Effect of Dew Retting and Maceration on Fibre Properties of Hemp and Flax in Manitoba

by

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ABSTRACT

Hemp (*Cannabis sativa* L.) and flax (*Linum Usitatissimum* L.) are both bast fibre plants and have historically been used in paper, rope, thread, yarns and other materials. Recently, companies like the Composites Innovation Center (CIC) Inc. of Winnipeg, Manitoba have explored the use of hemp and flax as environmentally sustainable alternatives to glass/carbon fibres in biocomposites. In order to easily extract fibres from hemp and flax stalks, the stalks must first go through a process known as "retting" in which the cellulosic fibres are separated from the outer cuticle and woody core. Various retting methods exist and have an impact on fibre quality and ease of extraction. Dew or field retting is an environmentally appealing retting method that has regained popularity, particularly in Europe, but has not been rigorously studied in Manitoba. During dew retting, bast fibre straw is permitted to lay in the field in swathes and the rain and morning dew grants the necessary moisture while micro-organisms secrete the necessary enzymes to degrade the pectinaceous matrix encasing the natural fibres. As the retting process greatly influences the quality of the end product, methods such as maceration have been applied to bast fibre straw in an attempt to increase retting efficiency. The goal of this research was to determine the effects of dew retting in Manitoba and maceration using flat rollers on flax and hemp fibre properties. The properties investigated were fibre yield, quality of pectin matrix degradation using scanning electron microscopy and single fibre tensile stress. Water retted and unretted hemp and flax were used as means for comparison. Results indicate that dew retting is possible in Manitoba climate and requires further optimization, and maceration using flat rollers has no significant effect on retting efficiency.

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List of Abbreviations

ALA	Alpha Linolenic Acid
ATR	Attenuated Total Reflectance
Au-Pd	Gold-Palladium
CDC	Crop Development Center
CIC	Composites Innovation Center Inc.
ETD	Everhart-Thornley Detector
FTIR	Fourier Transform Infrared Spectroscopy
GLM	General Linear Model
HV	Accelerating/High Voltage
IQR	Interquartile Range
MIM	Manitoba Institute for Materials
PIHP	Plains Industrial Hemp Processing Ltd.
RH	Relative Humidity
SEM	Scanning Electron Microscopy
SOP	Standard Operating Procedure
THC	Delta-9-tetrahydrocannibinol

1 INTRODUCTION

Natural fibres such as flax (*Linum usitatissimum L.)* and hemp (*Cannabis sativa L.)* have been used for thousands of years as a source of material for textiles and other products such as rope, sails and paper. (D E Akin 2013; Andresen and Karg 2011). In order to easily extract natural fibres from bast fibre plants they must undergo a process referred to as retting. Retting is a method of extracting useful fibres from plant material by allowing bacteria, fungi, enzymes or chemicals to break down the pectinaceous matrix that binds the natural fibres together. In the dew retting method, the plant straw is laid in the field for several weeks while the morning dew grants the necessary moisture for indigenous micro-organisms to break down the pectinaceous matrix thereby releasing the natural fibres. This is commonly done in flax and hemp growing areas in Eastern Europe due to its low cost and environmentally friendly nature but has not been rigorously studied in North America. Other methods of retting include water or enzymatic/chemical retting both of which yield better quality fibre than dew retting but have other disadvantages. Water retting is not environmentally sound at an industrial scale due to the large volumes of polluted water and the foul odour produced from the anaerobic process. Furthermore, enzymatic retting is currently too expensive to be economically viable. Since the retting process greatly influences the fibre quality, methods such as maceration prior to retting have been applied in an attempt to increase retting efficiency. Maceration is a pre-treatment of the bast fibre straw with the goal of breaking the outer cuticle layer thereby granting micro-organisms easier access to the straw interior. Currently flax and hemp are grown solely for seed in Manitoba and little of the fibre found within the stalk is being used in industry. The great majority of the straw is currently being burned or chopped and spread over the fields making this waste stream a largely untapped sustainable and renewable resource. Due to rising environmental concerns and the effort of companies such as the Composites Innovation Center (CIC) Inc. of Winnipeg, Manitoba, interest in renewable sources of fibre for use in biocomposites has increased.

The goal of this project was to determine the effects of dew retting in Manitoba and maceration using flat rollers on hemp and flax fibre properties. Hemp and flax fibre properties investigated in this study were whole fibre, long fibre and hurd/shive yield, signs of pectin matrix degradation using scanning electron microscopy (SEM) and single fibre tensile tests. Water retted and unretted hemp and flax straw were also investigated as a means of comparison.

The remainder of this thesis contains the: literature review; materials and methods; results and discussion; and conclusions. The literature review describes the backgrounds of hemp and flax: their importance in Canadian agriculture and their current uses in industrial products; information on natural fibres and current retting methods and treatments for fibre extraction; and finally, the motivations, advantages and obstacles to using natural fibres in biocomposites. The materials and methods section describes the flax and hemp sample collection, retting methods, maceration treatment, fibre extraction and yield measurements, sample preparation, data acquisition as well as outlier determination. The results and discussion section describes the data analyses, the results, discusses their ramifications and suggests what future work should be done. Finally, the conclusions section summarizes the research accomplished during this project, its importance and the final results.

2 LITERATURE REVIEW

2.1 HEMP

Hemp (*Cannabis sativa L.)* has historically been used for the fabrication of textiles, rope, cloth, sails, paper among other products as early as 10 000 years ago (Sisti et al. 2016) and continued to be popular up until the 1800s with the introduction of cotton and polyester. These new, easier to process and less expensive sources for textiles along with the anti-drug movement of the United States ceased hemp production in North America until the 1990s with the development of low delta-9-tetrahydrocannibinol (THC) varieties (MAFRI 2017). In 1998, Canada started permitting the growth of hemp with THC levels below 0.3%, which is no longer considered an illegal narcotic according to Health Canada (MAFRI 2017). As part of the licence requirements for growing hemp, growers are required to have their crops tested by a third party approved by Health Canada to ensure that the THC levels are within allowable limits.

Hemp is currently grown in many countries including China, France, Chile, Russia, Turkey, United States and Canada (Nair et al. 2015). Hemp grows well in Manitoba as it can thrive under a wide variety of climates and soil types. Hemp is also attractive to farmers as it breaks the crop disease cycles that affect cereals and is a lower risk crop than edible beans and sunflowers in the Manitoba area (MAFRI 2017). The varieties of hemp grown in Manitoba include Joey, Canda, Jutta and many more all having unique attributes in terms of height, maturity, seed size, oil content, oil composition and fibre content. Varieties that growers are permitted to grow are updated annually by Health Canada and must be strictly adhered to throughout the growing season (Seed Manitoba 2017).

The main market attraction for hemp is in the grain sector of the hemp industry where it is used for the production of food products such as oil, dehulled hemp seeds(toasted or raw) and protein powder as well as cosmetic products such as shampoo, conditioner and lotions (MAFRI 2017). However, the hemp fibre industry in Manitoba is currently making headway with the activities of companies such as the Composites Innovation Center Inc. (CIC) located in Winnipeg, Manitoba, which works alongside industry and academia and is involved in research concerning natural fibres and the development of biocomposites and other materials (CIC 2017).

Another Manitoban company involved in the hemp fibre industry is Plains Industrial Hemp Processing (PIHP) located in Gilbert Plains, Manitoba. PIHP is the leading producer of hemp fibre and hurd products in North America and produces hemp fibres, matting, insulation, building material, absorbent material, barbecue pellets and animal bedding (PIHP 2017).

Hemp is a bast fibre plant, wherein the fibres are located in the phloem region of the outer layer of the plant stem or "bark"(Paridah et al. 2011; Kaczmar, Pach, and Burgstaller 2011). The outer cuticle layer of bast fibre plants is composed of lipids, waxes and cutin and acts the main barrier against moisture loss and invading pathogens (D E Akin 2013). The inner woody core is referred to as the hurd in the case of hemp. It is composed of sugars (glucose, cellulose, xylose, hemicellulose and pectins) and aromatic compounds (D E Akin 2013). This section acts as the primary support of the plant as well as the structure for water transport in the plant stem and it is currently being used in other applications such as animal bedding, hemp concrete, acoustical ceiling tiles and other products that take advantage of its high absorbance and good thermal and acoustic properties (PIHP 2017). The bast fibres are sometimes referred to as lignocellulosic fibres as they are mainly composed of cellulose microfibrils held tightly together by lignin and hemicellulose, which form elementary fibres with cross-sections measuring roughly 10-30µm. Elementary fibres are bound together in bundles and embedded in a matrix consisting of hemicellulose, lignins and pectins that form technical fibres, which are further covered with more pectin and waxes (Sisti et al. 2016; Paridah et al. 2011; Nair et al. 2015). The bast fibres grow parallel to the stem axis and are produced in the phloem region in the periphery of the stem, between the outer cuticle layer and the inner woody core (Booth et al. 2004; Ribeiro et al. 2015). Like other bast fibre plants such as flax, jute and kenaf, hemp requires a process referred to as retting to degrade the pectinaceous matrix that technical fibres are embedded within to ensure easy extraction and separate the lignocellulosic bast fibres from the outer cuticle layer and inner woody core (Paridah et al. 2011; D E Akin 2013; Jankauskiene et al. 2015).

2.2 FLAX

Flax (*Linum usitatissimum L.*) has historically been used to produce textiles (linen) due to its highly desirable qualities such as strength, water absorption, comfort and feel (Musialak et al. 2008). Linen production has been traced back roughly 10 000 years to both ancient Egypt and Swiss lake dwellings. Linen made its lasting appearance in Europe roughly 2000 years ago through traders and became the predominant material for clothing throughout the Middle Ages until it was brought over to North America by European colonists. Linen continued to be one of the dominant materials for clothing, being grown in North America from as early as 1640 until the 1900s when, similar to hemp, textiles were overrun by the popularity of cotton and the appearance of synthetic fibres. In Europe, the fibre flax industry has also declined but remains a niche market for long, strong fibres used in high-value applications and in cotton/flax fibre blends (D E Akin 2013). However, within North America, the only flax fibre available is procured through the linseed flax industry. Canada is world's largest producer of linseed flax yielding 860 000 tons of linseed in the 2016-17 growing year according to the Flax Council of Canada (D E Akin 2013; FCC 2017).

Linseed flax is shorter and more branched, therefore yields more seed than traditional fibre flax (D E Akin 2013). The seeds contain alpha linolenic acid (ALA) and iodine, both of which are important to the seed's end use. ALA is an essential fatty acid for human nutrition while iodine imparts drying capacity for its use in various industrial products. Commonly grown linseed varieties in Manitoba include Crop Development Center (CDC) Sorrel, CDC Sanctuary, CDC Bethune, Lightning and Hanley. These varieties all have unique attributes, which include seed colour, maturity, lodging resistance, seed size, iodine value and ALA content (Seed Manitoba 2017; FCC 2017). Flax is an attractive crop for farmers for its high return and value in rotation as it breaks disease and insect population cycles common to cereals and other oilseed crops (FCC 2017). Flax seeds are currently being used in the food industry for oils and whole seeds in breads as well as in printing inks, paints and stains (FCC 2017). Roughly 20% of the flax straw leftover from the linseed industry is being used in the pulp and paper industry while much of the remainder (roughly 1 million tons) is still being burned or spread out on the fields as a waste product (D E Akin 2013). Recently however, with the efforts of companies such as the CIC, attention is being brought to using this excess linseed flax fibre for the production of various products such as particle board, horticultural mulch, animal bedding and biocomposites (FCC 2017; D E Akin 2013; Foulk, Akin, and Dodd 2008).

Flax is also a bast fibre plant and therefore the lignocellulosic fibres are found in the phloem region of the stem between the outer cuticle layer and the inner woody core referred to as shive in the case of flax (Foulk, Akin, and Dodd 2008; D E Akin 2013; Hu et al. 2012). Being a bast fibre plant, flax also requires retting to degrade the pectinaceous matrix, which binds the lignocellulosic fibres together to ensure ease of fibre extraction and separation.

2.3 RETTING METHODOLOGIES

Retting is a process applied to bast fibre plants to facilitate lignocellulosic fibre extraction (Paridah et al. 2011). Traditionally, this process involves various micro-organisms that use enzymes to degrade the pectinaceous matrix in which the cellulosic fibres are embedded, although there are more modern techniques that ret material without the use of micro-organisms (D E Akin 2013; Jankauskiene et al. 2015). This pectinaceous matrix is mainly composed of pectins, hemicelluloses and lignin and its degradation by enzymes allows separation of the fibre bundles from the matrix as well as the elementary fibres within the bundles from each other (Booth et al. 2004). The types of microorganisms involved in the retting process are mainly dictated by the type of retting being applied to the stem. Different types of retting include: water retting, dew retting, enzymatic and chemical retting as well as more modern retting techniques such as microwave assisted retting (D E Akin 2013; Nair, Gopu et al. 2013).

Water retting is a retting method in which dried bast fibre straw is immersed in water for a period of one to three weeks depending on the plant type and size of the stalks (Paridah et al. 2011). Historically, dating back to the late Bronze Age and early pre-Roman Iron Age (800-250 B.C.), water retting has been applied to bast fibre straw by submerging the material in rivers, ponds or man-made retting pits dug into the ground (Andresen and Karg 2011). Modern water retting plants in China submerge straw in large aerated tanks kept at optimally warm temperatures for microbial activity (D E Akin 2013; Ruan et al. 2015). During the water retting process, anaerobic bacteria secrete the enzymes that degrade the pectinaceous matrix (Paridah et al. 2011). Water retting can yield high quality fibres but is not environmentally sound at an industrial scale due to the large volume of polluted water and odour produced by the anaerobic fermentation, which also transfers to the fibres themselves (Danny E. Akin et al. 2007; Fila, Manici, and Caputo 2001; Ribeiro et al. 2015; D. E. Akin, Dodd, et al. 2000). The environmental aspect, raising wages and the high cost of drying has caused water retting to be largely replaced with dew retting (G Henriksson et al. 1997).

