while chapter three discusses the results of the performance of the composites. The summary attempts to draw conclusion and to state recommendation for future research.

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## **1 LITERATURE REVIEW**

The use of Natural fibres in place of glass fibres as reinforcement in the production of polymer composites is increasingly becoming the case in the automotive industry. The market growth is continually increasing, and forecast shows that this trend is to increase in the future. (Wambua et al., 2003).

Industrial and academic focus is currently veered towards addressing the growing environmental and global energy crisis using bio-based materials. These materials are environmentally friendly and may help alleviate the current situation above mentioned. On the other hand, Carbon or glass fibres are manmade products deriving their production from synthesis. These products in contrast to natural fibres only serve to further degrade the environment especially during production, after the end of life and when disposed. (Bledzki & Gassan, 1999).

The desire for a sustainable process in the automobile industry was not just borne out of the need to improve cost efficiency or mitigate environmental impact, it was also based on requirements by the European Commission. The European guideline {2000/53/E} stipulated that by 2005, 85% of the weight of vehicles had to be recyclable and an increase of 10% was set for 2015. To improve vehicle sustainability and to reduce weight and cost of production in this sector, the use of bio-fibres thus gained considerably more attention and extensive use as an interior or exterior material. (Koronis et al., 2013).

However, of all known natural fibres, hemp fibre (HF) is currently becoming the favourite option ahead of other materials such as sisal, jute and flax for reinforced polymer composite production because of its superior mechanical, sterile, thermal and acoustic property. (Suardana et al., 2011).

A major drawback in the use of natural fibres has been how to improve the fibre matrix interface bonding, decrease the formation of aggregates and improve moisture resistance (Puglia *et al.* 2008).

## **1.1** Hemp fibre (Cannabis Sativa L.)

Hemp fibre (HF) is an inexpensive high quality natural fibre that has gained increased application as interior material in the automobile industry because of its' excellent mechanical properties when compared to other natural fibres used in the manufacture of fibre reinforced thermoplastic (RFP) composites. (Suardana et al., 2011).

As a wood product, hemp fibre just like wood is made up of cellulose, hemicelluloses and lignin. In the use of HF for RFP composites, fibres are typically extracted by retting. (Sisti et al., 2017). Figure 1.1 shows already retted hemp fibre while table 1.1 shows the ratio of these constituents by weight of the hemp fibre.



Figure 1.1. Hemp fibre (HF). Source: (Denis et. al (2016))

Table 1.1 The chemical composition of hemp fibers. Source: (Suardana et. al., 2011)

Hemp Fibre	Cellulose	Pectin	Hemicelluloses	Lignin	Waxes & oils
wt.%	70.2-76.12	0.9-1.55	12.28-22.4	3.7-5.7	0.8-1.59

As shown in Table 1.2, Hemp fibre has the highest stiffness and strength properties of all known natural fibres. (Pickering et al., 2005) and its' performance as a reinforcement for thermoplastic composites is dependent on the growth conditions and method of extraction of the fibre. (Hepworth et al., 2000).

Fibers	Density (g/cm3)	Diameter (mm)	Tensile strength (MPa)	Young modulus (GPa)	Elongation at brake (%)	Price (USD/kilo)
Flax	1.50	40–600	345–1500	27–39	2.7–3.2	3.11
Hemp	1.47	25–250	550-900	38–70	1.6–4	1.55
Jute	1.3–1.49	25–250	393–800	13–26.5	1.16–1.5	0.925
Kenaf	1.5–1.6	2.6–4	350–930	40–53	1.6	0.378
Ramie	1.5–1.6	0.049	400–938	61.4–128	1.2–3.8	2
Sisal	1.45	50–200	468–700	9.4–22	3–7	0.65
Curaua	1.4	7–10	500–1100	11.8–30	3.7–4.3	0.45
Abaca	1.5	10–30	430–813	31.1–33.	2.9	0.345
E-glass	2.55	15–25	2000–3500	70–73	2.5–3.7	2

Table 1.2 Properties of selected natural fibres and E-glass fibre. Source: (Koronis et. al., 2013).

### **1.2** Polymer matrices

An important element in the fibre reinforced polymer (FRP) composite manufacture is the polymer matrix. There are currently two classes of polymers; Thermoplastic and thermosetting. FRP composite Manufacture has mainly explored the use of thermoplastic matrices such as Polypropylene (PP), Polyethylene (PE) and Poly (vinyl) chloride (PVC). (Malkapuram et al., 2008).

Polymers can be generally categorized into synthetic or Bio based. Synthetic polymers are those materials obtained from petroleum-based products while Bio based, or biodegradable polymers are materials obtained from carbohydrate rich substances like core and sugar cane. (Mohanty et al., 2005).

Of all these polymers, Polypropylene (PP) remains the most commercially available and commonly used polymer matrix for fibre composite products. They have low density (0.9 g/ml), good heat stability, impact resistance, ease of processing and low investment input. Furthermore, PP Polymer matrix also enhances the chemical and stain resistance of the resulting composite. (Denis et al., 2016), (Harutun, 2003). Though the mechanical properties of the resulting composite are mainly defined by factors such as the nature of the fibre and conditions of production, these properties still play an important role in predicting the mechanical properties of the reinforced thermoplastic material produced. (Harutun, 2003).

## 1.3 Biodegradable polymers

The combination of biofibres such as kenaf, hemp, flax, jute, henequen, pineapple leaf fibre, and sisal with either renewable or non-renewable has been sorted to produce composite materials that are competitive with synthetic composites. (Mohanty et. al, 2002). However, increased research in areas of composites from natural fibre-reinforced polypropylene, polyethylene or polystyrene have gained significant commercial attraction especially in the automobile industries. But, these composites are not so environmentally friendly because these polymer matrices are nonbiodegradable.

The process to manufacture plastics from crude oil causes production of pollutants such as carbon dioxide which is a major contributor to climate change. Crude oil is a non-sustainable and nonrenewable product. To reduce the overdependence on crude oil, research has been made over the past decades in the production of plastics from plants rather than crude oil. These types of plastics are referred to as bioplastics.

**Bioplastics** are produced when sugar in plants is converted into plastic. Examples of plants used to achieve this are sugar cane, sugar beets, wheat and potatoes. Polymers that are obtained from these renewable raw materials such as cellulose, starch, natural monomers such as polylactic acid (PLA) and microbial fermentation of poly(hydroxybutyrate) and polyhydroxyal-kanoate (PHA) are referred to as natural polymers. Among all these, PLA is the most common at present to produce natural fibre reinforced bio composite because of availability, low price and improved manufacturing practices. (Lunt, 1998).

Bioplastics can be used in injection and compression molding to produce composite products. Felix et al. researched on the development of rice protein bio-based plastic materials processed by injection molding in 2015. Rice protein concentrate (RPC) was used for the development of this biobased plastic materials processed through injection molding using 30% glycerol (GL) as plasticizer, sodium bisulfite as a reducing agent and glyoxal and L-cysteine as cross-linking agents to ensure good processing, during injection and to avoid cross linking, a temperature of 87 °C was used while the mould temperature was 130 °C to enhance cross linking. (Felix et al., 2015).

The RPC from rice husks used contained ca. 80 wt.% protein. Different blends of various RPC/GL ratios and additives at concentrations of 0.3 wt.% SB, 3.0 wt.% GLX and 1 wt.% Cyst were produced. Mixtures were mixed in Haake Ploy lab QC two-blade counter-rotating batch mixer at 50 rpm for 60

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min and 25 °C. A dough-like material was obtained and processed by injection moulding using a Minijet Piston Injection Moulding System (ThermoHaake) to obtain bioplastic specimens.

Islam et al, examined the suitability of using PLA as a natural resin to produce hemp fibre reinforced polymer composites through the compression moulding method. In this research, retted hemp hurd fibres were used. First, the hemp fibres were treated in a pulp digester at 120 °C for 60 min using 2 wt.% Na<sub>2</sub>SO<sub>3</sub> and 5 wt.% NaOH solution after which the fibres were washed in a pulp and paper fibre washer for about 45 min and then dried in an oven for 48 h at 70 °C. To produce the composite material, short hemp fibres (30 wt. % fibre) and PLA powder were used. Water was poured on a mixture of 308 g of PLA powder and 132 g of short fibre in a disintegrator at a speed of 72,000 rpm. After thorough mixing, the fibre/PLA mats were dried at 100 °C for 24 h, pressed in a compression mould preheated to 170 °C and a pressure of 1 MPa for 10 min. (Islam et al, 2010).

**PLA** is a corn-based polymer which has been studied over the past decade. The good mechanical properties obtained from natural fibre reinforced bio composites have led to increased utilization in the automotive industry. Composites from PLA are aesthetically appealing and offer good strength with ease of processing. (Mohanty et. al 2002).

The biggest producer of PLA is NatureWorks. To produce PLA, corn kernels are used. These are milled and processed to extract dextrose, a chemical substance present in the corn kernels. This substance is then fermented by a bacteria or yeast in big vats to produce lactic acid. This acts as a repeating unit to make PLA. (NatureWorks).

The effect of processing parameters on the strength of biodegradable composites was also researched for compression moulded samples of PLA. Parameters such as heating temperature, pressure, number of plies, fibre, and matrix were studied. This research was aimed at developing an optimized manufacturing process to reduce costs and production time of high strength bio composites. Composites of PLA with Jute, flax and cotton were compared. (Rubio-Lopez et al., 2015).

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## 1.4 Factors affecting the use of HF in the production of effective FRP Composite

According to (Ho et al., 2011), factors such as effective fibre/polymer matrix interface adhesion, fibre content and processing parameters/conditions are the main issues that affect the performance/strength properties of RFP composites produced from natural fibres like hemp fibre. **Fibre treatment** is used to achieve better application of hemp fibres in RFP composites. Llignin and hemicellulose content are removed. (Rowell, 1995).

Currently, the two common treatment methods for natural fibres such as hemp include treatments with either alkaline solution or silane. Mild treatment with alkaline to extract cellulose fibres has been shown to create better fibre properties due to better packing and orientation of the chain molecules. (Gassan & Bledzki, 1999).

As seen in figure 1.2 (a & b), there was overall increase in the tensile and flexural strengths of chemically treated HF. However, while there were improved strength properties with alkaline treatment, the silane-treated composites gave almost similar tensile and flexural strengths compared to those of the untreated hemp fibre/PP composites as shown in (Figure. 1.2(b)). This shows that the strength properties of the composites is not significantly influenced by silane treatment.



Figure 1.2 Tensile and flexural strengths of a) alkali and b) silane-treated hemp fiber-reinforced PP composites. Source (Suardana et al., 2011)

The tensile and flexural strengths of the 6% NaOH-treated fibre composite are lower than the 2% and 4% wt. NaOH-treated composites. This may be attributed to the low presence hemicellulose and lignin content in the fibre, since these compounds are responsible for binding the structure of

the fibre, lower presence will result to easy pull out of fibrils. As seen in Table 1.3, the most suitable treatment method for HF to achieve strong fibres and better separation is when HF is treated with 10% wt. NaOH at a temperature of 160 °C for 45 min.

No	NaOH concentration (%)	Maximum process temp. (C)	Hold time (min)	Max. Tensile strength (MPa)	Standard deviation	Kappa no.
1	10	160	15	664	208	5.43
2	10	160	45	677	187	4.09
3	10	180	15	449	121	3.58
4	15	160	15	632	185	5.12
5	15	160	45	532	137	2.80
6	15	180	15	280	101	2.44
Control	_	_	_	607	210	_

Table 1.3 Effect of fibre treatment on the tensile strength and lignin content of hemp fibres. Source: (Pickering et. al, 2007)

It can also be seen from the presented result that alkaline concentration and treatment temperature influences the tensile strength of the resulting fibre. Lower values were recorded at temperatures of 180 °C due to degradation of cellulose at temperatures above 160 °C. In the case of lower tensile strengths at increased treatment with alkaline, this as earlier stated may be attributed to the fact that amount of binding material such as lignin is available in much smaller amount. The amount of lignin available after treatment is represented by the Kappa no.

**Fibre/Polymer matrix interface** plays an important role in the manufacture of FRP composites. A major drawback associated with the use of hemp fibres as peculiar to natural fibres is the high rate of moisture sorption as well as the heterogeneous nature. This gives rise to poor stress transfer between materials (fibre and matrix), which induce weak fibre/polymer matrix interface. Current researches have been trying to establish how this setback can be addressed to improve the adhesive fibre-matrix interface bonding. This draw back can however be easily addressed by surface treatment (Wambua et al., 2003).

Natural fibres are mainly hydrophilic in nature because of the presence of cellulose while polymer matrices are hydrophobic in nature. This gives rise to the incompatibility for bonding because of low wetting of the fibres by the molten polymer. This results in low dispersion, insufficient reinforcement and poor mechanical properties. (Harutun, 2003). To address this issue, chemical modification of the surface of the fibre is used to increase hydrophobicity of the fibre, thereby creating a better adhesion between fibre and polymer matrix (Denis *et. al* 2016 & Malkapuram et al., 2008).

Treatment of HF with 25%. alkaline alone without the addition of a coupling agent was found to increase the young modulus of the resulting composite by 50%. In most cases, treatment of HF with alkaline and a combination of Maleic anhydride (MA) grafted PP (MAPP) has yielded composite with improved tensile strength and young modulus. (Pickering *et. al* 2007)

Improving the fibre/matrix interface can significantly be enhanced using a coupling agent such as Maleic anhydride. Coupling agents are substances that couples or adheres two materials together. They improve the reaction between the fibre and the matrix. (Ho et al., 2011). These chemicals can modify the mechanical properties of the thermoplastic matrix making them more polar. They react with both the polymer and fibre surface to improve adhesion. (Harutun, 2003).

(Pickering et al. 2007) investigated this in his research, using hemp fibre obtained from New Zealand, it was discovered that, strong fibres with good separation and a low lignin content, can average a tensile strength of 47.2 MPa and Young's modulus of 4.88 GPa if allowed a maturity of 114 days, treated with alkali of 10% wt. NaOH solution at a maximum temperature of 160 C for 45 min. and coupled with 3% MAPP through injection moulding process.

Other past researches especially those conducted with matrices of PP and PE, Maleic anhydride (MA) grafting have shown that MAPP significantly enhanced adhesion between fibre/polymer matrix interface, thereby improving tensile strength of the resulting composite material while also ensuring high elastic modulus. (Roumeli et al., 2015).

According to (Ku et al., 2011), adding fibre to polymer matrix will significantly increase the tensile properties of the resulting composites. This is because, these fibres generally have more strength and stiffness values than the polymer matrices. This is buttressed by Table 1.4 shown below, where HF records a tensile strength of 690 MPa. Polymers generally have lower values as shown. The highest value was 78 MPa for Low density polyethylene (LDPE). Other details for High density poly ethylene (HDPE) and polystyrene (PS) are also shown.

Material	Density (g/cm3)	Elongation (%)	Tensile strength (MPa)	Elastic modulus (GPa)	References.
Hemp	1.47	2–4′	690	70	(Nabi & Jog, 1999)
PP	0.899 – 0.920	15–700	26–41.4	0.95–1.77	
LDPE	0.910 - 0.925	90–800	40–78	0.055–0.38	(Holbery &
HDPE	0.94 – 0.96	2.0–130	14.5–38	0.4–1.5	Houston, 2006), (Malkapuram et
PS	10.4 - 1.06	1–2.5	25–69	4–5′	al., 2008)

Table 1.4 Some Properties of hemp and selected polymer matrices

Samples	Tensile strength (MPa)	Elastic modulus (GPa)
РР	31.1(±2.2)	1.3(±0.2)
10HF	26.7(±0.7)	1.6(±0.1)
20HF	28.9(±0.9)	2.3(±0.2)
30HF	28(±1.5)	2.8(±0.2)
40HF	28.1(±0.6)	3.6(±0.2)
50HF	29.3(±2.4)	4.4(0.2)
60HF	24.8(±4.0)	3.6(±0.4)
30HF2.5MAPP	39.7(±2.0)	4.0(±0.2)
30HF 5MAPP	39.3(±2.3)	3.3(±0.2)
40HF 2.5MAPP	36.5(±3.0)	3.0(±0.2)
40HF 5MAPP	40.8(±3.3)	4.3(±0.2)
30HF 2.5MAPOE	34.5(±0.8)	3.2(±0.4)
30HF 5MAPOE	39.0(±1.8	3.0(±0.1)
40HF 2.5MAPOE	35.6(±3.5	3.2(±0.3)
40HF 5MAPOE	37.0(±0.9)	2.7(±0.1)

Table 1.5 Tensile strength of fabricated composite samples. Source: (Etaati et al. 2014).

However, it is generally observed that increasing fibre content produces improvements in the yield strength of composites and furthermore when a coupling agent is used in the formation process. This is all shown in Table 1.5 above.

When reasonable interfacial strength is established, composite strength commonly peaks with fibre contents of 40–55 m% for injection moulded thermoplastic matrix composites with reduction at higher contents explained as being due to poor wetting leading to reduced stress transfer across the fibre–matrix interface and increasing porosity. Stiffness has been found to increase up to higher fibre contents of around 55–65 m% with similar materials, possibly due to less dependency on interfacial strength than composite strength.

In figure 1.3. shown next page, comparison was made between the two coupling agents, MAPP & Maleic anhydride grafted Poly (ethylene octane (MAPOE). Stronger impact was recorded for the addition of MAPP than MAPOE.

Furthermore, (Mieck et al., 2003), was able to show that for a natural fibre like HF which undergoes little plastic deformation, strength properties will most likely be influenced by the presence of defects. This is determined by the fibre length. Fibres are made up of links, with each link having

their own flaws, short single fibres are more likely to have better strength properties than longer fibres because they are made up of less linkages.

However, in the research carried out by (Pickering et al., 2007), FRP composite samples produced from HF of 1.5 mm fibre length had an average strength of 786 MPa, while 10 mm length HF fibre only averaged a strength of 677 MPa but an increase in reinforcing fibre length could improve the composite strength and stiffness of composites with 30 wt.% or 40 wt.% fibre as shown by 10 mm length fibres.



Figure 1.3 The influence of coupling agent type and their content on tensile strength of the noil hemp fibre reinforced polypropylene composites. Source: (Etaati et al., 2014).

It was further gathered from his report that, short fibre composite, have tensile load transferred into a fibre from the matrix through shear at the fibre/matrix interface, as tensile strength tends to be zero at the end of the fibre while it increases along the length. there is thus a need for fibre to have a length greater than a critical length ( $L_c$ ) for the fibre to be able to be broken during tensile loading of a composite. At the critical length, just prior to fracture, the fibre would theoretically only have been carrying half of the load compared to that of a continuous fibre at the same composite strain. However, to allow for efficient reinforcement of a composite, fibre length would be much greater than the critical fibre length to such that most of the fibre could be loaded as if it were a continuous fibre.  $L_c$  can be expressed as follows: (Pickering et al., 2016).

$$\frac{L_c}{d} = \frac{\sigma_f}{2\tau_i} \tag{1.1}$$

Where,

d - is fibre diameter,

 $\sigma_f$  - is tensile strength of fibre,

 $\tau_i$  - is the interfacial strength

# 1.6. Some other factors influencing the mechanical performance of Hemp fibres in FRP composites

Good fibre dispersion is required for FRP composites to decrease voids by ensuring that fibres are fully surrounded by the matrix to promote better interfacial bonding. Processing parameters such as temperature and pressure may influence fibre dispersion while there is even more possibility for longer fibres to have lower dispersion than shorter fibres. The use of additives such as stearic acid has been used in PP and PE to modify dispersion. In addition to this, using twin-screw extruder rather than a single screw extruder has been shown to give better fibre dispersion. However, processing temperature and screw configuration should be carefully aligned to avoid fibre damage and reduction in fibre lengths. (Pickering et al., 2016).

**Mechanical properties of FRP composites generally improves with better fibre orientation**. For best performance, it is important to align the fibres parallel to the loading direction. Fibre orientation can best be improved using injection moulding. These can be achieved by using longer fibres and employing textile adoptive manual alignment of the fibres. Fibres that are randomly oriented have been shown to give good formability and more economical to produce than those with highly directional fibre composites (Zampaloni et al., 2007). However, directional fibres give more mechanical advantage.

**Manufacturing processes** and parameters such as processing temperature, pressure and speed affect the mechanical properties of the composite product. The low processing temperature required to prevent degradation of natural fibres purely provides limitations for the type of polymer matrices to be used. Commonly, extrusion, injection, compression and resin transfer moulding processes are the most common processing methods utilized. (Pickering et al., 2016). Injection moulding NFRP's is regarded as having the best potential for industrial applications (Fowler, Hughes et al., 2006).

Thermoplastic is generally used in beads or pellets form in the extrusion process. Process involves softening of matrix before mixing with the fibre. Single or two rotating screws is used to transport the mixture, which is compressed and forced out of the chamber at a steady rate through a die. Air entrapment may arise due to high screw speed, excessive melt temperatures and fibre breakage.

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While there would be inadequate fibre wetting due to poor mixing if speeds are low. Better mechanical performance and effective fibre dispersion is obtained mainly with the use of Twin screw systems compared to single screw extruders. (Pickering et al., 2016).

**Injection moulding (IM)** is the common manufacturing process used for thermoplastics although may also be employed for thermoset matrices. Natural fibre content is usually limited to 40% because of viscosity requirements. The draw back here is composite strength may be reduced by residual stress in the composite because of pressure gradient, difference in temperature profiles, non-similar expansion of fibre and matrix as well as alignment of the polymer chain. Alignment is often affected by fibre content. (Ho et al., 2012).

**The compression moulding (CM)** is most common where loose chopped fibre or mats of short or long fibre either randomly placed especially for production of thermoplastic matrices. Thermoset matrices can also be used. Pressure and heat are applied after stacking of the fibres which is often alternately with thermoplastic matrix sheets. Precise control of matrix viscosity, pressing and heating is needed, this is to ensure that the matrix is fully impregnated in the space between fibres. Holding time and temperature influence the quality composites that can be produced. (Ho et al., 2012). Temperature and time are the major parameters that need to be carefully monitored. Natural fibres generally loose strength properties at temperatures even as low as 150 to 200 °C, normal for the processing of the matrices. Holding time of 10 mins has been shown to decrease strength properties by about 10%, while 180 °C was found to be the most favourable temperature for a range of mechanical properties. (Mohanty et al., 2000).

The best method for thermoset matrices is the resin transfer moulding (RTM). In this method, a fibre preform in mould is injected with liquid thermoset resin. Factors such as processing temperature and pressure, resin viscosity, mould configuration as well as performance architecture influences the mechanical performance of FRP composite manufacturing method. The limitation of this method is that, unlike glass fibres, natural fibres such as HF are difficult to compact because of low degree of fibre alignment and the closing of inherent cell lumen. (Pickering et al., 2016).

## **1.6** Factors influencing the selection of matrices/properties

Polymeric matrices are the most common matrices currently used for FRP composites due to their light weight and the possibility to process them at low temperature. Natural fibres generally become unstable and degrade at temperatures above 200 °C. To produce composites with natural fibres such as HF as reinforcement, thermoplastic and thermoset polymers have both been used as matrices.

(Holbery & Houston, 2006). However, because of temperature limitations, only polyethylene (PE), polypropylene (PP), polyolefin, polyvinyl chloride (PVC), polystyrene and thermosets that can cure below the stipulated temperature are mostly used. Based on a fully biodegradable polymer composite, polylactic acid PLA has shown comparable/higher strength properties especially when compared to PP. (Faruk et al., 2014).

There is far much growing interest in composites production with thermoplastics matrix. Thermoplastic/matrix composites compared to thermoset/matrix composites, have better toughness, better aptitude to resist chemical attacks, faster processing cycles and, above all, better recyclability. It is possible to recycle thermoplastic/matrix composites simply by re-melting and remoulding these materials while this is impossible for thermoset/matrix composites. (Boufaida et al., 2015).

As shown in figure 1.4, Natural fibre reinforced polymer (NFRP) composites can be produced by a combination of a plant/cellulose fibre with either a thermoplastic or thermosetting matrix material.