Modern techniques of retting include water retting with the addition of enzymes such as hemicellulases, endoploygalacturonase or pectinases and chemicals such as calcium chelators (oxalic acid and ethylenediaminetetraacetic acid) to degrade the pectinaceous matrix of bast fibre straw without the use of micro-organisms. These techniques are referred to as enzymatic or chemical retting and are typically done with a mix of both enzymes and chemicals either together or in a sequential process (D E Akin 2013; Gunnar Henriksson et al. 1997; Danny E. Akin et al. 2007). Enzymatic/chemical retting yields high quality fibres with optimal fibre properties in terms of fineness, tensile strength and composite matrix adherence but has not yet reached industry due to high costs (D E Akin 2013; Hu et al. 2012). Spray enzyme retting has been researched to reduce the volume and hence cost of commercial enzymes needed to ret bast fibre straws, but more research is required to optimize the process (D. E. Akin, Dodd, et al. 2000).

Due to rising environmental concerns and constraints, the ancient method of dew or field retting, has regained popularity, particularly in Europe (D. E. Akin, Epps, et al. 2000). During dew retting, a practice that is older than water retting, the straw lays in the field in swathes while rain and morning dew grant the necessary moisture for aerobic fungi and bacteria to thrive and secrete enzymes that degrade the pectin matrix encasing the cellulosic fibres (D. E. Akin et al. 2002; D E Akin 2013; G Henriksson et al. 1997). Past studies have shown that the microflora involved during dew retting is diverse, varies in proportions and is affected by soil type, harvest dates and retting durations in the field (Ribeiro et al. 2015). Studies have also shown that some bacterial/fungal strains present in certain soils have significant cellulase activity, which can have a detrimental effect to the retting process, degrading the lignocellulosic fibres and thereby reducing fibre quality (Fila, Manici, and Caputo 2001). Apart from being an environmentally friendly process dew retting is also a lower cost retting method compared to water retting or modern techniques. However, dew retting generally produces fibres of lower quality than water retting, is dependent on climate (temperature and humidity) during retting and therefore has geographical restrictions in terms of where the process is feasible. The process can be highly variable in terms of fibre quality, requires more time than water retting and occupies field space during the retting period (D E Akin 2013; Fila, Manici, and Caputo 2001; D. E. Akin, Dodd, et al. 2000).

Since the retting process is a key step in the extraction and production of bast plant fibres, ways of optimizing the retting process to produce higher quality fibres, with greater yield and with less time have been investigated. Maceration or crimping is a mechanical pre-treatment of the plant straw prior to retting with the goal of disrupting the outer cuticle layer and thereby granting microbes, enzymes and chemicals faster access to the straw interior thus accelerating the retting process (Foulk, Akin, and Dodd 2001). Studies performed by Henriksson et al. found an increase in retting efficiency when applying a mechanical pre-treatment, which split flax straw open prior to enzyme retting (Gunnar Henriksson et al. 1997). Foulk et al. (2001) refers to unpublished data in which they performed studies comparing crimping using spring-loaded fluted rollers to flat steel rollers and found a significant increase in retting efficiency when using fluted rollers (Foulk, Akin, and Dodd 2001). Consequently, spring-loaded fluted rollers have been used as a mechanical pre-treatment for numerous studies involving retting bast fibre straw (Foulk, Akin, and Dodd 2001; D. E. Akin, Dodd, et al. 2000; D E Akin 2013; Danny E. Akin et al. 2007).

Another method for optimizing dew retting in particular is "turning" in which the swathes are flipped over in the field at a certain time during the retting process. This method is meant to encourage even and proper retting. Proper turning however requires knowledge on the part of the producer of when to flip the swathes and is suspected of being one of the reasons that dew retting is successful in certain regions of Europe, aside from having an appropriate climate (D E Akin 2013).

2.4 NATURAL FIBRES IN COMPOSITES

Rising environmental concerns have resulted in increased interest in using sustainable and renewable sources such as hemp and flax for fibre reinforcements in the composites industry (D E Akin 2013; Zuccarello and Zingales 2017). Natural fibres are currently being used for the production of bio-based composites in various industries such as non-structural components in the automotive, building and furniture industries as well as products such as musical instruments, sporting equipment, packaging and household electrical appliances (Kaczmar, Pach, and Burgstaller 2011; D E Akin 2013; Ribeiro et al. 2015). Natural fibres have numerous advantages over synthetic/carbon based fibres including: coming from a sustainable and renewable source, being biodegradable, they are lower cost, lower weight, have high vibration damping performance and have comparable mechanical properties in terms of fibre strength and stiffness (D E Akin 2013; Kaczmar, Pach, and Burgstaller 2011; Zeng, Mooney, and Sturrock 2015; Virk, Hall, and Summerscales 2012; Moriana et al. 2014). Natural fibres do have some disadvantages including: poor water resistance, susceptibility to rot; and a large variability in fibre yield and mechanical properties. The severity of these disadvantages is influenced by: plant variety, growing season, time of cutting, retting method, and when dew retting: weather during retting and the variability of indigenous soil micro-organisms (Kaczmar, Pach, and Burgstaller 2011; Virk, Hall, and Summerscales 2012; Haag and Mussig 2016; Shah, Nag, and Clifford 2016; Ribeiro et al. 2015; Martin et al. 2013). Other obstacles to industrializing natural fibres in composites include difficulties in natural fibre/composite matrix adhesion, the lack of standards for testing fibre tensile strength and estimating cross-sectional area; and the lack of modelling required to predict composite behaviours based on natural fibre properties (Kaczmar, Pach, and Burgstaller 2011; Shah, Nag, and Clifford 2016; Haag and Mussig 2016). Therefore, more study on natural fibres is required to bridge the gap between natural fibres and industrial structural composites.

3 MATERIALS AND METHODS

3.1 HEMP AND FLAX

The hemp and flax straw materials used for this project were provided through cooperation with the Composites Innovation Center Manitoba Inc. (CIC) of Winnipeg, Manitoba, Canada. The hemp plants grown were of two different varieties and from two different locations: The Joey variety was grown in Roseisle, Manitoba, and the Canda variety in Minto, Manitoba. The Joey hemp from Roseisle and the Canda hemp from Minto were cut and samples for both unretted straw and water retting were gathered on 9 September 2016 and 28 September 2016, respectively. Hemp plants were cut roughly 12 cm from the ground.

The flax was the CDC Sorrel variety and was grown in two different locations: Minto, Manitoba, and Portage la Prairie, Manitoba. The flax from Minto and from Portage were cut and samples for both unretted straw and water retting were gathered on 23 September 2016 and 15 October 2016, respectively. Flax plants were cut roughly 5 cm from the ground. Collected samples were kept in a cool, dry and well-ventilated area.

3.2 MACERATION

Roughly half of the hemp and flax straw used for this project was macerated prior to retting. Maceration of the straw was completed in all fields using the Macerator 6620 (Les Machineries Pronovost Inc.), a local agriculture machine typically used to condition straw for silage that employs flat steel rollers operating at different speeds which flattens and shears the straw. Macerated material for both unretted straw and subsequent water retting studies were also collected from Roseisle, Manitoba; Minto, Manitoba and Portage la Prairie, Manitoba on the same dates mentioned in the previous section.

3.3 DEW RETTING

The hemp and flax straw not collected on the day of cutting was left in the field in swathes varying in height from 18 to 36 cm where it was left undisturbed during dew retting. Roseisle (Joey hemp) and Minto (Canda hemp) straw was left in the field to dew ret until 12 October 2016 and 22 October 2016 granting a total of 33 and 24 days of dew retting, respectively. The Sorrel flax straw from Minto and Portage were left in the field to dew ret until 22 October 2016 and 15

November 2016 granting a total of 30 and 31 days of dew retting respectively. The time of retting was governed by a retting assessment performed by the farmers. Farmers were provided with a standard operating procedure (SOP) for assessing the degree of retting by the CIC to aid them in their assessment. The degree of retting was mainly dictated by colour change (from green to brownish/black) of the hemp and flax straw and ease of fibre separation from the hurd/shive.

The dew retted samples from Roseisle were collected by hand while the samples from Minto and Portage were baled and provided by the CIC. During the time of dew retting, Roseisle received an average rainfall of 1.14 ± 0.70 mm with average minimum and maximum temperatures of 6.0 ± 0.9°C and 18.0 ± 1.2°C respectively, Minto received an average rainfall of 3.08 ± 1.63 mm with average minimum and maximum temperatures of 1.5 ± 0.8°C and 11.4 ± 1.3°C respectively and Portage received an average rainfall of 1.76 ± 0.79 mm with average minimum and maximum temperatures of 1.5 ± 0.4°C and 11.6 ± 0.8°C respectively (Environment Canada 2017).

3.4 WATER RETTING

Samples of dried hemp stalks, each weighing 300g, were prepared from material at each of the growing locations in both macerated and non-macerated condition. The 300g of hemp stalks had to be trimmed to a length of 25 to 40 cm to fit in the container used for water retting, thereby discarding the thin top branched ends. For dried flax stalks, samples of 300g were prepared for both growing locations in non-macerated condition only. Macerated flax material from both locations were not water retted as the straw was quite damaged by the maceration process and therefore was already partially decorticated. It was presumed that water retting this macerated flax straw would result in material that would be nearly impossible to process by hand.

Each 300g sample was processed individually in a 10 gallon “InfuSsion” Mashtun (Brewtech Inc.), after the mashtun was filled with fresh tap water up to the 10-gallon mark. A stainless-steel mesh was placed on top of the straw sample to prevent it from floating above the water line. The hemp and flax straw samples were left undisturbed during water retting. The hemp samples were retted in water for 12 days while the flax straw samples were retted for 8 days. The retting time was based on preliminary tests with fibre separation being the key characteristic. After retting was complete, the straw sample was removed from the water and was permitted to dry for three days under a hood-vent, flipping the straw daily, to dry thoroughly.

3.5 FIBRE DECORTICATION AND YIELD MEASUREMENTS

The decortication method used in this study was based on a method used by Bennet et al. (Bennett, Snell, and Wright 2006) and modified for our use. Fibre decortication was accomplished by first conditioning the hemp/flax straw sample at 21°C at 65% relative humidity (RH) in an environmental chamber for at least 48 hours. Samples were taken of flax and hemp for each combination of: unretted, water-retted and dew-retted; growing location; and maceration condition. These samples were divided into subsamples of 50g each for decortication. Nominally, six subsamples were taken from each sample to be decorticated. The available dew retted hemp and flax straw from Minto provided by the CIC was insufficient for six subsamples resulting in an unbalanced experimental design. Minto dew retted macerated and non-macerated hemp samples consisted of three 50 g subsamples each while the Minto dew retted macerated and non-macerated flax samples consisted of five 50 g subsamples each.

The decortication process used in this study was based on traditional fibre separation methods involving crushing, scutching and hackling (D E Akin 2013). Subsamples were first crushed by passing through a MillMaster Grain Mill (MashMaster Ltd.), which consists of a set of two adjustable stainless-steel fluted rollers. Rollers were adjusted, depending on the thickness of the stalks, to crush the straw without tearing the fibres. The crushed subsamples were then passed through a coarse “Viking Hackle” (Valkyrie Supply Inc.) to remove all remaining hurd/shive from the fibre. Finally, samples were passed through the “Fine Hackle” (Valkyrie Supply Inc.) to separate long fibres from short fibres. Weights of the subsamples were taken after each step of the decortication process and used for post-crusher hurd/shive, whole fibre and long fibre yield calculations.

Post-crusher hurd/shive yield ($Y_{(H)}$) was calculated as:

$$Y_{(H)} = (1 - (\text{Mass after MillMaster Grain Mill/Initial subsample mass})) \times 100\%$$

Whole fibre yield ($Y_{(W)}$) was calculated as:

$$Y_{(W)} = (\text{Mass after “Viking Hackle”/Initial subsample mass}) \times 100\%$$

Long fibre yield ($Y_{(L)}$) was calculated as:

$$Y_{(L)} = (\text{Mass after "Fine Hackle"} / \text{Initial subsample mass}) \times 100\%$$

3.6 TENSILE STRENGTH MEASUREMENTS

Single fibre tensile strength measurements were performed on non-macerated flax and hemp fibres from both growing locations and for unretted, dew retted and water retted samples. Tests were accomplished using the Diastron single fibre testing equipment outfitted with a Linear Extensometer LEX 810 capable of handling loads of up to 10N and a Fibre Micrometer FDAS760 sample holder with Mitutoyo LSM6000-500s laser scanner for rapid analysis of cross sectional data of small fibres. This equipment is the property of CIC and was used under their guidance.