Figure 1.4 Natural fibres and matrices for polymer composites. (Sanjay et al., 2010).

# 1.7 Properties of synthetic and natural fibre reinforced polymer composites

Natural fibres such as sisal, hemp and jute fibre composite materials are replacing the glass and carbon fibres owing to their easy availability and cost as well as their advantage as materials to develop biodegradable composite products to help reduce the issue of ecological and environmental problems. (Sanjay et al., 2010).

Of interest and highest consideration for replacing glass in FRP's are materials such as hemp and flax obtained from plant hurd and leaf plants such as sisal. Microbial process known as 'retting' is commonly used to extract the fibre from the plant stem. Retting breaks down the chemical bond holding the plant stem together by decomposition of lignin and hemicellulose. (Thygesen, 2006).

A key focus is on their lower weight which gives better advantage in reducing manufacturing, fabrication, transportation and construction cost. For instance, while glass has a density of 2.6 g/cm<sup>3</sup> that of hemp fibre is only 1.5 g/cm<sup>3</sup>. (Halliwell, Reynolds 2004).

However, compared to glass and some other synthetic fibres, natural fibres have low mechanical properties but nevertheless, can reduce the tool wear when processing, respiratory irritation and may serve as alternatives for artificial fibre composites in the increasing global energy crisis and ecological risks. (Hoi-yan et al., 2009).

The mechanical properties of hemp and other natural fibres is greatly affected by species, weather conditions, yield and exposure. In view of this, accurate analysis of mechanical properties is difficult, typical of the heterogeneous nature of wooden materials. Regardless of all these, the strength of a fibre can accurately be determined by the orientation of micro fibrils. Parallel orientation of micro fibrils gives excellent/stronger fibres. Table 1.6 shows the areas in which natural fibres have distinct advantages over the most commonly utilized synthetic fibre in polymer composites.

Properties	Natural fibers	Glass fibers
Density	Low	Twice that of natural fibers
Cost	Low	Low, but higher than NF
Renewability	Yes	No
Recyclability	Yes	No
Energy consumption	Low	High
Distribution	Wide	wide
CO <sub>2</sub> neutral	Yes	No
Abrasion to machines	No	Yes
Health risk when inhaled	No	Yes
Disposal	Biodegradable	Not biodegradable

Table 1.6 Comparison between natural and glass fibres. Source: (Wambua et al., 2003).

A commonly used Synthetic fibre reinforced polymer (SFRP) composite is Glass fibre reinforced polymer (GFRP) composites. GFRPs are polymer composites produced from plastic matrices reinforced with glass fibres. The use of glass fibres in polymer composites is related to their excellent

properties. In addition to this, they are lightweight, strong, and robust material used in different industries. (Kasama & Nitinat, 2009).

As shown in Table 1.7, we observe some of the strength properties of Glass fibre/PP composites. GFRPs have low cost combined with good mechanical properties. However, they are far less stiff and have lower strength properties compared to carbon fibres. Nevertheless, they are the preferred choice for reinforcing plastics because of their good strength properties, ease of moulding and most especially, their low cost, compared to carbon and aramid. (Ramesh et al., 2013).

Tensile strength (MPa)	E-Modulus (GPa)	Flexural modulus (GPa)	Flexural strength (GPa)	Charpy impact strength (kJ/m2)
88.6	6.2	4.38	60.0	54.12
±7.8	±0.14	±0.38	±5.5	±10.40

Table 1.7 Mechanical properties of glass fibre mat/polypropylene composites. Source (Wambua et al., 2003)

Although natural fibre reinforced polymer composites offer some advantages relating to the use of natural, renewable materials (natural fibres – reinforcing phase), they however have some limitations in strength properties compared to synthetic fibre reinforced composites such as GFRPs because they have lower modulus, lower strength, and relatively poor moisture resistance. Notwithstanding, these composites reflects outstanding and comparable mechanical and dynamic mechanical properties to steel and aluminium, leading to extended applications for special engineering materials such as automotive, aerospace industry and construction structures. (Saba et al., 2015).



Figure 1.5 Tensile strength & Tensile modulus of fibre reinforced polypropylene composites. (Wambua *et al.* 2003).

As seen in figure 1.5, HF/PP composites recorded the highest (52 MPa) tensile strengths. Considering the fibre volume fraction (30%), glass mat polypropylene composites showed a tensile strength of about 32 MPa in a research carried out by (Lee & Jang, 1999). However, higher values (88 MPa) at 22% fibre volume fraction have been reported been reported. Results for measured tensile moduli shows again that HF/PP as well as Kenaf/PP composites recorded the highest modulus of about (6.8 GPa) like that of glass mat/PP (6.2 GPa) as shown earlier (Table 2).

One of the major issues bothering around the utilization of SFRPs is their recyclability and the method of disposal at the end of use. Natural fibres composites on the other hand reflects positive impact on the environmental situation and variety of applications. Even though glass fibre-reinforced plastics have excellent thermal and mechanical properties, problem still lingers with the proper way to dispose these materials after the end of use. (Sanjay et al., 2010).

Similarly, to HFRP composites, GFRP ultimate stress depends on several factors, chief among them being the properties of the reinforcement and matrix and the fibre volume fraction. As for NFRP composites it's been stated in report 1 that, the maximum reinforcement effect may be obtained at fibre volume ratio of 40–50 wt.% level, above this level the strength of the material is decreased by adding more fibre. (Thomason, 2015).

Figure 1.6. shows the relationship between fibre weight fraction for kenaf fibre/PP and the ultimate stress. As seen, peak stress is obtained at fibre weight fraction of about 55% wt.



Figure 1.6 Effect of fibre weight fraction on the tensile strength of KFRP composites. Source: (Thomason, 2015).

## **1.8** Some applications of natural composites

Innovation and technology in the use of Natural fibres composites has led to the creation of opportunity for extensive applications in diverse fields such as consumer goods, low cost housing and civil structures, and for many other common applications where the cost of reinforcements material may need to be considered. The natural fibre reinforced composite has low maintenance requirements, high stress to weight ratio, high corrosion, impact resistance, non-conductive, avoid electrical hazards, reduced cost, easy installation due to light weight and fire retardant. Now, NFCs are used extensively in engineering products for the automotive and construction industries. Other applications where NFCs have gained increase utilization include, the aerospace industry where it is used in the manufacture of tails, wings, propellers; bicycle frames; boat hulls; fishing rods; storage tanks; baseball bats; ice skating boards; door panels; construction material for buildings; marine application and sporting goods industry. Natural fibres offer low-cost engineering applications and can compete with artificial glass fibres. In automotive application, natural fibre composites improve fuel efficiency and reduce emissions because of their light weight. Increasing awareness in sustainable product design, have further led to the natural based fibre materials gaining more popularity to replace synthetic based fibre in the formulation of composites. Composites can be used in components such as pipes for carrying coal dust, helicopter fan blades, desert roof structures and industrial fans. (Sanjay et al., 2010).

The single largest use for hemp fibre reinforced thermoplastic composite now is in the automotive industry. Composites are mainly a combination of hemp fibres and polypropylene or polyester to create nonwoven materials. Blends of HF/PP are produced using hemp mat in molten PP placed in a mould and compressed under pressure. Materials obtained in this nature are used as trunk liner, door panels and window pillar. (Sanjay et al., 2010).

Limited long-term data and unreliable evidence detailing performance of NFRP's currently hampers their widespread application for construction. Prior to research for hemp fibres in NFRP's, hemp has been applied in the construction industry for making hemp/lime concrete, hemp insulation and compressed straw board. (Mohanty 2009).

Natural fibre reinforced plastics (NFRP's) from hemp fibres can easily be formed into structural component such as beams, reinforcing bars and production of racking panels from board materials. NFRP's can also be utilized for self-supporting structural applications and in making corrugated form work for concrete bridge decking. (Grow2Build-data base).

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Hemp fibre thermoplastic composites for architectural and functional application may seem to be the most suitable application for NFRP's produced from hemp fibres as it comprises of application for non-structural architectural and functional areas such as in making door and window frames, skating boards and other decorative uses. NFRP's are easy to form into different products and have high aesthetic appeal making them suitable for a wide range of architectural application. NFRP's also give products suitable for applications where thermal bridging is of interest. Natural fibres are generally known to have good thermal properties. Applications in areas where acoustical dampening properties are of importance is currently being researched. (Grow2Build-data base).

Hemp fibre thermoplastic composites as consumer product can be produced using a combination of different moulding methods. Products such as luggage, musical instruments, sound reinforcing gears and furniture can be produced. The trend now is glass fibres being substituted by hemp fibres as reinforcement fibres in the products where glass fibres are currently being used.

## 2 MATERIALS AND METHODS

## 2.1 Materials

#### 2.1.1 Hemp & polymer

Hemp bundles were supplied by Hempson OÜ. This is because of the profound interest shown by a company to obtain productive ideas from research in FRP composites especially in the ability to suitably apply HFRP composites to produce environmentally friendly building materials. However, these hemp fibres were not clean processed and thus still contained a lot of hurd. It was required to first separate this hurd from the hemp fibre before commencing on production of test samples as this part of the plant is more brittle and will influence negatively on the mechanical properties of the resulting composite. Figure 2.1 shows the hemp as obtained from the company. The polymer used was polypropylene powder and was obtained from ICORENE (ICORENE® PP CO14RM) with a density of 0.9 g/cm<sup>3</sup> and melt flow rate (MFR) of 13 g/ 10 min.



Figure 2.1 Hemp fibre bundles

#### 2.1.2 Coupling agents.

Chemical agents were used to modify the hemp fibres. Agents used include sodium hydroxide NaOH granules (98% concentration), tap water, distilled water, ethanol (96.7% concentration), acetic acid was Lachner and had a concentration of 99.8%, molar mass of 60.05 g/mol while silane (3-Aminopropyl-triethoxy silane) was from SIGMA-ALDRICH with a 98% concentration known as APTES and litmus paper was used. Other equipment used are the press, beakers, stirrer and weighing balance. As a short overview, table 2.1 shows the list of materials used in the modification process and details.

No	Items	Quantity
1	Hemp fibres	150 g
2	NaOH (98% conc.)	5 wt.%
3	Tap water	1000 ml
4	Silane (3-Aminopropyl-triethoxy silane – APTES) 98% conc.	3 wt.%
5	Ethanol (96.7 % conc.)	450 ml
6	Distilled water	50 ml
7	Acetic acid (99.8% conc.)	20 ml

Table 2.1 Modificatio	n data table for	one fibre board material
		one nore bourd material

## 2.2 Methods

The following tables 2.2 and 2.3 suffices for the material calculation. Calculation is based on weight, considering the densities of the composite material - Hemp fibre –  $1.47 \text{ g/cm}^3$  & average density of Polypropylene –  $0.91 \text{ g/cm}^3$ .

Table 2.2. shows the required material estimate to produce hemp fibre reinforced polypropylene composites from hemp fibre with fibre matrix ratio of 60/40 and fibre length approximately 50 mm in length and polypropylene powder. More test samples were produced from hemp fibres of 100 mm and 150 mm lengths. Therefore, total material required for all HF fibre length variations without any form of modification – 1200 g and 1200 g more for modified fibres. Total amount of hemp fibres required to produce samples was 2400 g of hemp fibre. This is as shown in table 2.3 next page.

Test types	Volume (cm)3	No of samples	PP (g)	HF (g)
Flexural	3,20	7	8,15	19,76
Charpy impact	3,20	10	11,65	28,22
Tensile	25,00	7	63,70	154,35
Compressive	4,40	7	11,21	27,17
Water absorption & thickness swelling	10,00	7	25,48	61,74
Air permeability test	40,00	3	43,68	105,84
Total		41	163,87	397,08

#### Table 2.2 Material required for 60/40 (HF/PP) composite test samples

Table 2.3 Gross material estimation

Description	PP (g)	HF (g)	5% NaOH (g)	3% Silane (g)
Total (without) modification	491,61	1191,24	-	-
With modification (approx.)	500	1200,00	60	36
Total	1000	2400,00	60	36

#### 2.2.1 Preparation of hemp fibres

The first process taken was to manually separate the hemp fibrDees from the hurd. A sharp knife was used to cut the hemp from the large fibre bundles while separation was done by hand. The separated fibres were visually inspected for any residual hurd. After removal of hurd, the fibres were cut into lengths of 50 mm, 100 mm and 150 mm using a cutting knife/edge. Considering the size of the test specimen required, a moulding template was prepared. Template was made to a dimension of 150 x 250 mm from which samples will be cut to standard test sizes.