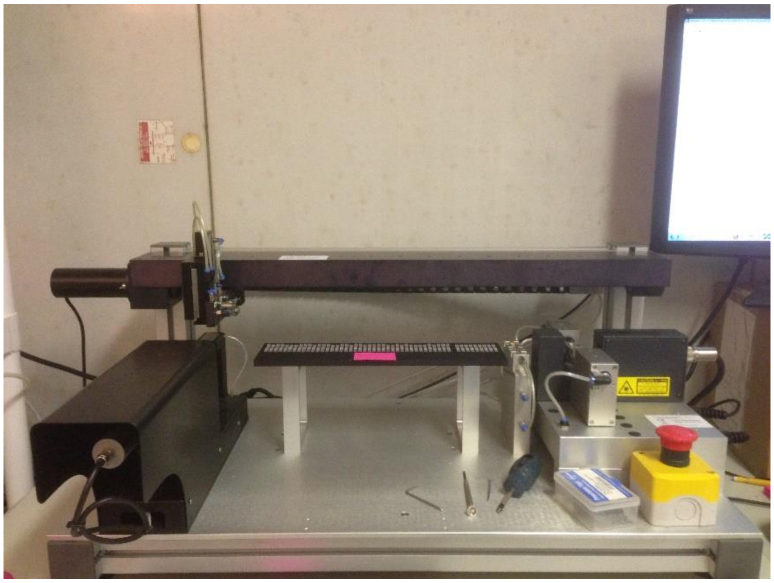

Figure 3.1 TARDIS single fibre testing equipment used in this study

Fibres were tested on 4 mm gauge cassettes; cross sectional analysis was performed with four rotations at one slice and tensile testing was performed with an extension rate of 0.0066 mm/sec with a maximum elongation of 10%. Fibre samples were prepared by hand using tweezers. Prepared samples were as thin as possible and free of frays, loose threads or kinks in a 4 mm section while still being at least 15 mm long to be properly mounted into the cassette. Fibres were attached to sample tabs using a UV cured Acrylated Urethane adhesive (Ultra Light-Weld® 3094 by Dymax Corporation). All testing was done in an environmental chamber set at 23°C and 50% RH. Fifty fibres were prepared and tested for each treatment.

Figure 3.2 Prepared 4mm gauge cassette with flax fibres

3.7 Analyses Performed on Tensile Strength Data

3.7.1 Fibre Cross-Sectional Area

The diameter measurement of each fibre was performed with four rotations at the center point of the 4 mm fibre samples. These four rotations were then overlaid on top of each other producing Scan versus Diameter graphs similar to those shown in Figure 3.3 to Figure 3.6.

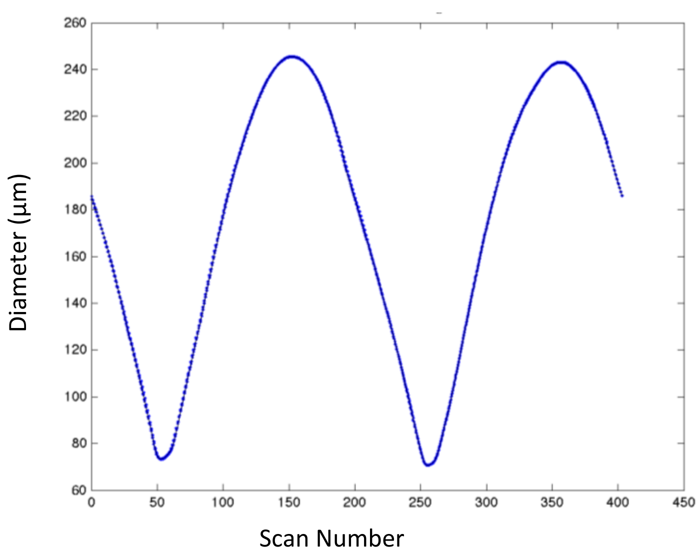

Figure 3.3: Diameter Scan of Flax Fibre: Example 1

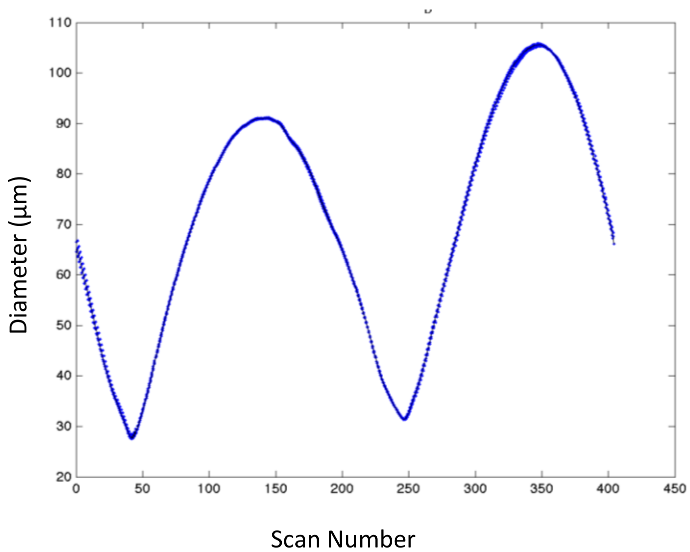

Figure 3.4: Diameter Scan of Flax Fibre: Example 2

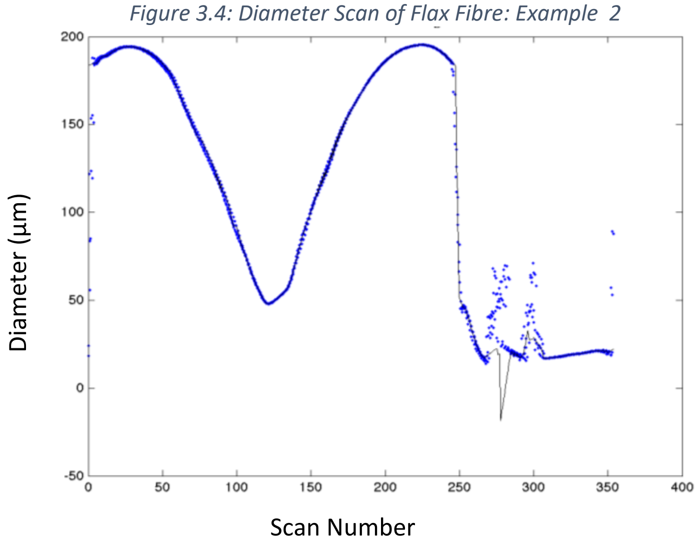

Figure 3.5: Diameter Scan of Flax Fibre: Example 3

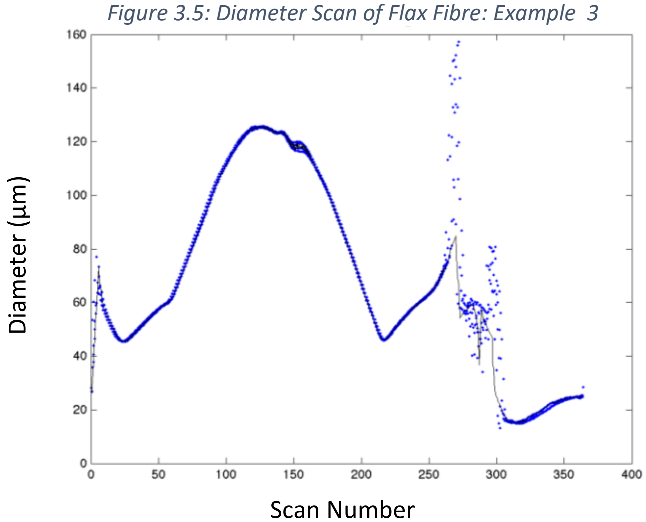

Figure 3.6: Diameter Scan of Flax Fibre: Example 4

It was determined that at least one maximum and one minimum reading was necessary to adequately estimate the fibre cross-sectional area and therefore certain dimensional scans had to be rejected due to unknown interference, possibly a hair or dust. Figure 3.7 and Figure 3.8 are examples of scans that had to be rejected due to not having at least one discernible maximum and minimum reading.

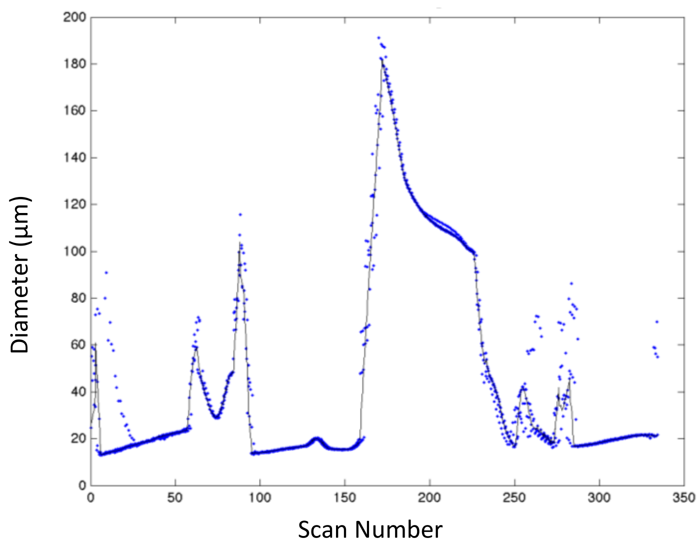

Figure 3.7: Rejected Diameter Scan of Flax Fibre: Example 1

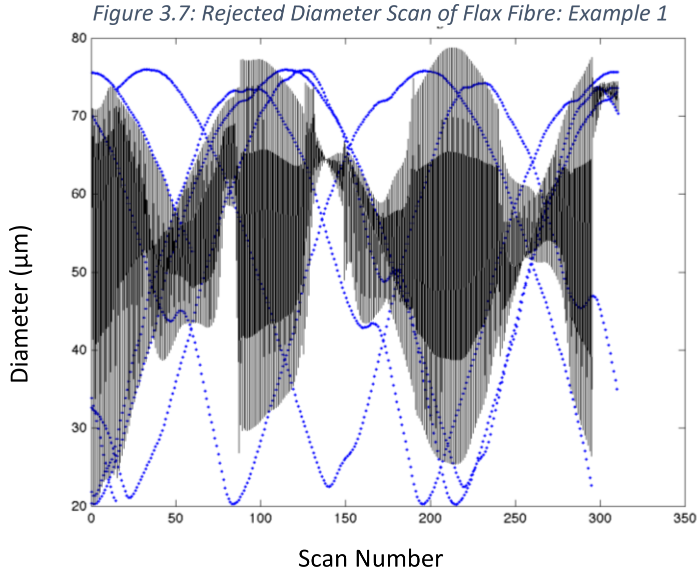

Figure 3.8: Rejected Diameter Scan of Flax Fibre: Example 2

It is known that the cross-sectional shape of technical fibres is varied and the assumed shapes in previous studies is equally varied. (Baley 2002; Del Masto et al. 2017; Liu et al. 2015). Based on recommendations from the CIC it was assumed that each fibre had an elliptical shape using the average of the two maximum readings (if two are present) for the major axis and the average of the two minimum readings (if two are present) for the minor axis to calculate the cross-sectional area (A_c) of each fibre (see Equation 1). With the equipment available it was not possible to measure the cross-sections of the lumens within each technical fibre and therefore the presence of lumens within the technical fibres were not considered when calculating A_c.

$$A_c = \pi ab \qquad \text{(Equation 1)}$$

where a = major axis / 2 and b = minor axis / 2.

Outliers were then removed based on the standard boxplot approach described in Equation 2 (Wilcox 2012).

$$X_i < q_1 - k(q_2 - q_1) \text{ or } X_i > q_2 + k(q_2 - q_1) \qquad \text{(Equation 2)}$$

where X_i is the sample value being tested, q_1, q_2 are the lower and upper quartiles of the samples respectively, and $k = 1.5$. For the areas, the only outliers were samples that had abnormally large cross-sectional areas. The corresponding boxplots for hemp and flax can be found in Figure 3.9 and Figure 3.10 respectively. The number of outliers removed and the total number of samples for hemp and flax are found in ***Table 3-1*** and ***Table 3-2*** respectively.

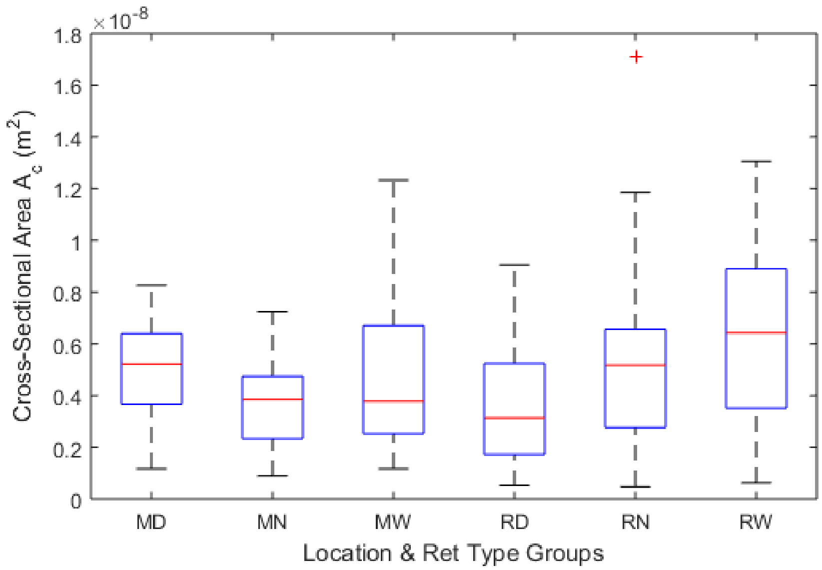

Figure 3.9: Hemp Cross-Sectional Area by Location and Ret Type Groups

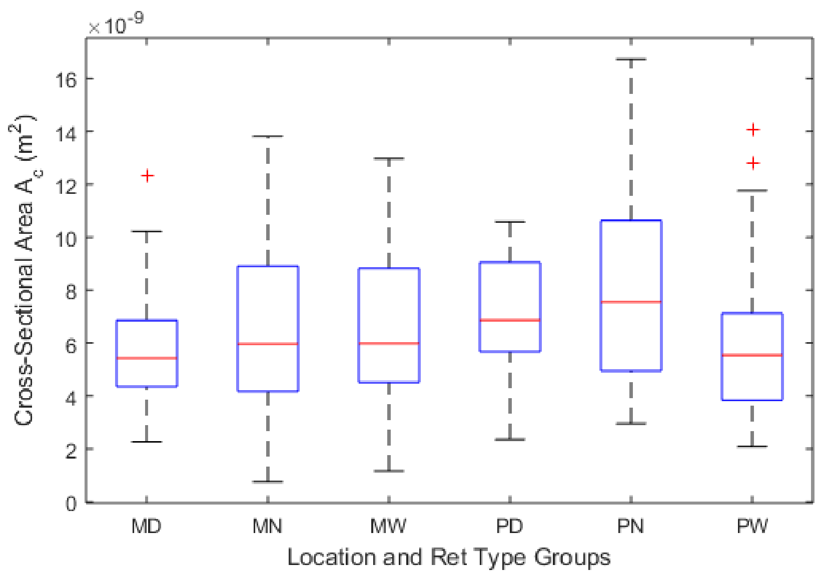

Figure 3.10: Flax Cross-Sectional Area by Location and Ret Type Groups

3.7.2 Single Fibre Tensile Strength

The single fibre tensile strength tests were performed as stated in section 3.6 producing Force (N) versus Strain (% elongation) graphs similar to those shown in Figure 3.11 and Figure 3.12. Certain fibre samples did not break after a maximum elongation of 10% producing graphs similar to Figure 3.13 and therefore were rejected.