#### 2.2.2 Modification of hemp fibres

150 g of hemp fibres and 5wt% (by weight of hemp fibres) NaOH granules (7.5 g) were weighed with a Mettler Toledo PL202-s having a capacity of 210 grams and increments of 0.01. Fibres were weighed more than 135 grams to cover for material loss while the weighed granules were dissolved in 1000 ml of tap water. The NaOH solution was poured on the hemp fibres until completely submerged and left for about 30 mins at room temperature. Fibres were drained after 30 mins and washed in tap water. A litmus paper was used to ascertain that there were no residual alkaline, this Was done until a pH of 7 (neutral) was obtained. The washed fibres were then placed in the oven at a temperature of 80 °C for 24 h to dry. To complete modification, silane was used. First 3 wt% (by weight of the hemp fibre) Silane was measured, this was about 4.05 g for 135 g of hemp fibres. For silane hydrolisation, the solution of ethanol and distilled water was made in a beaker at ratio of 9:1 (ethanol: water). Silane was poured in the solution and then steered for 30 mins to activate the silane solution. 20 ml of Acetic acid was used to neutralize the solution before stirring to obtain a pH of 7 using the litmus paper. Solution was poured on the hemp and the treated fibres were oven dried at a temperature of 80 °C for 24 h to remove moisture that may affect the quality of the final product. Figures 2.3 shows the fibre modification process.



Figure 2.2 Modification of hemp fibre

#### 2.2.3 Production of test specimens

The production process for unmodified/modified hemp fibre composite is as shown in figure 2.4. The aim was to allow easy production of the board by compacting the fibres into boards because the fibres were cumbersome to handle. First, fibres were cleaned and cut to lengths. 135 g of hemp fibre was then measured using the weighing balance (Mettler Toledo PL202-s) and then soaked in water for 10 min.

The water was drained, and the hemp fibres were aligned on the template as shown and cold pressed for 10 min at a pressure of 1.65 MPa based on the calibration of the hydraulic press used to remove excess moisture. The formed hemp fibre board was removed from the template and transferred to an oven to dry at a temperature of 80 °C for 24 h to remove the remaining bound moisture.

The procedure for modified and unmodified fibre boards were the same. Polypropylene powder was weighed to the ratio 60/40 wt.%. HF/PP. Since the fibre boards weighed 135 g, the amount of PP powder was 54.6 g.

The weighed PP powder was divided into two equal amounts. The first part was uniformly distributed to the base of the template. The formed (oven dried) hemp fibre board (modified/unmodified fibre) was then placed in the template and then hot pressed.

#### **Pressing procedure**

- ✓ Hot press was pre-heated to a temperature of 190 °C.
- Prepared mixture was placed in the press, which was closed and maintained for 15 min without pressure. This was done to allow flow of the polymer matrix into the hemp fibre board.
- ✓ Maximum pressure was applied after 15 min for 10 min while temperature was maintained between 190 °C and 210 °C.
- $\checkmark$  This procedure was repeated for the other face of the board.

Figure 2.4 shows the schematic of the board production process while table 2.4 shows the board variants produced.


Figure 2.3 Schematic of the hemp fibre board reinforced polypropylene production

S/N	Hemp fiber boards (fiber length) mm	Hemp fiber wt. %	PP powder wt. %
2.	50	60	40
3.	100	60	40
4.	150	60	40

	Table 2.4.	HFRP va	ariants	produced
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# 2.3 Testing

A total of 30 composite boards was produced across three fibre length variations (10 boards for each lengths). These samples were cut to standard test sizes.

The evaluated test is as detailed in table 2.5 below. Strength properties of these boards was evaluated and then compared. Fibres were modified, and results were compared with that of boards from unmodified fibres. Specimens are designated as shown in table 2.6.

No	Test	Method	Description	No of samples
1	Tensile test	ISO 527-4:1997	250 x 25 x 4 mm	8
2	Compressive properties	EN ISO 14126:1999	110 x 10 x 4 mm	7
3	Flexural test	EN ISO 14125:2000	80 x 10 x 4 mm	7-10
4	Charpy impact test	EN ISO 179-1:2000	80 x 10 x 4 mm	10
5	Water absorption and thickness swelling test	EN 317:1993	50 x 50 x 4 mm	7
6	Air permeability	EN 12114:2000	14:2000 100 x 100 x 4 mm	
7	FTIR	Laboratory method	Small piece was cut from the samples using a scapular	-

Table 2.5 Test plan

Table 2.6 Sample description

No	Specimen	Description					
1	S50	Hemp fibre reinforced polypropylene composite (50 mm long hemp fibre)					
2	S50M	Modified Hemp fibre reinforced polypropylene composite (50 mm long hemp fibre)					
3	S100	Hemp fibre reinforced polypropylene composite (100 mm long hemp fibre)					
4	S100M	Modified Hemp fibre reinforced polypropylene composite (100 mm long hemp fibre)					
5	S150	Hemp fibre reinforced polypropylene composite (150 mm long hemp fibre)					
6	S150M	Modified Hemp fibre reinforced polypropylene composite (150 mm long hemp fibre)					

#### 2.3.1 Tensile test

Tensile test was performed according to EN - ISO 527-4. Per each variable, 8 specimens were tested and a total of 48 specimen for all variants. Test specimen had a dimension of 150 mm x 25 mm as shown in Figure 2.5, while the thickness of each sample was taken and recorded before testing.



Figure 2.4 Tensile test specimen

Testing was conducted using the Instron 5688 at a temperature of 23 °C, relative humidity of 20% and test rate of 5 mm/min. In performing the test, specimen was placed between two grips and load was applied until failure.

The tensile stress ( $\sigma$ ) and strain ( $\varepsilon$ ) of the specimens was calculated as shown:

$$\sigma = \frac{F}{A'} \quad \varepsilon = \frac{\Delta L_0}{L_0'},\tag{2.1}$$

Where

 $\sigma$  – is the tensile stress value, MPa;

F – is the measured force concerned, N;

A – is the initial cross-sectional area, mm<sup>2</sup>;

 $\epsilon$  – is the strain value in question, expressed as a dimensionless ratio, or in percentage;

 $L_0$  – is the gauge length of the test specimen, mm;

 $\Delta L_0$  – is the increase in the specimen length between the gauge marks, mm;

Furthermore, the nominal strain ( $\varepsilon_t$ ) and Young's modulus of elasticity ( $E_t$ ) was calculated as shown below.

$$\varepsilon_t = \frac{\Delta L}{L},\tag{2.2}$$

$$E_t = \frac{\sigma^2 - \sigma^1}{\varepsilon^2 - \varepsilon^{1'}} \tag{2.3}$$

#### Where,

- $\varepsilon_t$  nominal tensile strain, expressed as a dimensionless ratio or percentage, %;
- L- initial distance between grips, mm;
- $\Delta L$  increase of the distance between grips, mm;
- $E_t$  is Young's modulus of elasticity, MPa;
- $\sigma_1$  is the stress; 0.0005 mm/mm 0.0025 mm/mm;
- $\sigma_2$  is the stress;
- $\varepsilon_1$  is the strain;
- $\varepsilon_2$  is the strain;
- $\Delta \sigma$  Difference in applied tensile stress between the two strain points, MPa;
- $\Delta \varepsilon$  Difference between the two strain points

#### 2.3.2 Compressive test

This test was carried out according to EN-ISO 14126 test standard for composites. The test specimen measured  $110 \pm 1$  mm in length and  $10 \pm 0.5$  mm in width. Prior to testing, all specimens were covered 50 mm from each end with veneer sheets about 2 mm in thickness as shown in Figure 2.6 using a polyvinyl acetate (PVA) glue. The veneer sheet had a dimension of 50 mm by 10 mm, leaving a gap of 10 mm at the middle. 7 samples per variable were tested and a total of 42 samples were tested. The testing was carried out using the same machine as the tensile test. Prior to testing, all samples were measured for thickness. Test speed was 1 mm/min $\pm$  0.5 mm/min.



Figure 2.5 Compressive test specimen

The compressive strength  $\sigma$ cM and compressive modulus *Ec* were calculated using the below equation:

$$\sigma cM = \frac{F_{max}}{bh},\tag{2.4}$$

$$E_c = \frac{\sigma_c^{n^*} - \sigma_c'}{\varepsilon_c^- - \varepsilon_c},\tag{2.5}$$

Where

Fmax – is the maximum load, N;

b – is the width, in mm, of the test specimen;

h – is the thickness, mm, of the test specimen;

 $\sigma_c$ " – is the compressive stress at  $\varepsilon_c$ "= 0.0025, MPa;

 $\sigma_c$  ' – is the compressive stress at  $\varepsilon_c$  '= 0.0005, MPa;

#### 2.2.3 Flexural test

EN-ISO 14125 was used as the test standard for the flexural test. 10 specimens were used for all variables except the hemp modified variables of fibre lengths 150 mm and 100 mm where 7 specimens were used due to some defaults with 3 specimens. The dimension of specimens was 80 mm in length and 10 mm in width as shown in figure 2.7, while the thickness of each specimen was measured before commencing the test.



Figure 2.6 Flexural test specimen

This test was carried out using the Instron 5688 machine at standard laboratory atmosphere of 23 °C and 20% relative humidity.

The following equation show the calculation for the flexural stress parameters:

$$\sigma_f = \frac{3FL}{2bh^2},\tag{2.6}$$

#### Where

 $\sigma_f$  – is the flexural-stress parameter in question;

F – is the applied force N;

L – is the span (mm);

b – is the width (mm) of the specimen;

h – is the thickness (mm) of the specimen

The following equation was used to calculate the flexural strain parameters:

$$\varepsilon_f = \frac{6sh}{l^2} \times 100\%, \tag{2.7}$$

Where

 $\epsilon_f$  –is the flexural strain parameter in question, expressed as a dimensionless ratio or as a percentage;

s - is the deflection(mm);

h - is the thickness(mm)of the test specimen;

L - is the span(mm)

Deflection was calculated on the given values of the flexural strain = 0.0005 and = 0.0025 using the following equation:

$$S_i = \frac{\varepsilon_{fi} L^2}{6h} \ (i = 1 \text{ or } 2),$$
 (2.8)

Where

 $S_i$  – is one of the deflections, mm;

 $\epsilon_{fi}$  – is the corresponding flexural strain, whose values  $\epsilon$ f1 and  $\epsilon$ f2 are given above;

L – is the span, mm;

h - is the thickness, mm, of the specimen

The flexural modulus was calculated after values of deflection  $S_i$  ( $S_1 \& S_2$ ) have been obtained using the following equation:

$$\varepsilon_f = \frac{\sigma_{f_2} - \sigma_{f_1}}{\varepsilon_{f_2} - \varepsilon_{f_1}},\tag{2.9}$$

Where

 $\sigma_{f1}$  – is the flexural stress, MPa, measured at deflection s<sub>1</sub>;

 $\sigma_{f2}$  – is the flexural stress, MPa, measured at deflection  $s_2$ 

## 2.2.4 Charpy impact test

The Charpy impact test was performed according to EN ISO 179-1:2000 testing standard. Test was to determine the ability of the composite material to withstand impact.

Test specimen included a total of 10 samples per variable and specimens' dimension we: length = 50 mm, width = 10 mm & thickness was on the average 4 - 6 mm. The dimension of each specimen was taken before the test was carried out. A total of 60 specimens were tested. Figure 2.8 shows the test specimen, while figure 2.9 shows the notch type used (Type A notch).



Figure 2.7 Charpy impact test specimen



Figure 2.8 Notch type (Source: EN ISO 179-12000)

Test specimen were conditioned to 23 °C and 50% relative humidity after notching in accordance with ISO 291 for Charpy edgewise impact test with single-notched specimen. Figure 2.10 shows a schematic of this as obtained from the test standard and direction of blow.