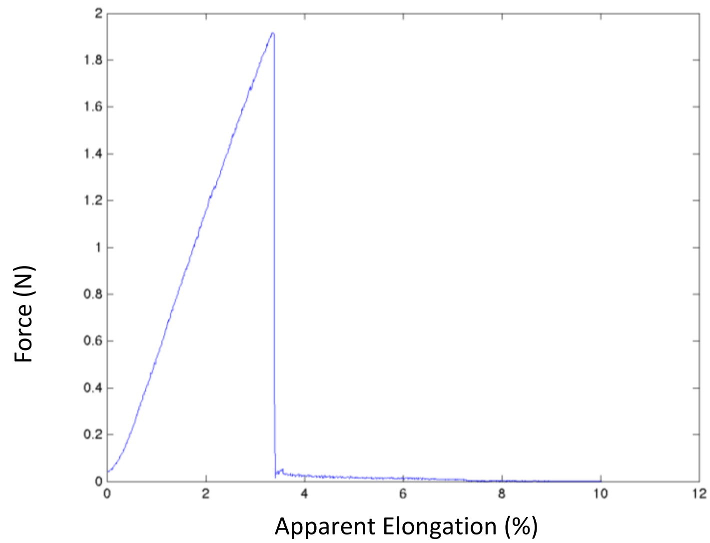

Figure 3.11: Tensile Test of Flax Fibre with One Break

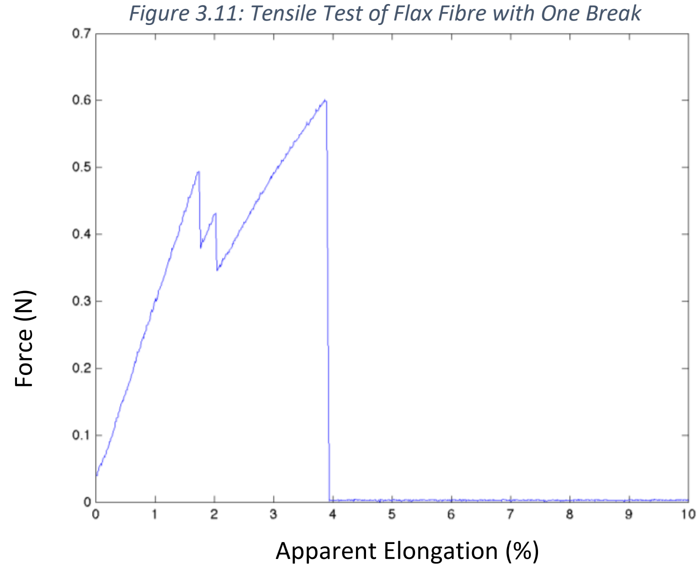

Figure 3.12: Tensile Test of Flax Fibre with Multiple Breaks

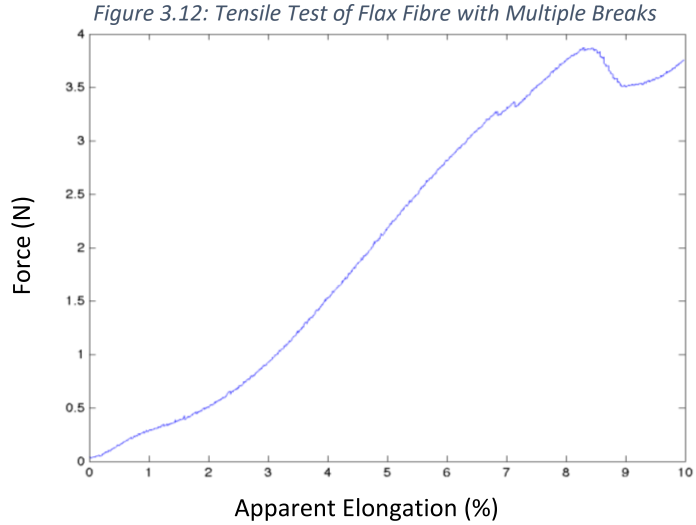

Figure 3.13: Tensile Test of Hemp Fibre without Break

As seen in Figure 3.12, certain samples had multiple breaks instead of having only one final break as seen in Figure 3.11. It is assumed that because the fibre samples are technical fibres consisting of multiple elementary fibres (as confirmed by SEM analysis of the fibre samples, see Section 4.2) the appearance of multiple breaks are caused by elementary fibres breaking earlier than the entire technical fibre. Due to early breaks, the estimated cross-sectional area A_C is no longer accurate because the area of the technical fibre has decreased due to the break of elementary fibres. Based on this assumption, it was determined that the force at the first break (F_i) would be used to calculate initial yield stress (σ_i) according to Equation 3.

$$\sigma_i = {}^{F_i}/_{A_C} \qquad \text{(Equation 3)}$$

Outliers were then removed based on the IQR (Wilcox 2012) of the ratio of the strain at first break ε_i to the strain at the final break ε_f in order to remove samples that had early partial breaks (see Equation 4).

$$q = {}^{\varepsilon_i}/_{\varepsilon_f} \qquad \text{(Equation 4)}$$

These early partial breaks were removed due to the abnormally low stress reading of that sample compared to the rest of the sample population. Figure 3.14 is an example of an outlier removed for this reason. Outliers were then removed based on the standard boxplot approach described in Equation 2 in Section 3.7.1. The corresponding boxplots for hemp and flax can be found in Figure 3.15 and Figure 3.16 respectively. The number of outliers removed based on the IQR of q for hemp and flax can be found in **Table 3-1** and **Table 3-2** respectively. The outlier removal

computations were done using the entire set of data for each fibre, location and ret type independently for A_c and q. No sample was an outlier in both cases.

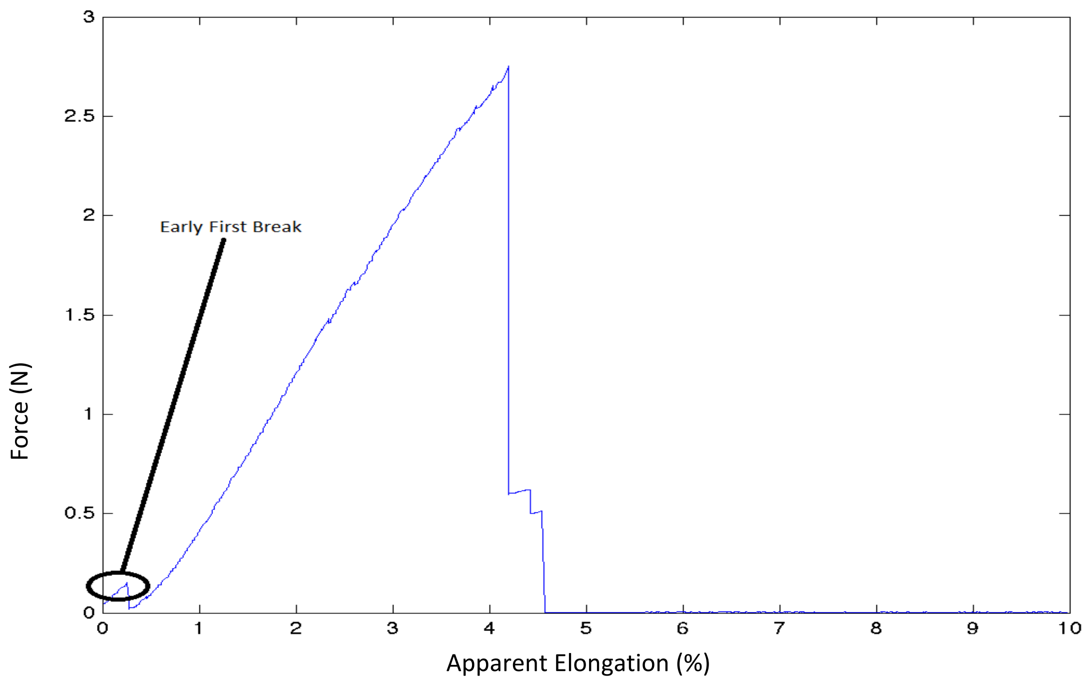

Figure 3.14: Tensile Test of Flax Fibre with Early Break

Table 3-1: Number of Outliers Removed from Hemp Fibres

	Total samples	q Outliers removed	A_c Outliers removed
Minto Dew Retted (MD)	30	3	0
Minto Unretted (MN)	35	8	0
Minto Water Retted (MW)	30	3	0
Roseisle Dew Retted (RD)	39	7	0
Roseisle Unretted (RN)	32	7	1
Roseisle Water Retted (RW)	41	10	0

q = ratio of the apparent strain at first break to the apparent strain at final break
A_c = estimated cross-section area

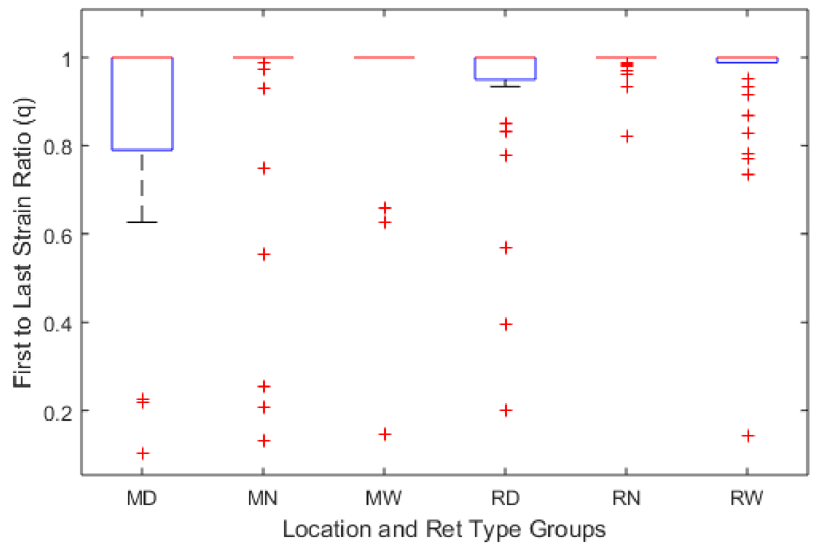

Figure 3.15: First to Last Strain Ratio (q) of Hemp by Location and Ret Type Groups

Table 3-2: Number of Outliers Removed from Flax Fibres

	Total samples	q Outliers removed	A_c Outliers removed
Minto Dew Retted (MD)	34	4	1
Minto Unretted (MN)	28	3	0
Minto Water Retted (MW)	40	3	0
Portage Dew Retted (RD)	35	6	0
Portage Unretted (RN)	42	10	0
Portage Water Retted (RW)	42	1	2

q = ratio of the apparent strain at first break to the apparent strain at final break
A_c = estimated cross-section area

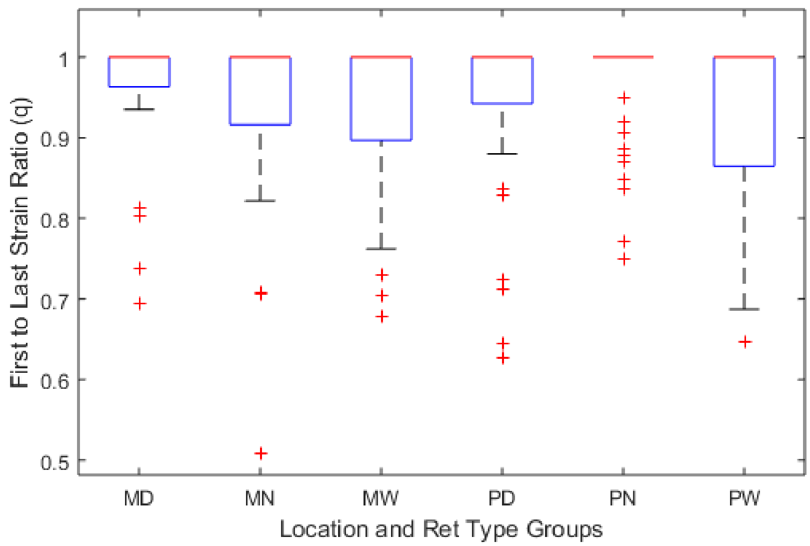

Figure 3.16: First to Last Strain Ratio (q) of Flax by Location and Ret Type Groups

3.8 Scanning Electron Microscopy (SEM) of Fibres

Scanning Electron Microscopy (SEM) images were taken longitudinally of non-macerated flax and hemp fibres for both growing locations and for unretted, dew retted and water retted samples using the FEI Quanta FEG 650 Scanning Electron Microscope at the Manitoba Institute for Materials (MIM) laboratory.