Figure 2.9. Charpy impact edge wise schematics (Source: EN ISO 179-12000)

The Charpy impact strength of notched specimens,  $a_{cU}$ , in kilojoules per square metre is calculated using the following equation:

$$a_{cU} = \frac{E_C}{h.b} \times 10^3 \tag{2.10}$$

Where

 $E_c$  is the corrected energy, in joules, absorbed by breaking the test specimen;

*h* is the thickness, in millimetres, of the test specimen;

 $b_N$  is the remaining width, in millimetres, of the test specimen

#### 2.2.5 Water absorption and swelling test

The international test standard used for the water absorption and thickness swelling test was EVS-EN 317:1993.

There were seven test specimens for each variable of composite boards produced and in total 42 specimens were used. As shown in Figure 2.11, the test specimens had a dimension of (50 x 50 mm) while thickness of the specimen was measured using the veneer calliper before the commencement of the test. Wax was first used to cover the edges of the specimen to prevent the absorption of water directly through the edges into the composite material.



50 mm

Figure 2.10 Water absorption and thickness swelling specimen

For the test procedure and apparatus, the mass of the specimens was measured to  $\pm$  0.1 mg. Thickness was taken at 3 points, which were marked, and the average of these thicknesses was used as the initial. Specimens were placed in a container filled with water at a depth approximately 30 cm above the specimens. The P.H value of the water was determined using a litmus paper to ensure that it is neutral. Temperature was maintained at 23 °C  $\pm$  2 °C. The specimens were placed in an upright state and caution was taken to ensure that there was no contact between specimens. After immersion for 24 hrs. Specimens were removed from the water, cleaned using filter paper, weighed and then measured for change in thickness at the marked point from which the initial thicknesses were taken.

The percentage change in mass (C) and thickness (T) of the specimen were calculated based on the initial mass and thickness measured by using the following equations:

$$C = \frac{m_2 - m_1}{m_1} \times 100\%, \tag{2.11}$$

Where

 $m_1$  is the mass of the test specimen, in grams (g), after initial drying and before immersion;  $m_2$  is the mass of the test specimen, in grams (g), after immersion.

$$T = \frac{t_2 - t_1}{t_1} \times 100\%, \tag{2.12}$$

Where

 $t_1$  is the average thickness of the test specimen, in millimetres (mm), after initial drying and before immersion;

 $t_2$  is the average thickness of the test specimen, in millimetres (mm) after immersion.

This process was repeated after 48 hr, 96 hr, 168 hr, 336 hr, 672 hr and 1344 hr of immersion.

## 2.2.6 Air permeability

This test was carried out in accordance with international standard EN 12114:2000. The aim was to determine the applicability of the material as an insulation material and the susceptibility to leakage in the transportation of air or liquid. In addition, to determine the effect of fibre modification on the airflow resistivity of the composite material.

**Test specimen** used for this test had a dimension of 100 mm x 100 mm as shown in table 2.7. Specimens edges were first sealed with specific special air permeability tape called seal flex from tesa to prevent air leakage through the edges as shown in figure 2.12.

Specimen	Mean thickness (mm)	Area (m²)		
50 mm	5,66	0,01		
50MOD	6,49	0,01		
100 mm	5,86	0,01		
100MOD	6,35	0,01		
150 mm	5,74	0,01		
150MOD	6,73	0,01		



Figure 2.11 Air permeability test specimen.

**The apparatus** for the test consists of a metal box, square in shape, a pressure in let with a valve and a manometer to measure pressure difference. An airflow meter, air filter with pressure difference regulator and air compressor. Figure 2.13 shows the apparatus setup schematics for carrying out the air permeability test specimen in place in the test apparatus, while figure 2.14 shows the specimen in the equipment.



Figure 2.12 Equipment's complex scheme for carrying out the air permeability test. Source: (Villu K., 2016).

![](_page_47_Picture_0.jpeg)

Figure 2.13 Air permeability specimen in test box

Pressure was administered through a small pipe through the bottom of the box. Maximum pressure difference  $\Delta p_{max}$  was chosen to be 1000 Pa and minimum  $\Delta p_{min}$  50 Pa. Three pulses of pressure were administered to the specimens and maintained for at least 2 mins. Each pulse produced a pressure difference of 1100 Pa. While some specimens were airtight at this pressure. Further testing was done at pressures of 1000, 652, 425, 277, 181, 118, 77 and 50 Pa for specimens with air flow until there was no airflow recorded. Specimens that are airtight at 1100 Pa (Stage 1) required no further testing at these pressures (Stage 2).

In calculating the second phase test pressures, the following equation was used

$$\Delta p_i = 10^i \, \frac{\log \Delta p_{max} - \log \Delta p_{min}}{N} + \, \log \Delta p_{min} \tag{2.13}$$

Where

 $\Delta p$  – pressure difference, Pa;

N – total number of pressure steps;

*i* – number of pressure steps.

For this test,  $\Delta pmax$ = 1000 Pa,  $\Delta pmin$ =50 Pa as shown in table 2.8.

Number of pressure steps (i)	Pressur steps values, Δpi (Pa)	Maximum pressure difference, Δpmax (Pa)	Minimum pressure difference, Δpmin (Pa) 50		
0	50	1000			
1	70	1000	50		
2	118	1000	50		
3	181	1000	50		
4	277	1000	50		
5	425	1000	50		
6	652	1000	50		
7	1000	1000	50		

Table 2.8. Pressure difference values in Pa for each pressure steps in stage 2.

## 2.2.6 Fourier transform infrared spectroscopy (FTIR)

To ascertain the effectiveness of the fibre modification process and nature of the fibre after modification, Fourier transform infrared spectroscopy (FTIR) was used. This was taken with an Interspectrum FTIR spectrometer (Interspec 200-X) having a specac Attenuated Total Reflection (ATIR) unit using KBr disc method. The range of the spectra was 4000 – 800 cm<sup>-1</sup> and resolution of 4 cm<sup>-1</sup>.

To perform the spectroscopy, thin sheet specimen was cut from unmodified and modified HFRP composites using a scalpel. The specimens were each separately placed under a clamp, the spectra were measured, and the peak points were marked. To measure the fibre spectra, piece of the unmodified fibres and modified fibres were used.

## **3 RESULTS AND DISCUSSION**

## **1.1** Tensile properties

Tensile test was performed as shown in figure 3.1 below. As can be seen sample specimen after testing is also shown with fracture occurring in the middle as clearly seen from the side view. Specimen shown in figure 3.1 is from HFRP composites from 100 mm length modified fibre (S100M). However, all tested samples showed the same features.

![](_page_49_Picture_3.jpeg)

Figure 3.1 Tensile test process with test specimen after testing

The maximum tensile strength of all specimen is depicted in figure 3.2. Generally, the tensile strength of the HFRP composite increases with increase in fibre length but decreases with modification. The best result was obtained with S150 (25 MPa). There was a decrease of 47% in ultimate tensile strength for composites of the same fibre length after modification. However, the biggest change was between S50 and S50M (59% decrease in tensile strength for composites from the modified fibres). Puech et al., 2018 reported a 24.5  $\pm$  0.1 and modulus of 2.6 MPa for untreated hemp fibre of 2 mm length with a fibre matrix loading of 20/80 produced using co-rotating twin screw extruder.

There was no significant difference between the tensile strength of S100 and S50. Tensile strength only increased by 4% when fibre length was increased two times compared to over 20% increase in strength when three times the initial length of 50 mm was used. Also, results show that 18% more strength will be obtained when 150 mm fibres are used in composite than 100 mm. HFRP from modified fibres showed similar result. However, compared to the unmodified fibres, there was a significant 28% increase in strength for S100M compared to S50M and 40% more strength is obtained with S150M. These shows that, if modification were to be appropriately done, fibres of 150 mm length will perform far much better than shorter ones. Tensile strength and Young's modulus of 24,5 MPa and 2,5 GPa were reported by Laurent et. al, 2018 in their research on the Investigation of the impact behaviour of short hemp fibres reinforced polypropylene bio composites through high speed imaging and finite element modelling. Although the average fibre length of hemp used was 2 mm and the PP/HF fraction was 80/20.

While there is currently limited literature regarding the use of a combination of such long fibres and loading up to 60%, previous research (Theresa et. al, 2017) analysed the effect of fibre treatment on the mechanical properties of hemp fibre reinforced polypropylene composite. In that research, hemp fibre was modified with 5%MAPP and 5% NOAH, the resulting tensile strength for a 15% and 30% fibre loading were 18 MPa and 29 MPa respectively. While the untreated fibre gave a result of about 17 MPa and 18 MPa respectively. Comparing this to the obtained result, we can say that the results shown by the composites from unmodified fibres conform with previous research.

![](_page_50_Figure_2.jpeg)

Figure 3.2 Tensile strength of HFRP composites

The Young's modulus of HFRP composite is shown in figure 3.3. As can be observed, the same result is obtained with the Young's modulus as was with the Tensile strength. Composites from 150 mm long fibres gave the best results while the unmodified fibres performed better than modified ones. Also, just as in the case of tensile strength, there was no significant difference in the result of elastic modulus of unmodified fibre composites of 50 mm and 100 mm fibre lengths. Overall, S150 had the highest elastic modulus at approximately 4,5 GPa while the lowest was S50M at 1,8 GPa. There was a 53%, 34% and 38% decrease in tensile modulus of the modified composites across fibre lengths of 50 mm, 100 mm and 150 mm respectively compared to unmodified fibre composites. As shown by the % decrease, the greatest change in strength was between S50 and S50M.

![](_page_51_Figure_1.jpeg)

Figure 3.3 The Young's modulus of HFRP composite

It is pertinent to note that, these results are not exactly unexpected as a recent research by Sepe et al., 2018 on hemp fibre reinforced epoxy composites produced by vacuum infusion process shows that tensile strength does decrease after treatment especially when 5% alkaline solution was used to modify the hemp fibre. A 25% decrease in tensile strength was reported. In addition to this, the tensile modulus was 7% lower after treatment. However, same research also shows that when silane alone was used, the tensile modulus was significantly higher (10% and 15%) compared to that of untreated and alkaline treated fibre composites. The decrease in tensile strength is purely attributed to excessive removal of lignin and hemicellulose while in the case of the modulus, increase for silane treated fibres was attributed to improved bonding between the matrix and fibre. Since there was a decrease in the obtained tensile modulus for this research, the bond between the polymer and matrix for the modified fibre was not good and hence the modification was not effective

# 3.2 Compressive properties

Figure 3.4 (a) shows the test specimen held between grips during the compressive test process. The failure mode is shown in figure 3.4 (b). All test specimen presented the same type of failure mode (delamination).

![](_page_52_Picture_3.jpeg)

![](_page_52_Picture_4.jpeg)

Figure 3.4 Compressive test (a) & failure type (delamination)(b)

The compressive strength of the fibre composites is shown in figure 3.5. As seen from the result, compressive strength was negatively affected by modification of the hemp fibres. The best result was obtained for composites from unmodified fibres of 150 mm (S150) fibre lengths (21 MPa). While composites from modified fibres of 150 mm (S150M) length showed improved compressive strength (18 MPa) than other specimens, it was however still 12 % less than that of S150. The composites from modified fibre of 50 mm length gave the worst result 11 MPa, this translates to a 35% decrease in the compressive strength after fibre modification compared to S50.

If we consider the influence of fibre length on the compressive strength of the HFRP composites, we realize that for the modified fibres, the compressive strength increases with increase in fibre length. There was a 16% increase in compressive strength when the fibre length was increased from

50 mm to 100 mm and 27% increase when we use 150 mm instead of 100 mm modified fibres. 39% more strength will be obtained if we modify the fibres and use 3 times the fibre length of 50 mm.

On the other hand, the results for the unmodified fibres was a bit different, while there was significant increase in compressive strength (16%) for 150 mm compared to 50 mm hemp fibres, there was a decrease of 12% in strength when 100 mm fibres were used instead of 50 mm. There has been no research where such lengths of fibres have been compared to draw up a conclusion regarding influence of such fibre lengths on compressive strength.