Figure 3.18 Scanning Electron Microscope used in this study (Photo taken by: Leif Norman, MIM)

Figure 3.17 Prepared hemp fibre sample stubs for SEM

Fibre samples were prepared identically to fibres prepared for tensile testing and then were mounted onto carbon tape and finally Gold-Palladium (Au-Pd) coated using the Denton Desk II Sputter Coater. Three fibres per sample type were mounted to each sample stub. When taking the images, the Everhart-Thornley Detector (ETD) was used at an Accelerating/High Voltage (HV) of 5kV. To help avoid bias, images were taken by arbitrarily selecting a horizontal location along the first fibre of a sample stub and systematically taking images at that same horizontal location on the second and third fibres. Images were taken at 100x, 500x and 1000x magnification.

4 RESULTS AND DISCUSSION

4.1 FIBRE YIELD

4.1.1 HEMP DATA ANALYSES

For hemp, the resulting experiment measured the effects of three factors: retting, maceration and location on three yield measurements: hurd yield, whole fibre yield and long fibre yield. While macerated was compared to non-macerated; three types of retting were examined: unretted, dew and water retted; and compared two growing locations: Roseisle and Minto Manitoba. All hemp samples were picked by hand, except the dew retted hemp from Minto that was baled and then provided by the CIC.

The first analysis uses samples from a single location (Roseisle) and assumes that each factor has a constant effect and two factor interactions were also assumed to be constant. This results in the following models for each yield type:

$$Y_{(H)jkl} = \bar{Y}_{(H)R} + R_{(H)j} + M_{(H)k} + RM_{(H)jk} + e_{(H)jkl}$$

$$Y_{(W)jkl} = \bar{Y}_{(W)R} + R_{(W)j} + M_{(W)k} + RM_{(W)jk} + e_{(W)jkl}$$

$$Y_{(L)jkl} = \bar{Y}_{(L)R} + R_{(L)j} + M_{(L)k} + RM_{(L)jk} + e_{(L)jkl}$$

Where $Y_{(H)jkl}$ is the post-crusher hurd yield for retting type j, maceration level k and sample l; $\bar{Y}_{(H)R}$ is the average post-crusher hurd yield from Roseisle, $R_{(H)j}$ is the retting effect of retting type j on post-crusher hurd yield, $M_{(H)k}$ is the effect of maceration level k on post-crusher hurd yield; $RM_{(H)jk}$ is the interaction effect between the retting type j and maceration level k on post-crusher hurd yield; and $e_{(H)jkl}$ is the random error for post-crusher hurd yield for the specific retting type, maceration level and sample. The remaining variables refer to (W) whole fibre yield or (L) long fibre yield. Six replicates of each of the six treatments creates three analyses with n=36. The SAS 9.4TS procedure General Linear Model (GLM) was used for the statistical analysis of these type III effects and their significance.

The second analysis used samples from both locations, for water and unretted material that was either macerated after harvest or remained non-macerated. As with the first analysis, it was assumed that each factor and two factor interactions have a constant effect. This results in the following models for each yield type:

$$Y_{(H)ijkl} = \bar{Y}_{(H)} + L_{(H)i} + R_{(H)j} + M_{(H)k} + LR_{(H)ij} + LM_{(H)ik} + RM_{(H)jk} + e_{(H)ijkl}$$

$$Y_{(W)ijkl} = \bar{Y}_{(W)} + L_{(W)i} + R_{(W)j} + M_{(W)k} + LR_{(W)ij} + LM_{(W)ik} + RM_{(W)jk} + e_{(W)ijkl}$$

$$Y_{(L)ijkl} = \bar{Y}_{(L)} + L_{(L)i} + R_{(L)i} + R_{(L)j} + M_{(L)k} + LR_{(L)ij} + LM_{(L)ik} + RM_{(L)jk} + e_{(L)ijkl}$$

Where $Y_{(H)ijkl}$ is the post-crusher hurd yield for location i, retting type j, maceration level k and sample l; $\bar{Y}_{(H)}$ is the average post-crusher hurd yield, $L_{(H)i}$ is the effect of location i on post-crusher hurd yield, $R_{(H)j}$ is the retting effect of retting type j on post-crusher hurd yield, $M_{(H)k}$ is the effect of maceration level k on post-crusher hurd yield; $LR_{(H)ij}$, $LM_{(H)ik}$ and $RM_{(H)jk}$ are the interaction effects between location, retting type and maceration level; and $e_{(H)ijkl}$ is the random error for post-crusher hurd yield for the specific retting type, maceration level and location on each sample. The remaining variables refer to (W) whole fibre yield or (L) long fibre yield. Six replicates of each of the eight treatments created three analyses with n=48. The SAS 9.4TS procedure GLM was used for the statistical analysis of these type III effects and determination of their significance. Details of this analysis can be found in Section 4.1.2.

4.1.2 HEMP RESULTS

The average and standard errors of hemp hurd, whole fibre and long fibre yields for the different growing locations, retting types, and maceration levels are presented in **Table 4-1**. The effect significance of retting type and maceration level and the GLM equation parameters for Roseisle hemp yields can be found in **Table 4-2** and **Table 4-3** respectively. The effects and significance of retting type (water retted versus unretted), maceration level and growing location and the GLM equation parameters for hemp yields can be found in **Table 4-4** and **Table 4-5** respectively.

From Minto, the whole fibre yield (see **Table 4-1**) for the dew retted straw for both non-macerated and macerated conditions were significantly higher than for unretted and water retted samples. These samples provided by the CIC were already partially processed as they had been baled and samples were pulled from the bale by hand yielding a material that had far less hurd content than all other samples tested. As these samples are not consistent with the other samples used in this experiment the results were not taken into consideration when assessing retting efficiency using fibre yield alone. However, these findings emphasize the importance of identical processing across samples in future work.

Table 4-1: Measured Fibre Yields of Hemp from Roseisle and Minto Locations

	Roseisle (n = 36)			Minto (n = 30)		
Parameters	**Long fibre yield, %**	**Whole fibre yield, %**	**Hurd yield, %**	**Long fibre yield, %**	**Whole fibre yield, %**	**Hurd yield, %**
Condition: Non-macerated						
Dew	6.29 ± 0.74^{d}	14.1 ± 1.72^{fg}	36.8 ± 3.23^{h}	10.6 ± 1.32*	*34.0 ± 1.13**	13.1 ± 1.60*
Unretted	8.93 ± 0.66^{c}	15.8 ± 0.76^{fg}	10.1 ± 0.66^{k}	9.25 ± 1.24^{m}	16.5 ± 1.61^{o}	10.3 ± 0.89^{p}
Water	14.1 ± 1.02^{b}	25.5 ± 0.91^{e}	9.52 ± 1.96^{k}	22.6 ± 0.50^{l}	31.6 ± 0.46^{n}	2.86 ± 0.68^{q}
Condition: Macerated						
Dew	7.76 ± 0.87^{cd}	16.6 ± 1.21^{f}	28.3 ± 2.57^{i}	14.2 ± 12.2*	*40.9 ± 2.22**	9.24 ± 0.49*
Unretted	8.37 ± 0.64^{cd}	12.7 ± 0.90^{g}	16.2 ± 1.29^{j}	11.1 ± 1.07^{m}	20.1 ± 1.53^{o}	8.92 ± 0.59^{p}
Water	17.1 ± 0.85^{a}	28.2 ± 1.43^{e}	5.32 ± 0.48^{k}	23.8 ± 0.55^{l}	33.0 ± 0.30^{n}	3.99 ± 0.39^{q}

*Minto dew retted was excluded from model calculations due to incomparability
Items with same letters are not significantly different (analyses were done in a column-wise basis)

From Roseisle, the hurd yield for the dew retted hemp for both non-macerated and macerated conditions were significantly higher than for unretted and water retted samples (see **Table 4-2** and **Table 4-3**). As the hurd yield represents the amount of hurd lost during the crushing step of the decortication process this implies that the fibres from the dew retted hemp from Roseisle were brittle. This may be an indication of over-retting as over-retted fibres have been shown to become brittle in past literature (G Henriksson et al. 1997; Liu et al. 2015, 2016; Martin et al. 2013). Furthermore, for Roseisle, the long fibre yield of dew-retted hemp straw is significantly lower than both water retted and unretted hemp straw, while the whole fibre yield of dew retted hemp straw is significantly higher than unretted hemp straw (see **Table 4-2** and **Table 4-3**). Since subtracting the long fibre yield from the whole fibre yield grants short fibre yield, this demonstrates that the short fibre yield of the dew retted hemp straw from Roseisle was significantly higher than unretted straw. Since the proportion of short fibre is higher, this also implies that the fibres were brittle, further strengthening the hypothesis that over-retting had occurred at the Roseisle growing location. Possible factors that may have caused over-retting are inexperience on the part of the farmers asked to perform the retting assessment, the difference in variety as well as location or the possible presence of undesirable microflora in the soil at that growing location (G Henriksson et al. 1997; Fila, Manici, and Caputo 2001; Di Candilo et al.

2010). However, we assume that since over-retting is possible then appropriate retting may be achieved in future works through more careful monitoring and soil microflora analyses. It is also worth noting that there was no established method to measure the ease of fibre extraction but it was easier to decorticate dew retted straw compared to unretted straw.

Despite the fact that the dew retted material from Roseisle may have been over-retted and the dew retted material from Minto was not processed identically to all other hemp treatments, a significant difference can still be observed in whole fibre yield as well as long fibre yield between raw and water retted hemp straw (see **Table 4-4** and **Table 4-5**), the latter being significantly higher. This confirms that fibre yield through this study's decortication process may be used as an indicator of hemp retting efficiency as fibre yield of water retted hemp straw is significantly higher than that of unretted hemp straw, which is in agreement with previous literature (Adamsen, Akin, and Rigsby 2002b; Danny E. Akin et al. 2007; D E Akin 2013).

Table 4-2: Effect Significance of Retting Type and Maceration Level on Roseisle Hemp Yields

Source	DF	Type III SS	Pr > F
$R_{(H)j}$	2	4160*	<.0001
$M_{(H)k}$	1	43.9	0.1790
$RM_{(H)jk}$	2	340*	0.0025
$R_{(W)j}$	2	1180*	<.0001
$M_{(W)k}$	1	4.27	0.4877
$RM_{(W)jk}$	2	64.0*	0.0371
$R_{(L)j}$	2	503*	<.0001
$M_{(L)k}$	1	15.3	0.0568
$RM_{(L)jk}$	2	19.3	0.1011

*Indicates type III effect is significant to p<0.05

Table 4-3: Parameters for Retting Type and Maceration Level on Roseisle Hemp Yields

Parameter	Hurd (H)	Whole Fibre (W)	Long Fibre (L)
$\bar{Y}$	9.52 ± 1.96%*	25.5 ± 1.20%*	14.1 ± 0.80%*
R_{Dew}	27.3 ± 2.78%*	-11.4 ± 1.70%*	-7.85 ± 1.14%*
$R_{Unretted}$	0.526 ± 2.78%	-9.77 ± 1.70%*	-5.21 ± 1.14%*
$M_{Macerated}$	-4.20 ± 2.78%	2.67 ± 1.70%	3.01 ± 1.14%*
$RM_{Dew\ Macerated}$	-4.35 ± 3.93%	-0.188 ± 2.40%	-1.54 ± 1.61%
$RM_{Unretted\ Macerated}$	10.3 ± 3.93%*	-5.74 ± 2.40%*	-3.57 ± 1.61%*

*Indicates type III effect is significant to p<0.05
N.B. All unlisted parameters are 0.00% by definition.

Furthermore, when observing the effect of maceration on fibre yields from the Roseisle growing location there was no significant effect on hemp fibre yield (see **Table 4-2** and **Table 4-3**). Likewise, when observing the effect of water retting versus unretted hemp straw there was once again no significant effect of maceration on fibre yield (see **Table 4-4** and **Table 4-5**). This is in agreement with work done by Foulk et al. (2001) which showed no significant effect on retting efficiency of enzyme retted flax straw using flat rollers when compared to non-macerated flax straw. However, their study did show a significant increase in retting efficiency when macerating flax straw with fluted rollers. They surmised that using fluted rollers increases retting efficiency because of the crimping effect caused by fluted rollers versus the flattening/shearing effect caused by flat rollers. This crimping effect broke the outer cuticle layer of the flax straw granting the pectin degrading enzymes easier access to the straw interior. In future works, maceration using fluted rollers should be investigated and therefore using local agricultural maceration equipment may not be possible.

Table 4-4: Significance of Location, Ret Type (Unretted/Water) & Maceration Level on Hemp Yields

Source	DF	Type III SS	Pr>F	Source	DF	Type III SS	Pr>F	Source	DF	Type III SS	Pr > F
$L_{(H)j}$	1	169*	<.0001	$L_{(W)j}$	1	268*	<.0001	$L_{(L)j}$	1	247*	<.0001
$R_{(H)k}$	1	421*	<.0001	$R_{(W)k}$	1	2120*	<.0001	$R_{(L)k}$	1	1210*	<.0001
$M_{(H)k}$	1	2.20	0.6200	$M_{(W)k}$	1	15.4	0.1762	$M_{(L)k}$	1	22.6*	0.0320
$LR_{(H)k}$	1	46.0*	0.0275	$LR_{(W)k}$	1	8.70	0.3063	$LR_{(L)k}$	1	6.90	0.2281
$LM_{(H)k}$	1	0.700	0.7806	$LM_{(W)k}$	1	5.60	0.4130	$LM_{(L)k}$	1	111*	<.0001
$RM_{(H)jk}$	1	3.40	0.5376	$RM_{(W)jk}$	1	21.5	0.1119	$RM_{(L)jk}$	1	0.300	0.8053

*Indicates type III effect is significant to p<0.05

Table 4-5: Effects for Location, Ret Type (Unretted vs Water) and Maceration Level on Hemp Yields

Parameter	Hurd (H)	Whole Fibre (W)	Long Fibre (L)
$\overline{Y}$	7.92 ± 1.13%*	26.5 ± 1.09%*	14.7 ± 0.818%*
L_{Minto}	-3.46 ± 1.48%*	4.07 ± 1.43%*	7.42 ± 1.07%*
$R_{Unretted}$	3.72 ± 1.48%*	-11.8 ± 1.43%*	-6.24 ± 1.07%*
$M_{Macerated}$	-0.998 ± 1.48%	0.648 ± 1.43%	1.98 ± 1.07%
$LR_{Minto\ Unretted}$	0.480 ± 1.71%	-1.36 ± 1.65%	-6.07 ± 1.24%*
$LM_{Minto\ Macerated}$	-1.06 ± 1.71%	2.67 ± 1.65%	0.307 ± 1.24%
$RM_{Unretted\ Macerated}$	3.92 ± 1.71%*	-1.71 ± 1.65%	-1.51 ± 1.24%

*Indicates type III effect is significant to p<0.05
N.B. All unlisted parameters are 0.00% by definition.

4.1.3 FLAX DATA ANALYSES

In the case of flax, the resulting experiment once again measured the effects of three factors: retting, maceration and location on three yield measurements: shive yield, whole fibre yield and long fibre yield. While macerated was compared to non-macerated; three types of retting were examined: unretted, dew and water retted; and compared two growing locations: Portage and Minto Manitoba. Water retting was not applied to any macerated flax as the macerated material from both locations was heavily damaged by the maceration process and therefore was already partially decorticated. It was assumed that water retting partially decorticated material would result in tangled and possibly over-retted fibres, which would be difficult to process through this project's established decortication method. The dew retted flax from both Portage and Minto were baled and provided by the CIC, all other samples were picked by hand.

The first analysis uses non-macerated samples from both locations, for water and unretted material, and assumed that each factor and two factor interactions have a constant effect. This results in the following models for each yield type:

$$Y_{(S)jkl} = \bar{Y}_{(S)NM} + R_{(S)j} + L_{(S)k} + RL_{(S)jk} + e_{(S)jkl}$$

$$Y_{(W)jkl} = \bar{Y}_{(W)NM} + R_{(W)j} + L_{(W)k} + RL_{(W)jk} + e_{(W)jkl}$$

$$Y_{(L)jkl} = \bar{Y}_{(L)NM} + R_{(L)j} + L_{(L)k} + RL_{(L)jk} + e_{(L)jkl}$$

Where $Y_{(S)jkl}$ is the post-crusher shive yield for retting type j, location k and sample l; $\bar{Y}_{(S)R}$ is the average post-crusher shive yield for non-macerated flax, $R_{(S)j}$ is the retting effect of retting type j on post-crusher shive yield, $L_{(S)k}$ is the effect of location k on post-crusher shive yield; $RL_{(S)jk}$ is the interaction effect between the retting type j and location k on post-crusher shive yield; and $e_{(S)jkl}$ is the random error for post-crusher shive yield for the specific retting type, location and sample. The remaining variables refer to (W) whole fibre yield or (L) long fibre yield. Six replicates of each of the four treatments creates three analyses with n=24. The SAS 9.4TS procedure GLM was used for the statistical analysis of these type III effects and their significance.

The second analysis for flax uses unretted samples from both locations, for macerated and non-macerated material, and assumed that each factor has a constant effect and two factor interactions were computed. This results in the following models for each yield type:

$$Y_{(S)jkl} = \bar{Y}_{(S)U} + L_{(S)j} + M_{(S)k} + LM_{(S)jk} + e_{(S)jkl}$$

$$Y_{(W)jkl} = \bar{Y}_{(W)U} + L_{(W)j} + M_{(W)k} + LM_{(W)jk} + e_{(W)jkl}$$

$$Y_{(L)jkl} = \bar{Y}_{(L)U} + L_{(L)j} + M_{(L)k} + LM_{(L)jk} + e_{(L)jkl}$$

Where $Y_{(S)jkl}$ is the post-crusher shive yield for location j, maceration level k and sample l; $\bar{Y}_{(S)R}$ is the average post-crusher shive yield for unretted flax, $L_{(S)j}$ is the location effect of location j on post-crusher shive yield, $M_{(S)k}$ is the effect of maceration level k on post-crusher shive yield; $LM_{(S)jk}$ is the interaction effect between location j and maceration level k on post-crusher shive yield; and $e_{(S)jkl}$ is the random error for post-crusher shive yield for the specific location, maceration level and sample. The remaining variables refer to (W) whole fibre yield or (L) long fibre yield. Six replicates of each of the four treatments creates three analyses with n=24. The SAS 9.4TS procedure GLM was used for the statistical analysis of these type III effects and their significance.

The third analysis for flax uses the baled dew retted samples for both locations, for macerated and non-macerated material, and assumed that each factor has a constant effect and two factor interactions were computed. This results in the following models for each yield type:

$$Y_{(S)jkl} = \bar{Y}_{(S)D} + L_{(S)j} + M_{(S)k} + LM_{(S)jk} + e_{(S)jkl}$$

$$Y_{(W)jkl} = \bar{Y}_{(W)D} + L_{(W)j} + M_{(W)k} + LM_{(W)jk} + e_{(W)jkl}$$

$$Y_{(L)jkl} = \bar{Y}_{(L)D} + L_{(L)j} + M_{(L)k} + LM_{(L)jk} + e_{(L)jkl}$$

Where $Y_{(S)jkl}$ is the post-crusher shive yield for location j, maceration level k and sample l; $\bar{Y}_{(S)R}$ is the average post-crusher shive yield for dew retted flax, $L_{(S)j}$ is the location effect of location j on post-crusher shive yield, $M_{(S)k}$ is the effect of maceration level k on post-crusher shive yield; $LM_{(S)jk}$ is the interaction effect between location j and maceration level k on post-crusher shive yield; and $e_{(S)jkl}$ is the random error for post-crusher shive yield for the specific location, maceration level and sample. The remaining variables refer to (W) whole fibre yield or (L) long fibre yield. Six replicates of each of the two treatments from Portage and five replicates of each of the two treatments from Minto due to lack of material creates three analyses with n=22. The SAS 9.4TS procedure GLM was used for the statistical analysis of these type III effects and their significance. Details of this analysis can be found in Section 4.1.4.

4.1.4 FLAX RESULTS

The average and standard errors of flax shive, whole fibre and long fibre yields for the different growing locations, retting types, and maceration levels are presented in Error! Reference source not found.. The effect significance of retting type (water retted versus unretted) and growing location and the GLM equation parameters for non-macerated flax yields can be found in **Table 4-7** and **Table 4-8**, respectively. The effect significance of maceration level and growing location on the GLM equation parameters for unretted flax yields can be found in **Table 4-9** and **Table 4-10** respectively. Finally, the effect significance of maceration level and growing location on the GLM equation parameters for dew retted flax can be found in **Table 4-11** and **Table 4-12** respectively.

Table 4-6: Measure Fibre Yields of Flax from Portage and Minto Locations

	Portage (n = 30)			Minto (n = 28)		
Parameters	**Long fibre yield, %**	**Whole fibre yield, %**	**Shive yield, %**	**Long fibre yield, %**	**Whole fibre yield, %**	**Shive yield, %**
Condition: Non-macerated						
Dew	3.42 ± 0.39^{b}	7.98 ± 0.29^{e}	14.2 ± 1.23^{h}	3.76 ± 0.96^{j}	10.7 ± 2.48^{l}	16.2 ± 1.14^{p}
Unretted	3.33 ± 0.38^{c}	6.21 ± 0.51^{e}	32.9 ± 0.81^{f}	1.91 ± 0.13^{k}	4.05 ± 0.33^{m}	34.4 ± 2.60^{n}
Water	7.74 ± 0.67^{a}	20.3 ± 0.49^{d}	3.21 ± 0.46^{i}	5.28 ± 0.36^{j}	11.5 ± 0.14^{l}	4.75 ± 0.55^{q}
Condition: Macerated						
Dew	2.68 ± 0.18^{b}	7.58 ± 0.60^{e}	21.8 ± 3.27^{g}	5.58 ± 0.76^{j}	13.4 ± 1.93^{l}	14.6 ± 2.34^{p}
Unretted	2.98 ± 0.10^{c}	6.61 ± 0.44^{e}	20.7 ± 1.35^{g}	2.36 ± 0.37^{k}	4.71 ± 0.35^{m}	17.9 ± 1.23^{o}

Items with same letters are not significantly different (analyses were done in a column-wise basis)

As mentioned in section 4.1.3, macerated flax straw was not water retted as the material from both growing locations was heavily damaged by the maceration process and was therefore already partially decorticated. Also, mentioned in section 4.1.3, the dew retted material from both locations was baled, and the yield results from the dew retted straw from each growing location could only be compared to each other and not to the unretted or water retted flax material.

When observing the effect of water retting on non-macerated flax yields from both growing locations there is a significant increase in whole and long fibre yields (see **Table 4-7** and **Table 4-8**) in water retted flax straw. This supports the hypothesis that water retting increased the fibre yield and shows that this project's decortication method can be used to assess retting quality of flax as well as hemp based on results of this study found with hemp (see Section 4.1.2) , which is in agreement with previous literature (Adamsen, Akin, and Rigsby 2002a; Danny E. Akin et al. 2007; D E Akin 2013). Also noteworthy, is that location has a significant effect with Portage having significantly higher whole and long fibre yields than Minto. The idea that location can have a significant effect on fibre yield is not surprising as a variety of factors affected by location (i.e. growing condition) are known to affect fibre properties (Duval et al. 2011). However, the fact that unretted and water retted flax straw from

Portage had significantly higher yield than those from Minto will be noteworthy when observing the effect of location on the dew retted flax straw further in this analysis.

Table 4-7: *Effect Significance of Retting Type (Unretted vs Water) and Location on Non-Macerated Flax Yields*

Source	DF	Type III SS	Pr > F
$R_{(S)j}$	1	5280.0*	<.0001
$L_{(S)k}$	1	13.2	0.3049
$RL_{(S)jk}$	1	0.0164	0.9707
$R_{(W)j}$	1	695.0*	<.0001
$L_{(W)k}$	1	182.0*	<.0001
$RL_{(W)jk}$	1	67.3*	<.0001
$R_{(L)j}$	1	90.9*	<.0001
$L_{(L)k}$	1	22.5*	0.0002
$RL_{(L)jk}$	1	1.63	0.2404

*Indicates type III effect is significant to p<0.05

Table 4-8: Parameters for Retting Type (Unretted vs Water) and Location on Non-Macerated Flax Yields

Parameter	Shive (S)	Whole Fibre (W)	Long Fibre (L)
$\bar{Y}$	3.21 ± 1.41%*	20.3 ± 0.396%*	7.74 ± 0.430%*
L_{Minto}	1.53 ± 1.99%	-8.86 ± 0.560%*	-2.46 ± 0.609%*
$R_{Unretted}$	29.7 ± 1.99%*	-14.1 ± 0.560%*	-4.41 ± 0.609%*
$LR_{Minto\ Unretted}$	-0.105 ± 2.81%	6.70 ± 0.792%*	1.04 ± 0.861%

*Indicates type III effect is significant to p<0.05
N.B. All unlisted parameters are 0.00% by definition.

When observing the effect of maceration on unretted flax straw from both growing locations there is no significant effect on whole and long fibre yields (see **Table 4-9** and **Table 4-10).** This is once again in agreement with this project's results with hemp and with previous literature (Foulk, Akin, and Dodd 2001) where maceration using flat rollers had no significant effect on enzyme retted flax. In addition, location once again had a significant effect on whole and long fibre yields with flax yields from Portage being significantly higher than those from Minto.

Table 4-9: Effect Significance of Maceration Level and Location on Unretted Flax Yields

Source	DF	Type III SS	Pr > F
$L_{(S)j}$	1	2.78	0.6834
$M_{(S)k}$	1	1230.0*	<.0001
$LM_{(S)jk}$	1	26.7	0.2144
$L_{(W)j}$	1	24.7*	<.0001
$M_{(W)k}$	1	1.68	0.2120
$LM_{(W)jk}$	1	0.100	0.7562
$L_{(L)j}$	1	6.24*	0.0015
$M_{(L)k}$	1	0.0144	0.8623
$LM_{(L)jk}$	1	0.940	0.1706

*Indicates type III effect is significant to p<0.05

Table 4-10: Parameters for Maceration Level and Location on Unretted Flax Yields

Parameter	Shive (S)	Whole Fibre (W)	Long Fibre (L)
$\bar{Y}$	32.9 ± 1.65%*	6.21 ± 0.411%*	3.33 ± 0.278%*
L_{Minto}	1.43 ± 2.33%	-2.16 ± 0.581%*	-1.42 ± 0.394%*
$M_{Macerated}$	-12.2 ± 2.33%*	0.400 ± 0.581%	-0.347 ± 0.394%
$LM_{Minto\ Macerated}$	-4.22 ± 3.29%	0.259 ± 0.822%	0.792 ± 0.557%

*Indicates type III effect is significant to p<0.05
N.B. All unlisted parameters are 0.00% by definition.

When determining the effect of maceration on the baled dew retted flax straw from both growing locations there was no significant effect on whole and long fibre yields (see **Table 4-11** and **Table 4-12**) further “confirming” our hypothesis that maceration using the Macerator 6620 (Pronovost Inc.) has no significant effect. In addition, location once again had a significant effect on whole and long fibre yields with flax yields from Minto being significantly higher than

Portage in this case. As previously mentioned, the unretted and water retted flax from Portage had significantly higher whole and long fibre yields than Minto. This implies that the dew retting in Minto was more efficient than in Portage and this can be seen in the colour change of the dew retted fibres (see Figure 4.1 and Figure 4.2). The dew retted fibres from Portage are more brown in colour whereas the fibres from Minto appear to be more gray, which is a sign of greater retting (D. E. Akin, Epps, et al. 2000).