![](_page_53_Figure_2.jpeg)

Figure 3.5 The maximum compressive strength of modified and unmodified hemp fibre reinforced polypropylene (HFRP) composite.

The compressive modulus is shown in figure 3.6. Similar result is also seen in compressive modulus, it is observed that the compressive modulus for composites formed from unmodified fibres was higher than that from modified fibres of the same length. The compressive strength for S50, S100 and S150 was 39%, 28% and 29% higher than that of S50M, S100M and S150M respectively. In addition to this, all composites from modified fibres regardless of the length of the fibres all recorded lower compressive modulus compared to composites from unmodified fibres. The biggest decrease was for S50M which as earlier stated was 39% lower than S50.

Overall, the compressive modulus for modified fibres, increases with increase in length of the fibres. S150M recorded the highest compressive modulus, 1,26 GPa which was 27% and 22% more than that of S50M (0,91 GPa) and S100M (0,98 GPa) respectively. On the other hand, same does not

stand for composites from unmodified fibres, though S150 gave the best result (1,76 GPa), S100 was 8% less than S50.

![](_page_54_Figure_1.jpeg)

Figure 3.6 Compressive modulus of HFRP composite from modified and unmodified hemp fibres

# 3.3 Flexural properties

Results from flexural test is shown in figure 3.7. The results were very poor for the modified fibre composites. Flexural strength decreased to about 56% on average after modification. The biggest decrease was in composites from 150 mm fibres. S150 showed 63% more flexural strength that S150M.

Generally, flexural strength increased with increase in fibre length. This is particularly obvious with the unmodified fibre composites. The best result was for S150 at 32,67 MPa. This was 11% higher than that of S100 and 38% more than that shown by S50. There was no significant difference in the flexural strength between S100M (12,24 MPa) and S150M (12,05 MPa). However, both were considerably about 12% more than that of S50M.

Wambua et al., 2003, researched with about 40% by volume of hemp fibre, though no reference was made to fibre modification but a flexural strength of 54 MPa was recorded in this case. However, while the research affirmed that the flexural strength increased with fibre volume ratio, only 40% fibre volume ratio was considered.

![](_page_55_Figure_0.jpeg)

Figure 3.7 Flexural strength of HFRP composites

Figure 3.8 shows the flexural modulus of HFRP composites from modified and unmodified hemp fibres. Results show similarities to flexural strength. Flexural moduli of the modified fibre composites were very low compared to that of the unmodified fibre composites. The highest modulus was recorded by S150 (3 GPa), which was 84% higher than that from S150M (0,48 GPa). S150M had the lowest performance.

![](_page_55_Figure_3.jpeg)

Figure 3.8 Flexural modulus of HFRP composites

Generally, flexural modulus increased with increase in fibre length with over 50% increase at 3 times 50 mm length. On the other hand, 150 mm modified fibres had a 13% decrease in flexural modulus compared to 50 mm long modified fibres.

Overall, these results confirm with some past research that have shown that flexural strength increases with increase in fibre length. Previous researches by (Joseph et al., 2002; Sathishkumar et al., 2012; Thomason et al., 1996) has shown that the flexural properties of the composites can be enhanced by using fibres with higher initial lengths because they can carry higher bending loads. This explains the reason why the overall flexural properties for the HFRP increased with fibre lengths. However, for the modified fibres, the poor result may be associated to the ineffectiveness of the modification which prevented good interaction between polymer and matrix especially in load sharing. This is because a combined treatments of hemp fibre with NaOH and silane has been researched to increase the bonding between fibre surface and matrix leading to improved flexural properties even compared to single treatments with NaOH. However, over extraction of extractives from the hemp fibre will cause a deterioration in the flexural properties of the composite material. (Sood & Dwivedi, 2017).

## 3.4 Charpy impact properties

The Charpy impact strength of all specimens tested is shown in figure 3.9. Generally, the result shows very low impact strength for all the composites specimen tested as non-registered impact strength > 10 Kj/m<sup>2</sup>. Past research has shown impact strength to be >25 Kj/m<sup>2</sup> in composite produced from hemp/kenaf. (Wambua et al., 2003).

As can be observed, the average impact strength was higher for the unmodified specimens. This is not in general agreement with other researches considering the strength properties are expected to increase with modification of hemp fibres because studies have shown that impact resistance of fibre composites depends on interfacial bond strength, matrix and fibre properties. Considering that the matrix and fibre properties are the same, the poor result showed by modified fibres compared to unmodified fibres may be attributed to poor interfacial bonding. This may be that the modification process did not work effectively as expected.

The highest impact strength was from composites of unmodified hemp fibres of 100 mm in length  $6,15 \text{ Kj/m}^2$ . In contrast, composites from modified hemp fibre of the same length showed the lowest impact strength of 3,60 Kj/m<sup>2</sup>. Impact strength was overall higher for unmodified fibre to about an

average of 27% more than modified fibres. The greatest change in strength was recorded for 100 mm fibres, with a 59% reduction in impact strength for modified fibres of the same length. This is really a large decrease as same fibre and matrix have been used, it simply implies the use of a modifier rather the modification process negatively affected the impact strength which led to poor results.

![](_page_57_Figure_1.jpeg)

Figure 3.9 Charpy impact strength of HRFP composites

![](_page_57_Figure_3.jpeg)

a. Unmodified

![](_page_57_Picture_5.jpeg)

b. Modified

Figure 3.10 Impact failure mode for (a) and (b) HFRP composites

Observing figure 3.10, we can see that both modified and unmodified HFRP composites show similar type of failure, hinge break (designated by code H in the test standard). This is same for all samples tested. If we consider the influence of fibre length on the impact strength of the HFRP composites, we can observe that the impact strength of HFRP composites from 100 mm unmodified fibres (S100) was 15% and 25% higher than that of HFRP from 50 mm fibres and 150 mm unmodified fibres respectively. On the other hand, while there was slight significant difference between the impact strength of HFRP from 50 mm (S100M) and 150 mm (S150M), (8% increase in strength for 150 mm fibre length), HFRP from modified hemp fibres of 50 mm had an impact resistance 20% and 15% more than those from S100M and S150M respectively.

If we consider past researches however, objective has mainly been on fibre content not exceeding 50% with best results reported for fibre contents between 40 - 50% by Wambua et al., 2003. Also, most research have been based on hemp fibre lengths not >10 mm while for this research, fibre contents of 60% have been used and the fibre length was 50 mm. Although, a review by Pickering et al., 2015 mentioned that addition of natural fibres such as hemp to PP has been found to show reduce the impact toughness because PP is a tough polymer matrix. In addition to this, acetylation, alkali and silane treatments have been found to have a negative influence on the Charpy impact strength of the composite compared to acrylamide and permanganate fibre treatments.

## 3.5 Water absorption and swelling properties

The water absorption result for a total water immersion time of 1344 hr. (56 days) is shown in figure 3.11. According to standard test, a duration of 672 (28 days) is required for this test. However, specimens were retained in the water for another 28 days (672 hours). As can be seen, after 24 hr. of immersion, the composites from modified fibres gained more weight compared to unmodified fibre composites. There was a 65% increase in weight for S150M and 13% for S150 which showed the least water intake. Generally, unmodified fibres performed better, although, there was pretty much little difference in results between S100 and S100M.

After 672 hr of immersion, S150M had gained 20% more weight. This shows that there was massive water intake. However, it was discovered already as shown in figure 3.11 that most of the S150M samples had the wax used to cover the edge of the specimen came off during the soaking period, this was generally same for the other variants of modified composite fibres.

![](_page_58_Figure_4.jpeg)

Figure 3.11 Water absorption of HFRP composites

![](_page_59_Picture_0.jpeg)

Figure 3.12 Wax removal from specimen edge during soaking.

A closer look at figure 3.11 shows that there is no logical correlation between the length of the fibres and water intake for modified fibre composites. But if we consider that S100M had lower water intake than S50M (8%) and as mentioned and the issue of wax coming off for S150M samples, we could infer that the water intake of all the composites (modified and unmodified) decreases with increase in fibre length. A reference would be to that of S50 and S150 were there was 26% more absorption of water compared to S150M.

In figure 3.13, we can see also that thickness swelling decreases with increase in fibre length. In the first 48 hr., swelling was constant at 14% for S150 while the least change in dimension was for S50M at 11%. In all tested specimen, S50 swelled the most to about 27% the initial thickness. This was 2 % more than that of composite of modified fibre of the same fibre length (S50M) and 7% more than S150. There was no significant difference between the results shown by S100 and S100M while S150M swelled 3 % more than S150. As initially mentioned, this may be because of the wax removal issue. However, previous literature has shown that higher fibre volume fraction may promote more water uptake in the composite. (Pickering et al., 2015).

![](_page_60_Figure_0.jpeg)

Figure 3.13 Thickness swelling of HFRP composite

Considering the additional immersion period as shown in the result, there was no change in thickness for specimen S100 (20%), S150 (20%), S50M (25%) and S150M (23%) which implies that these specimens did not continue to swell after the first 28 days. S50 still swelled an additional 2% while there was just a 1%-dimensional increase for S100M.

However, the water absorption result shows that, all samples continue to absorb moisture even though the thickness swelling was constant. The highest moisture intake (after additional 672 hours) was from specimens S100M and S150M (4% more water intake for both variants). S50M and S150 showed the least water absorption of 2% this may further explain why there was no change in dimension for these specimens. But as earlier mentioned, even though S150M absorbed 4 % more moisture, there was no dimensional change. Overall, S150 performed the best with just 37% increase in dimension and 39% of water absorption. In contrast, the result from S150M was less impressive with almost more than 80% gain in weight and dimension. It can be inferred from the obtained results that modification led to susceptibility of the composite material to water absorption and thickness swelling.

As initially mentioned, the high-water uptake by this composite may have been because of the high fibre content in the composite. Hargitai et al., 2008 in their research with nonwoven fleece of PP fibres using fibre blends in the amount of 30, 40, 50, and 70% hemp by weight discovered that water sorption characteristics of the composite were affected by the fibre content with composite of 70% hemp fibre showing 42% water absorption after about 19 days of immersion.

## 3.6 Air permeability properties

Table 3.1 below shows the mean values of air flow in first and second stage pressure test. As seen from the results of the test, specimens from unmodified hemp fibres showed better results. Of all the 18 samples tested, only composites from hemp fibre of 50 mm length were completely air impermeable at the two stages if pressure test. However, all other samples appeared to be not so airtight. Overall, the non-modified fibre specimens were more airtight compared to the modified specimens. This is concurrent with previous research where it has been analysed that alkalization treatment results in a loss of basis weight and a decrease in air flow resistivity (Nazire et al., 2012). Comparing the composites based on fibre lengths, there does not seem to be a direct relationship between the fibre lengths and the air permeability.

Comparing the air flow of the composites based on fibre lengths, we can observe that the only material not air tight at 425 Pa are S100M and S150M. Looking at the growth rate (S150M/S100M) at 425 Pa, we see that it is constant with that of 652 Pa at 0.9. However, it increases more than 100% to 1.94 at 1000 Pa. Also, the air flow in S150M was twice much compared to S100M. This may be attributed to fiber length. However, this may be inconclusive as the flow at 1000 Pa is 41 % less in S100M compared to S50M.

There is relationship between fibre content and porosity. Even though it has been shown that maximum volume fractions of fibre occur around fibre contents of 50–60 m%, further addition may result in higher porosity. (Pickering et al., 2015). Although, the fibre volume fraction used in this research was 60%, there is no doubt that this may have influenced the airtightness of the composite.