Table 4-11: Effect Significance of Maceration Level and Location on Dew Retted Flax Yields

Source	DF	Type III SS	Pr > F
$L_{(S)j}$	1	37.5	0.2610
$M_{(S)k}$	1	49.6	0.1991
$LM_{(S)jk}$	1	115.0	0.0570
$L_{(W)j}$	1	98.0*	0.0097
$M_{(W)k}$	1	7.07	0.4470
$LM_{(W)jk}$	1	12.8	0.3087
$L_{(L)j}$	1	14.3*	0.0149
$M_{(L)k}$	1	1.60	0.3807
$LM_{(L)jk}$	1	9.00*	0.0470

*Indicates type III effect is significant to $p<0.05$

Table 4-12: Parameters for Maceration Level and Location on Dew Retted Flax Yields

Parameter	Shive (S)	Whole Fibre (W)	Long Fibre (L)
$\bar{Y}$	14.2 ± 2.16%*	7.98 ± 1.40%*	3.42 ± 0.574%*
L_{Minto}	1.97 ± 3.20%	2.70 ± 2.07%	0.337 ± 0.852%
$M_{Macerated}$	7.61 ± 3.05%*	-0.360 ± 1.97%	-0.743 ± 0.812%
$LM_{Minto\ Macerated}$	-9.19 ± 4.52%	3.07 ± 2.93%	2.60 ± 1.20%*

*Indicates type III effect is significant to p<0.05
N.B. All unlisted parameters are 0.00% by definition.

Figure 4.1: Portage Dew Retted Flax

Figure 4.2: Minto Dew Retted Flax

4.2 Scanning Electron Microscopy (SEM) of Fibres

4.2.1 Hemp

Figure 4.3 to Figure 4.8 show scanning electron micrographs of unretted, dew retted and water retted hemp fibres for both Minto and Roseisle growing locations. The longitudinal sections of hemp fibres have definite signs of fibre separation and pectinaceous matrix degradation when comparing the unretted hemp fibres to both the dew retted and water retted hemp fibres. Unretted fibres show no signs of fibre separation and the pectinaceous matrix can be easily seen covering the entire fibre sample (see Figure 4.3 and Figure 4.4). Water retted fibres appear to be more separated than dew retted fibres when comparing Figure 4.5 and Figure 4.6 with Figure 4.7 and Figure 4.8. The dew retted fibres are not as well separated as water retted fibres but still show signs of degradation and separation. These observations concur with the results found in the yield study (see section 4.1.2) where water retting had the highest whole fibre yield followed by dew retting. Additionally, the figures show that these fibre samples are technical fibres consisting of many elementary fibres. As these samples were prepared identically to the samples used in the single fibre tensile tests of this project, the samples used in tensile tests are technical fibres composed of many elementary fibres.

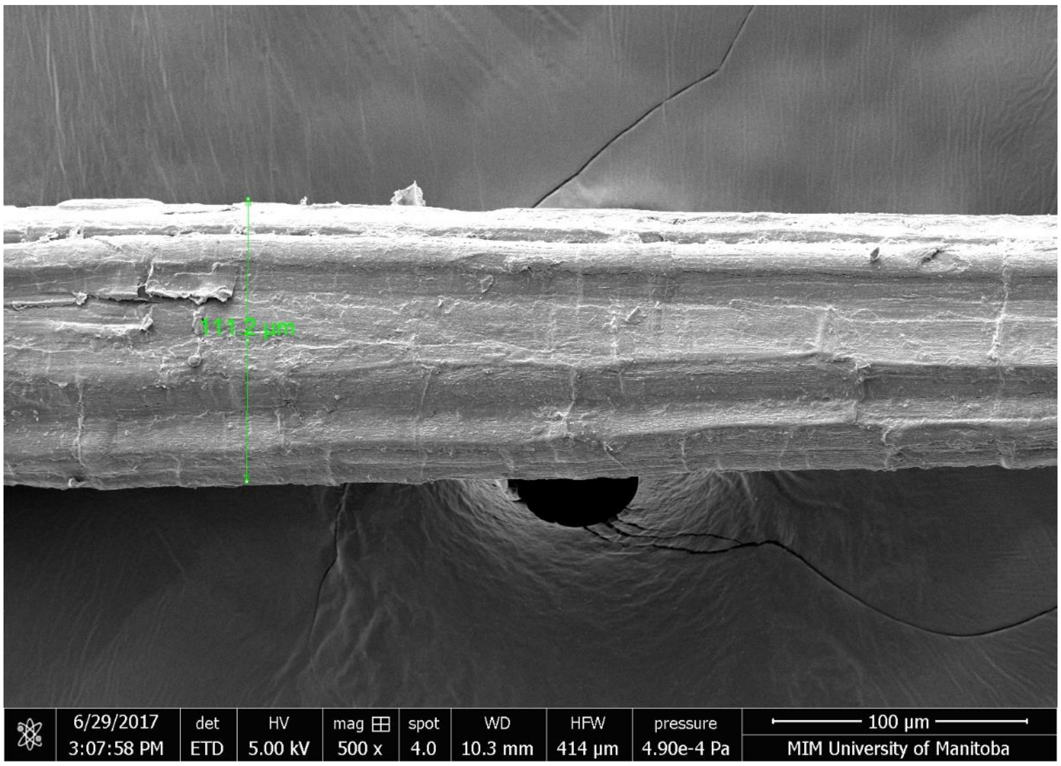

Figure 4.3: Minto Unretted Hemp Fibre (500x mag)

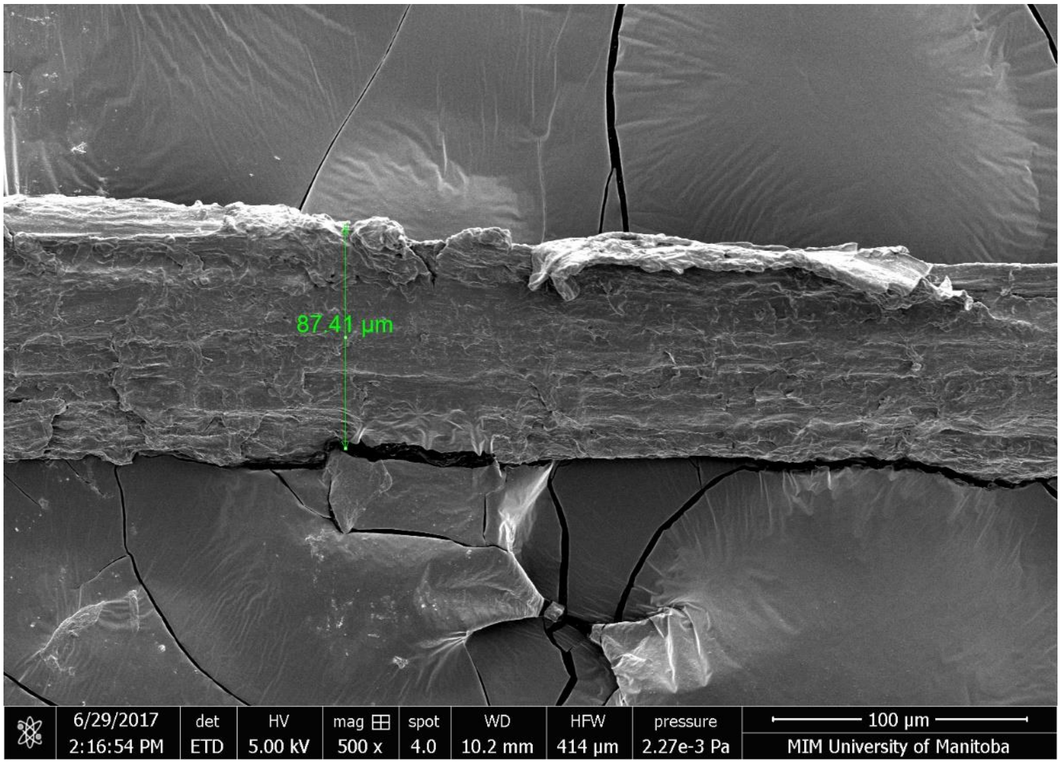

Figure 4.4: Roseisle Unretted Hemp Fibre (500x mag)

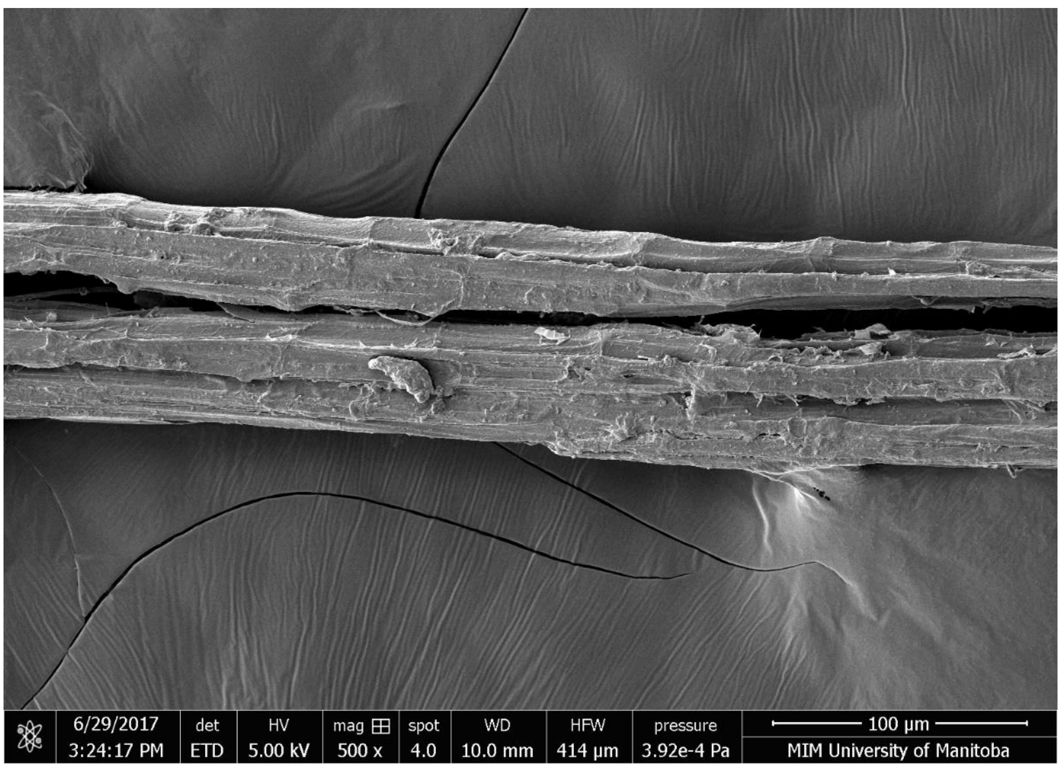

Figure 4.5: Minto Water Retted Hemp Fibre (500x mag)

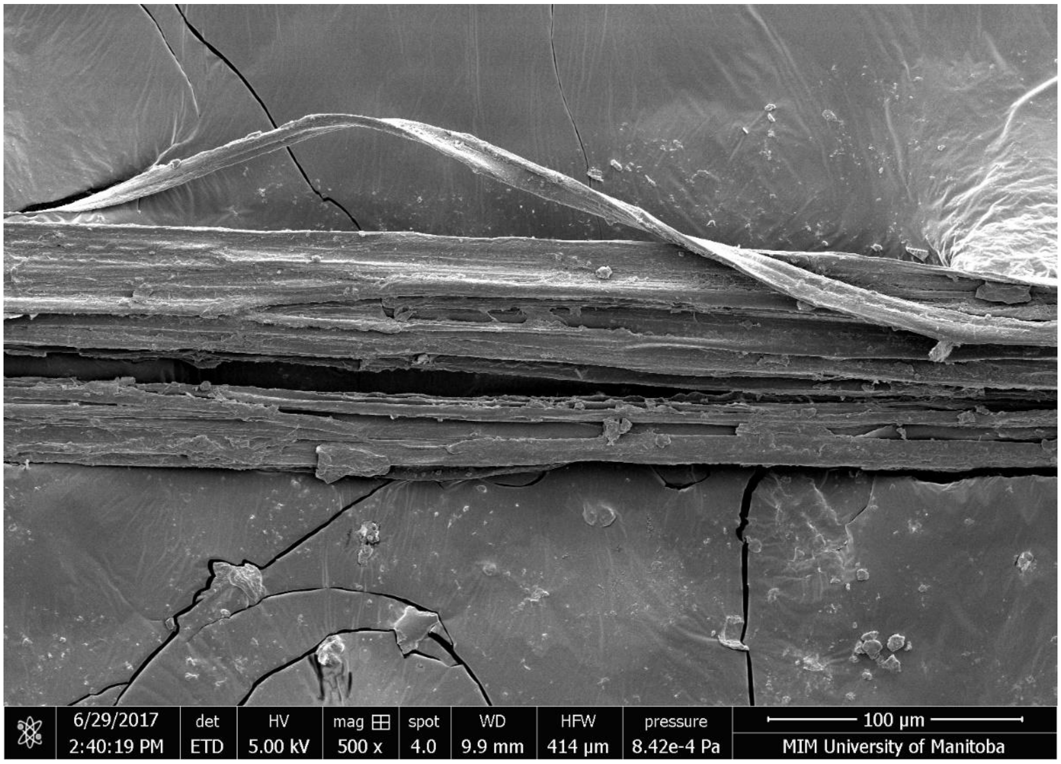

Figure 4.6: Roseisle Water Retted Hemp Fibre (500x mag)