Pressure stage	Test pressure (Pa)	Mean values of air flow of specimen S50 (I/min)	Mean values of air flow of specimen S50M (I/min)	Standard deviation S50M	Mean values of air flow of specimen S100 (I/min)	Standard deviation S100	Mean values of air flow of specimen S100M (I/min)	Standard deviation S100M	Mean values of air flow of specimen S150 (l/min)	Standard deviation S150	Mean values of air flow of specimen S150M (I/min)	Standard deviation S150M
	1100	0	0,3	0,09	0,12	0,20	0,25	0,27	0,09	0,15	0,36	0,12
1	1100	0	0,3	0,09	0,12	0,20	0,25	0,27	0,09	0,15	0,36	0,12
	1100	0	0,3	0,09	0,12	0,20	0,25	0,27	0,09	0,15	0,36	0,12
	50	0	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00
	77	0	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00
	118	0	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00
	181	0	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00
2	277	0	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00	0,00
	425	0	0,00	0,00	0,00	0,00	0,07	0,13	0,00	0,00	0,07	0,12
	652	0	0,08	0,14	0,07	0,12	0,11	0,19	0,00	0,00	0,10	0,17
	1000	0	0,27	0,09	0,11	0,19	0,17	0,29	0,08	0,14	0,32	0,11

Table 3.1 Mean values of air flow in first and second pressure test stages

Inferring from Figure 3.14, it is observed that the trendlines highlighting the growth rate with pressure shows that growth rate for specimens S50M, S100 and S150 is linear. However, it is a nonlinear growth rate for the other specimens (S100M & S150M). If we compare this with the results obtained in Table 3.1, we see that there is a sharp increase in airflow between the pressure difference of 652 Pa and 1000 Pa in specimens S100M & S150M. While as initially mentioned, this may be attributed to the modification, it may also be that the fibre length may be a contributing factor to this as composites from modified hemp fibre lengths of 50 mm showed a linear growth. Another main issue may have been that, the specimens from S100M & S150M are more porous compared to the rest of the other specimen. Porosity may have been affected by low ability of the fibres to compact as well as air being trapped in during the pressing process. As this were from modified fibres, the issue of limited wettability may be irrelevant compared to the results shown by the untreated fibres.

![](_page_63_Figure_1.jpeg)

Figure 3.14 Growth rate of air flows in second pressure test stage.

## 3.7 FTIR properties

The FTIR spectrums for the treated in black and untreated in blue hemp fibres are shown in figure 3.15. As seen, there seem to be no difference between the two spectra. In this regard one might

assume that the modification process nearly did not alter any morphology of the fibre. Although we can see in the region between 3500 to 3000 cm<sup>-1</sup> wide stretched peak which represent the hydrogen bond for water, showing the presence of moisture in both fibres.

![](_page_64_Figure_1.jpeg)

Figure 3.15. FTIR spectra of modified and unmodified hemp fibres

Dasong & Mizi, 2011 investigated the dislocation of natural fibres by Fourier-transform infrared spectroscopy and as can be inferred from the report, major differences in the spectra were obtained between the modified and unmodified hemp fibre for bands below 1500 cm<sup>-1</sup> for this FTIR analysis. Looking at this spectrum, we see 3 significant peaks below the 1500 cm<sup>-1</sup> i.e., 1314 cm<sup>-1</sup> (shows CH2, rocking vibration), 1203 cm<sup>-1</sup> (C -O-C symmetrical stretching) and 1104 cm<sup>-1</sup> (C-C, C-OH, C-H ring and side group vibrations). The low absorbance shown at 1203 cm<sup>-1</sup> represents weak cellulose bond in both fibres. The only difference between these two fibres as initially mentioned would be seen in the first section of the spectrum which appears to show the presence of water and by the fact that there is more stretching of the peak for the unmodified hemp fibre compared to that of the modified fibre, it may be easy to conclude the modified fibre consist of more water than unmodified HF.

In the spectra for HFRP composites shown in figure 3.16, there appears also to be no difference between the spectra of the unmodified and modified hemp fibre composite. Just before the 3000 cm<sup>-1</sup> wavenumber we observe stressing peaks for the modified and unmodified fibre composites. This peak shows hydrogen bond synonymous with water i.e. the presence of water in the

composites. From the absorbance depicted, there seems to be more water in the modified HFRP composite compared to the unmodified one. After 3000 cm<sup>-1</sup> (i.e., between 3000 & 2500 cm<sup>-1</sup>), we observe peaks for polypropylene in both composite variants which are similar and just below 2000 cm<sup>-1</sup> i.e. 1104 cm<sup>-1</sup>, shows silane attachment in the modified fibre composite.

The spectra show that all variants have similar properties within the peaks 1500 – 2500 cm<sup>-1</sup> especially showing the presence of aromatic overtones which suggest there is no change in the C=O stretching vibrations. On the other hand, the peak seems to intensify in the peaks below 1500 cm<sup>-1</sup> for the unmodified fibres compared to modified ones. This suggest the modification affected the C-O-C stretching which as can be seen resulted in the reduction in peak intensity for the modified fibre composite. Theresa et. al, 2017 reported that NaOH modification may have been responsible for this. While a study of Influence of chemical treatments on mechanical properties of hemp fibre reinforced composites by Sepe *et al.*, 2018 also showed a decrease in weak and strong peaks of 1734 cm<sup>-1</sup> and 1373 cm<sup>-1</sup> respectively for hemp fibre composites modified by different concentrations of alkali (1% wt., 5% wt. and 20% wt.) The decrease increases with concentration of NAOH used in treatment. This decrease is ascribed to the removal of a part of the hemicellulose from the fibres surface.

![](_page_65_Figure_2.jpeg)

Figure 3.16 FTIR spectra of modified and unmodified hemp fibre composite.

#### SUMMARY

In summary, the influence of fibre length as well as alkaline and silane modification of hemp fibre on the mechanical behaviour, air permeability and water uptake properties of hemp fibre reinforced polypropylene composite were evaluated. Hemp fibre lengths of 50 mm, 100 mm and 150 mm were used while the modification was carried out firstly in an alkaline solution (NaOH 5% by wt. of the hemp fibre) and then silane (3% by wt. of the hemp fibre). The FTIR analysis showed that the HFRP composites modified with NaOH and silane had more moisture in the fibres compared to composites from untreated fibres. The strength properties were thus poor for these composite

The tensile, flexural, impact, compressive and water uptake results showed that HFRP composite from modified fibers in this research compared unfavorably with that from unmodified fibers. While the air permeability test results were also better for the unmodified fibre composites. For example, HFRP composites with 50 mm unmodified hemp fibres were completely air tight compared to those from the modified fibre of the same length. However, this was studied in previous research and shown to be concurrent with modification as it reduces air flow resistance.

Results have also shown that longer fibres enhance mechanical performance. For instance, tensile strength increased by about 20% and 40% for HFRP composites from unmodified & modified fibers respectively when 150 mm long fibers were used instead of 50 mm fibers. The results from the Charpy impact test however were a bit different. There was no real correlation between fibre length and the impact energy it can withstand. While also the water intake and thickness swelling result overall shows that modification of the fibres led to higher water intake.

In the event of long time use of hemp fibre reinforced composites in pipes and tubes specially to transfer moisture/fluid, using fibre lengths of 100 mm will lead to less excessive loss of material compared to 50 & 150 mm fibres. For instance, after over 50 days, water uptake and thickness swelling were about 22% and 65% respectively for both the unmodified and modified hemp fibre composites. While the issue of air tightness may be negligible depending on the pressure of air flow, but shorter fibres will perform better in this regard.

In conclusion, further research on the use of HFRP composite is necessary in this regard. Fibre diameters were not uniform, and this could have impacted the results of the mechanical properties. Alignment of fibres was randomly performed, and press pressure was entirely limited to 1.65 MPa which was very low in accurately compacting the polymer and matrix.

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## KOKKUVÕTE

Magistritöös uuriti kanepi kiudude pikkuse mõju ja naatriumhüdroksiidiga (NaOH) ning silaaniga modifitseerimise mõju kanepikiududega armeeritud polüpropüleenist komposiitide mehaanilistele omadustele, õhuläbilaskvusele ning vee imavusele. Töös kasutati kanepikiude pikkistega 50 mm, 100 mm ja 150 mm. Kiudusid modifitseeriti esmalt 5% (kanepikiudude massist) naatriumhüdroksiidi lahuses ja seejärel 3% (kanepikiudude massist) silaani lahusega. FTIR spektroskoopia tulemused näitasid, et modifitseeritud kanepikiududega armeeritud komposiiditides oli rohkem niiskust sees, kui modifitseerimata kiududega komposiitides. Seetõttu olid ka tugevusomaduses modifitseeritud kiududega komposiitides.

Tõmbe, painde, löögi, surve ja veeimavuse tulemused näitasid, et modifitseeritud kanepikiududega komposiidid näitasid halvemaid tulemusi kõikides katsetes võrreldes modifitseerimata kanepikiududega komposiitidega. 50mm pikkusega modifitseerimata kanepikiududega komposiidid olid täiesti õhukindlad võrreldes samade pikkade modifitseeritud kiududega komposiitidega. Ka varasemad uurimused olid näidanud, et modifitseerimine vähendab õhuläbilaskvust.

Tulemused näitasid ka seda, et pikemad kiud suurendasid mehaanilist tugevust. Tõmbetugevus suurenes vastavalt 20% ja 40% nii modifitseerimata ja ka modifitseeritud kanepikiududega komposiitidel, kui 150mm pikkuseid kiude kasutati 50 mm kiudude asemel. Löögikatse tulemused erinesid teistest katsetest. Löögikatsetes ei olnud täpset seost kiudude pikkuse ja löögienergia vahel. Veeimavus ja pundumise katse näitasid, et kanepikiudude modifitseerimine suurendas veeimavust.

100 mm pikkusega kanepikiududega armeeritud komposiidist torude pikaaegne kasutamine vedelike transportimiseks on vastupidavamad kui 50 ja 150 mm kiududega komposiidid. Tulemused näitasid, et peale 50 päeva vees leotamist kasvas veeimavus modifitseerimata ja modifitseeritud kanepikiududega komposiitidel vastavalt 22% ja 65%. Õhuläbilaskvuses olid paremad tulemused lühemate kiududega armeeritud komposiitidel ning see oleneb ka kasutatavast õhusurvest.

Kokkuvõtteks võib öelda, et edasine uuring kanepikiududega komposiite arendamiseks on vajalik. Töös kasutatud kanepikiudude diameeter ei olnud ühtlane ja see mõjutas ka mehaanilisi omadus. Kiudude orienteerimine oli juhuslik ja pressimise surve (1.65 MPa) oli liiga madal, et kiude piisavalt ühildada polümeerse maatriksiga.

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#### **APPENDICES**

# **Appendix 1 Tensile test results**

Specimen	Thickness (mm)	Maximum load (N)	Tensile stress at Maximum load (MPa)	Modulus of Elasticity (GPa)
50mm	5,09	2485,67	19,73	3,73
50MOD	5,75	1151,69	8,04	1,76
100mm	5,04	2593,34	20,58	3,75
100MOD	6,03	1681,06	11,19	2,49
150mm	5,02	3128,34	25,03	4,48
150MOD	5,86	1947,15	13,34	2,76

Table 0.1 Tensile stress results

Table 0.2 Tensile stress at maximum load

No	Unmodified	Tensile stress at Maximum load (MPa)	STD.	Modified	Tensile stress at Maximum load (MPa)	STD.
1	50mm	19,73	5,63	50MOD	8,04	1,73
2	100mm	20,58	4,64	100MOD	11,19	2,13
3	150mm	25,03	6,07	150MOD	13,34	2,72

Table 0.3 Modulus of elasticity

No	Unmodified	Modulus of Elasticity (GPa)	STD.	Modified	Modulus of Elasticity (GPa)	STD.
1	50mm	3,73	0,92	50MOD	1,76	0,37
2	100mm	3,75	0,77	100MOD	2,49	0,39
3	150mm	4,48	0,87	150MOD	2,76	0,44

# **Appendix 2 Compressive test results**

Table 0.4 Compressive stress results

Specimen	Av. Maximum compressive stress (MPa)	STD	Av. Modulus of Elasticity GPa	STD			
	l	Unmodified					
50mm	17,58	6,49	1,49	0,29			
100mm	15,47	6,13	1,37	0,48			
150mm	20,99	5,33	1,76	0,09			
	Modified						
50mm	11,37	6,19	0,91	0,24			
100mm	13,62	4,05	0,98	0,37			
150mm	18,55	6,62	1,26	0,15			

## **Appendix 3 Flexural test results**

No	Maximum Compressive load (N)	Flexural stress (MPa)	Flexural modulus (MPa)	Compressive extension at Maximum Compressive load (mm)
1	45,06	16,63	751,55	3,36
2	24,61	8,13	561,48	3,89
3	63,26	22,29	974,88	1,86
4	109,10	43,27	4783,58	1,93
5	48,50	19,24	902,21	4,83
6	130,81	49,83	3356,64	2,26
7	26,08	8,59	126,8	6,29
8	22,57	8,20	209,04	4,43
9	35,00	15,08	783,8	4,36
10	31,46	10,32	736,26	2,89
Mean	53,65	20,16	1318,62	3,61
STD	37,46	14,82	1513,74	1,42

Table 0.5 Flexural test results for HFRP composite with 50 mm fibre length

Table 0.6 Flexural test results for HFRP composite with 100 mm fibre length

No	Maximum Compressive load (N)	Flexural stress (MPa)	Flexural modulus (MPa)	Compressive extension at Maximum Compressive load (mm)
1	62,91	20,48	208,39	2,26
2	108,56	36,28	2681,2	2,03
3	61,23	20,31	2195,44	3,09
4	40,43	13,02	1727,91	2,63
5	98,79	33,76	1849,22	2,06
6	144,74	51,78	2686,25	2,27
7	71,20	26,59	537,4	4,16
8	46,45	15,46	1283,53	7,79
9	142,87	43,43	2046,98	1,83
Mean	86,35	29,01	1690,70	3,12
STD	39,31	13,21	871,32	1,89

No	Maximum Compressive load (N)	Flexural stress (MPa)	Flexural modulus (MPa)	Compressive extension at Maximum Compressive load (mm)
1	119,41	45,49	3496,17	2,79
2	129,86	45,40	6671,88	1,29
3	127,66	60,25	3796,75	2,53
4	92,05	35,35	2246,05	2,19
5	46,74	19,08	2667,70	2,65
6	68,09	23,72	4005,44	1,56
7	87,97	33,92	3089,80	1,93
8	33,13	14,21	2068,41	1,93
9	50,73	17,54	812,67	4,06
10	77,09	31,72	1217,78	1,66
Mean	83,27	32,67	3007,27	2,26
STD	34,52	14,65	1662,98	0,80

Table 0.7 Flexural test results for HFRP composite with 150 mm fibre length

Table 0.8 Flexural test results for HFRP composite from modified hemp fibres with 50 mm fibre length

No	Maximum Compressive load (N)	Flexural stress (MPa)	Flexural modulus (MPa)	Compressive extension at Maximum Compressive load (mm)
1	36,09	9,62	411,69	5,79
2	44,96	13,33	1647,13	1,36
3	48,23	11,67	296,04	5,69
4	48,45	11,95	722,68	2,86
5	27,73	9,37	214,04	4,59
6	39,40	8,95	236,70	4,16
7	62,82	15,69	884,80	5,13
8	50,28	15,50	928,54	2,66
9	15,23	3,58	62,30	7,89
10	23,05	5,87	140,91	9,06
Mean	39,62	10,55	554,48	4,92
STD	14,36	3,90	493,13	2,36

No	Maximum Compressive load (N)	Flexural stress (MPa)	Flexural modulus (MPa)	Compressive extension at Maximum Compressive load (mm)
1	36,26	7,99	314,05	5 <i>,</i> 46
2	34,19	7,10	101,87	6,96
3	29,28	7,60	142,85	7,33
4	27,29	6,58	164,59	4,89
5	115,21	30,72	2749,51	2,26
6	58,61	14,00	1620,69	2,83
7	46,33	11,68	430,81	5,79
Mean	49,60	12,24	789,20	5,07
STD	30,88	8,59	1013,96	1,93

Table 0.9 Flexural test results for HFRP composite from modified hemp fibres with 100 mm fibre length

Table 0.10 Flexural test results for HFRP composite from modified hemp fibres with 150 mm fibre length.

				Compressive extension at
	Maximum			Maximum
_	Compressive load	Flexural stress	Flexural modulus	Compressive load
S/N	(N)	(MPa)	(MPa)	(mm)
1	26,80	6,16	98,95	8,53
2	44,61	11,07	434,24	5,29
3	45,27	10,61	97,15	10,96
4	54,37	17,19	1070,75	6,26
5	39,89	9,99	523,55	6,06
6	78,32	18,53	866,63	3,66
7	43,57	10,78	281,04	5,33
Mean	47,55	12,05	481,76	6,58
STD	15,88	4,32	372,78	2,42

#### **Appendix 4 Charpy impact test results**

No	h (mm)	Ec (I)	bN (mm)	a <sub>cu</sub> (Ki/mm2)
1	5.50	1.05	8.00	0.02
2	5,10	0,94	8,00	0,02
3	5,00	0,97	8,00	0,02
4	5,60	0,80	8,00	0,02
5	4,90	1,36	8,00	0,04
6	5,30	1,12	8,00	0,03
7	5,90	0,51	8,00	0,01
8	4,90	1,12	8,00	0,03
9	5,50	0,83	8,00	0,02
10	4,90	1,28	8,00	0,03
	5,20			
	6,72			

Table 0.11 Charpy impact test results for HFRP composite from 50 mm long fibres

Table. 0.12 Charpy impact test results for HFRP composite from 100 mm long fibres

No	h (mm)	Ec (J)	bN (mm)	a <sub>cu</sub> (Kj/mm2)
1	5,20	0,97	8,00	0,02
2	5,10	0,78	8,00	0,02
3	5,20	0,92	8,00	0,02
4	5,10	0,64	8,00	0,002
5	5,60	1,37	8,00	0,03
6	5,30	0,64	8,00	0,02
7	5,40	0,77	8,00	0,02
8	5,10	1,38	8,00	0,03
9	5,10	0,91	8,00	0,02
10	4,80	0,65	8,00	0,02
		6,15		
STD				8,38

Table 0.13 Charpy impact test results for HFRP composite from 150 mm long fibres

No	h (mm)	Ec (J)	bN (mm)	a <sub>cu</sub> (Kj/mm2)
1	5,00	0,48	8,00	0,01
2	5,00	0,67	8,00	0,02
3	5,10	1,05	8,00	0,03
4	4,90	0,90	8,00	0,02
5	5,10	0,70	8,00	0,02
6	5,36	0,86	8,00	0,02
7	5,30	1,39	8,00	0,03
8	4,70	0,95	8,00	0,03
9	5,30	0,90	8,00	0,02
10	5,30	1,27	8,00	0,03
		4,93		
STD				5,96

				<u> </u>
No	h (mm)	Ec (I)	bN (mm)	a <sub>cU</sub> (Ki/mm2)
	(1111)	(7)	(1111)	(Kj/11112)
1	6,40	0,57	8,00	0,01
2	6,30	0,62	8,00	0,01
3	5,70	0,90	8,00	0,02
4	5,80	0,95	8,00	0,02
5	5,70	0,90	8,00	0,02
6	5,90	0,58	8,00	0,01
7	5,70	0,93	8,00	0,02
8	6,20	0,71	8,00	0,02
9	5,90	0,54	8,00	0,01
10	5,70	1,12	8,00	0,03
		Mean		4,33

Table 0.14 Charpy impact test results for HFRP composite from modified 50 mm long hemp fibres

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4,58 Table 0.15 Charpy impact test results for HFRP composite from modified 100 mm long hemp fibres

No	h (mm)	Ec (J)	bN (mm)	α <sub>c∪</sub> (Kj/mm2)
1	5,70	0.90	8,00	0,02
2	6,20	0,80	8,00	0,02
3	5,40	0,81	8,00	0,02
4	5,60	0,74	8,00	0,02
5	5,60	0,79	8,00	0,02
6	6,80	1,20	8,00	0,02
7	5,60	1,47	8,00	0,03
8	5,80	0,70	8,00	0,02
9	6,20	0,93	8,00	0,02
10	5,80	0,52	8,00	0,01
Mean				3,60
STD			5,42	

Table 0.16 Charpy impact test results for HFRP composite from modified 150 mm long hemp fibres

	h	Ec	bN	a <sub>cU</sub>
No	(mm)	(L)	(mm)	(Kj/mm2)
1	6,20	1,11	8,00	0,02
2	5,90	0,69	8,00	0,02
3	5,80	0,62	8,00	0,01
4	5,40	1,18	8,00	0,03
5	5,70	1,06	8,00	0,02
6	6,10	1,04	8,00	0,02
7	6,40	0,83	8,00	0,02
8	6,20	0,67	8,00	0,01
9	6,00	0,82	8,00	0,02
10	5,70	0,82	8,00	0,02
		3,90		
		STD		4,45

### Appendix 5 Water absorption and thickness swelling results

Table 0.17 Water absorption results for HFRP from 50 mm long fibres

No	Time hr.	Water absorption	STD
1	24	47%	10%
2	48	53%	9%
3	96	55%	8%
4	168	59%	9%
5	336	62%	9%
6	672	63%	9%
7	1344	66%	10%

Table 0.1818 Water absorption results for HFRP from modified 50 mm long hemp fibres.

No	Time hr.	Water absorption	STD
1	24	54%	22%
2	48	60%	21%
3	96	63%	22%
4	168	65%	21%
5	336	69%	22%
6	672	71%	22%
7	1344	73%	22%

Table 0.1919 Water absorption results for HFRP from 100 mm long fibres

Table 0.20 Water absorption results for HFRP from	۱
modified 100 mm long hemp fibres.	

Νο	Time hr.	Water absorption	STD
1	24	41%	9%
2	48	48%	8%
3	96	51%	7%
4	168	55%	7%
5	336	58%	7%
6	672	61%	6%
7	1344	65%	6%

No	Time hr.	Water absorption	STD
1	24	38%	19%
2	48	49%	16%
3	96	55%	12%
4	168	59%	11%
5	336	61%	12%
6	672	63%	12%
7	1344	67%	12%

Table 0.201 Water absorption results for HFRP from150 mm long fibres

No	Time hr.	Water absorption %	STD
1	24	13%	14%
2	48	22%	13%
3	96	27%	12%
4	168	31%	12%
5	336	35%	12%
6	672	37%	12%
7	1344	39%	13%

Table 0.212 Water absorption results for HFRP from modified 150 mm long hemp fibres.

No	Time hr.	Water absorption	STD
1	24	65%	24%
2	48	72%	22%
3	96	71%	24%
4	168	79%	22%
5	336	82%	21%
6	672	84%	21%
7	1344	88%	22%

Table 0.22 Thickness swelling results for HFRP from 50 mm fibres

Thickness

swelling

STD

Time

hr.

No

1 24 16% 5% 2 48 20% 7% 3 96 23% 6% 4 168 26% 6% 5 336 26% 6% 6 672 27% 6% 7 1344 29% 10% Table 0.23 Thickness swelling results for HFRP from modified 50 mm fibres

No	Time hr.	Thickness swelling	STD
1	24	16%	4%
2	48	19%	4%
3	96	21%	4%
4	168	23%	3%
5	336	23%	4%
6	672	25%	5%
7	1344	25%	5%

Table 0.24 Thickness swelling results for HFRP from 100 mm fibres

No	Time hr.	Thickness swelling	STD
1	24	13%	6%
2	48	15%	4%
3	96	18%	5%
4	168	18%	5%
5	336	19%	5%
6	672	20%	5%
7	1344	20%	5%

Table 0.25 Thickness swelling results for HFRP from modified 100 mm fibres

No	Time hr.	Thickness swelling	STD
1	24	11%	4%
2	48	15%	4%
3	96	18%	5%
4	168	19%	4%
5	336	19%	3%
6	672	20%	4%
7	1344	21%	4%

Table 0.26 Thickness swelling results for HFRP from 150 mm fibres

No	Time hr.	Thickness swelling	STD
1	24	8%	4%
2	48	14%	3%
3	96	18%	5%
4	168	20%	4%
5	336	20%	5%
6	672	20%	4%
7	1344	20%	4%

Table 0.27 Thickness swelling results for HFRP from modified 150 mm fibres

No	Time hr.	Thickness swelling	STD
1	24	15%	5%
2	48	17%	5%
3	96	19%	5%
4	168	21%	5%
5	336	22%	6%
6	672	23%	5%
7	1344	23%	5%