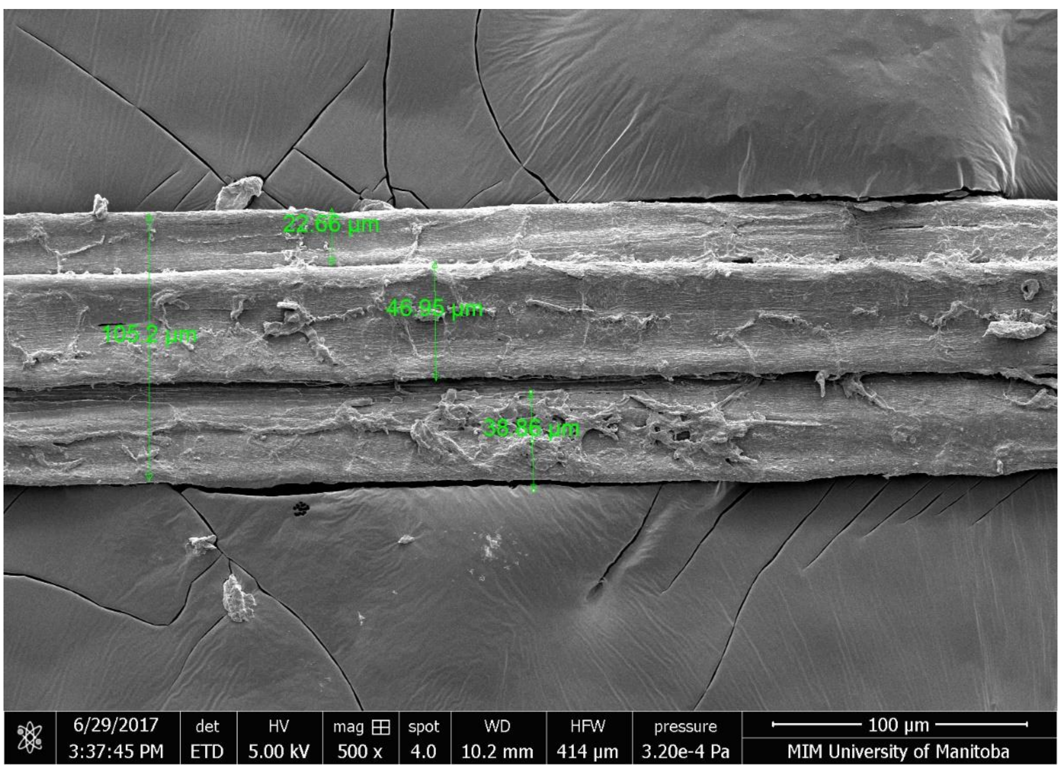

Figure 4.7: Minto Dew Retted Hemp Fibre (500x mag)

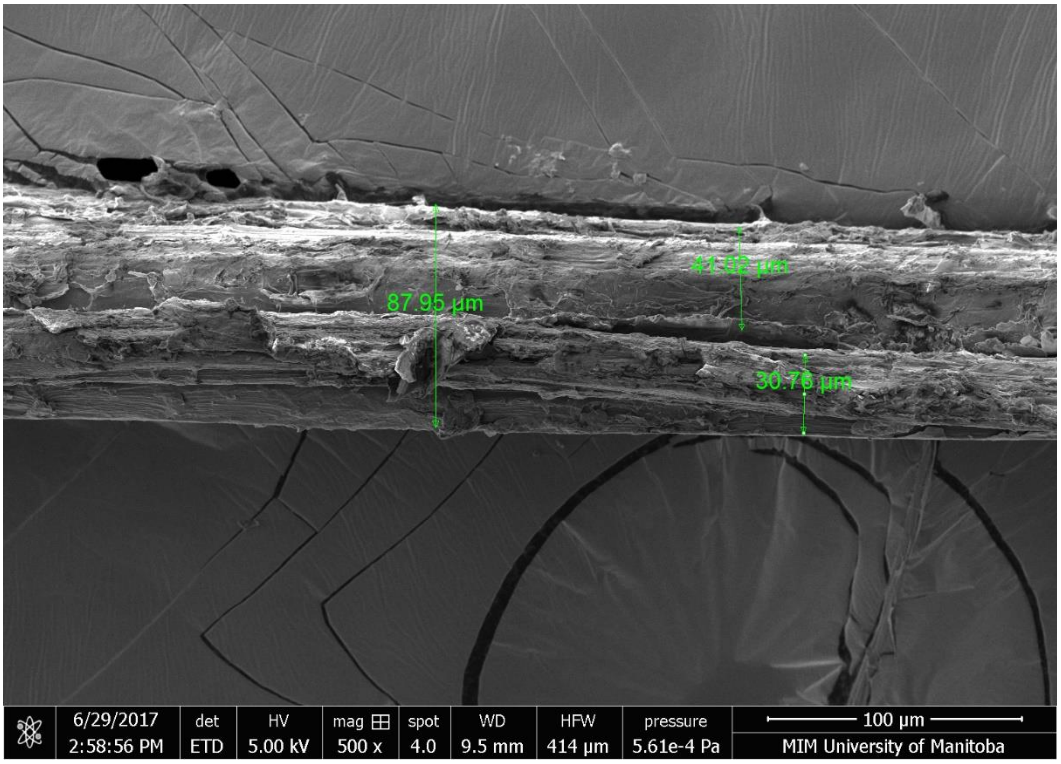

Figure 4.8: Roseisle Dew Retted Hemp Fibre (500x mag)

4.2.2 FLAX

Figure 4.9 to Figure 4.14 show scanning electron micrographs of unretted, dew retted and water retted flax fibres for both Minto and Portage growing locations. Similar to the SEM results from Hemp, the longitudinal sections of flax fibres show definite sign of fibre separation and pectinaceous matrix degradation when comparing the unretted flax fibres to both the dew retted and water retted flax fibres. Once again, water retted fibres appear to be the most degraded followed by dew retted fibres which supports results found in the fibre yield study (see section 4.1.4). Finally, SEM scans show that flax fibres prepared for SEM scans and single fibre tensile tests are technical fibres composed of many elementary fibres.

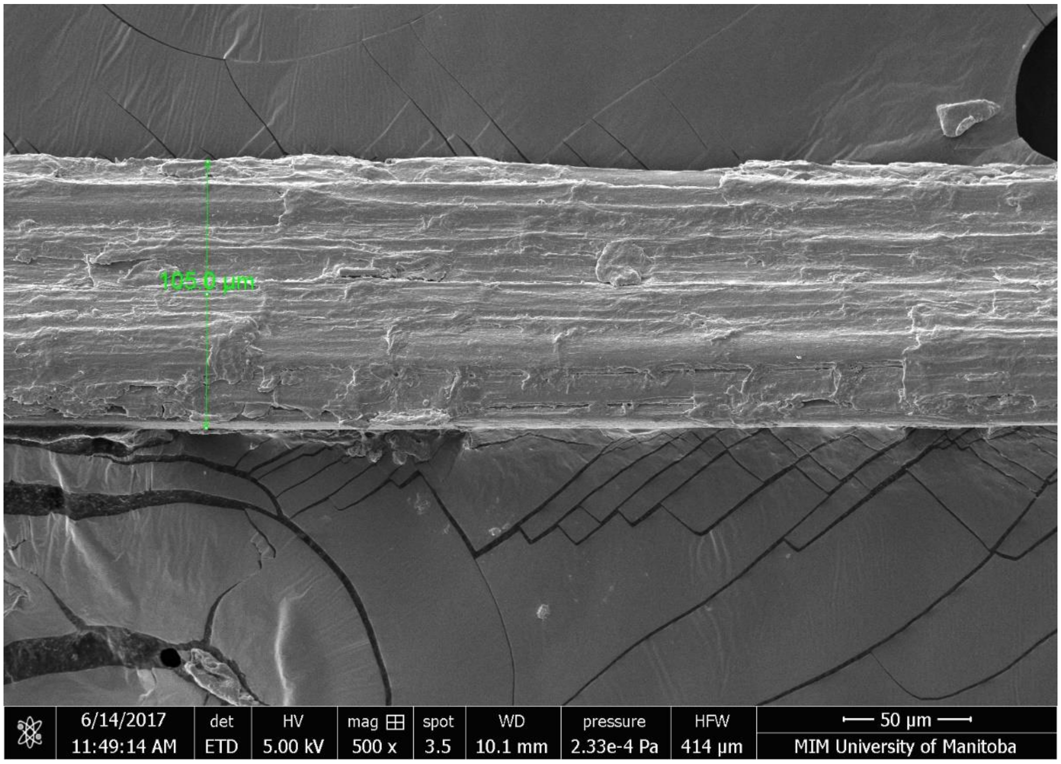

Figure 4.9: Minto Unretted Flax Fibre (500x mag)

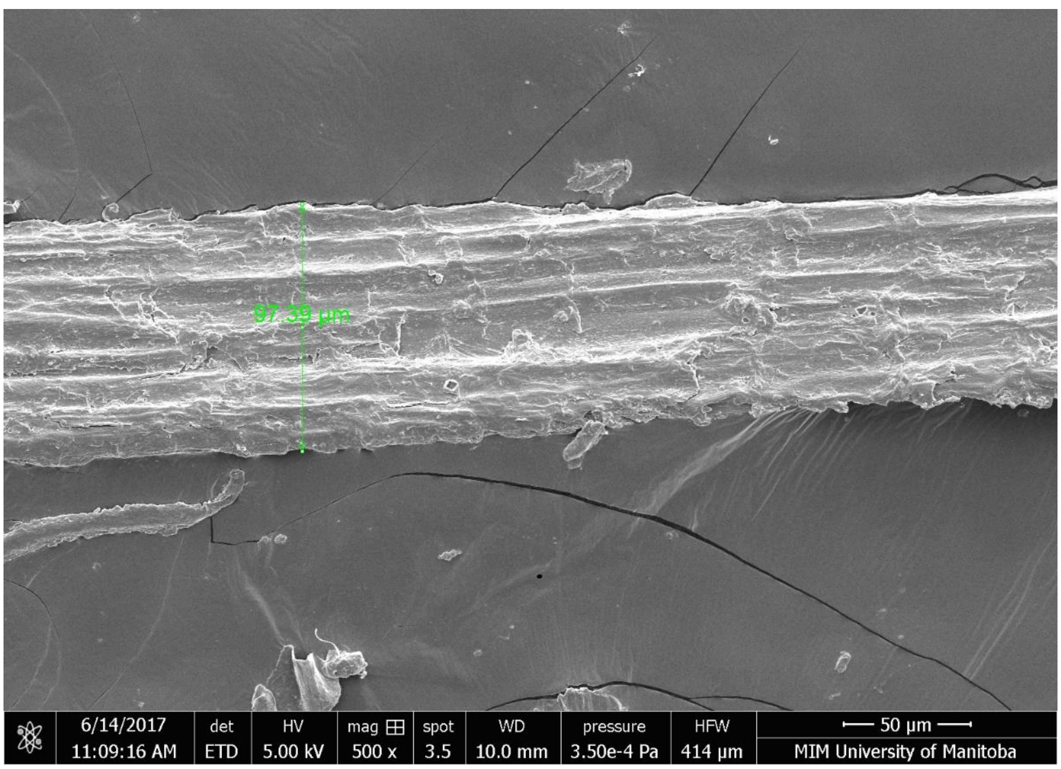

Figure 4.10: Portage Unretted Flax Fibre (500x mag)

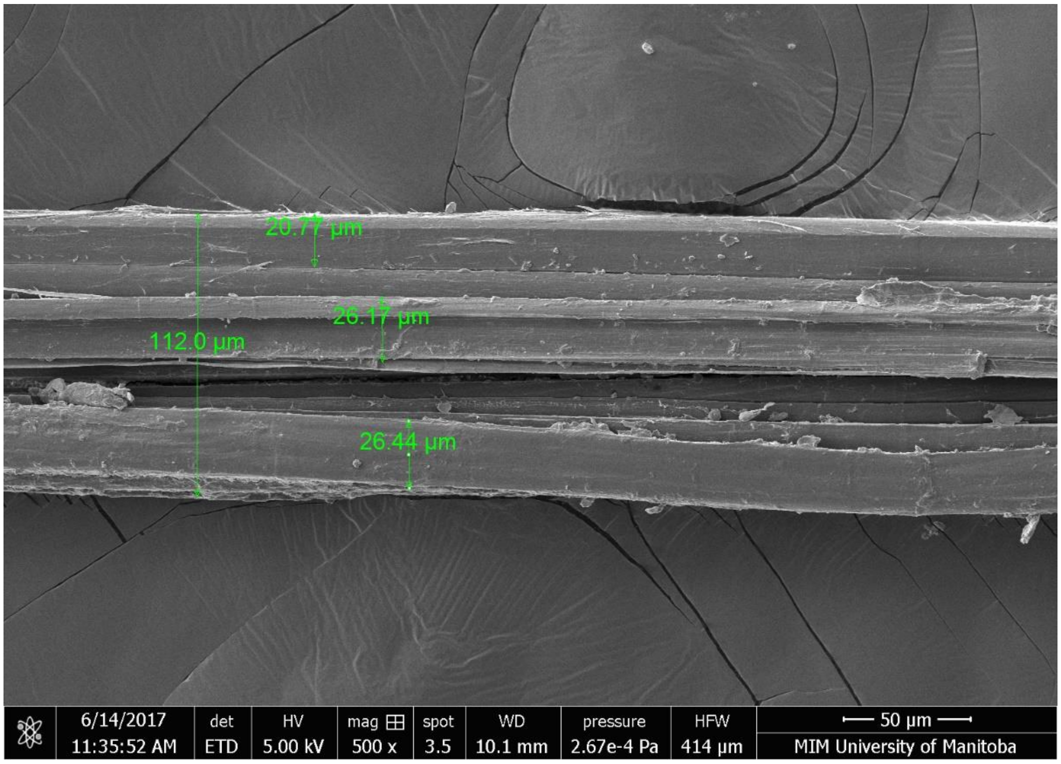

Figure 4.11: Minto Water Retted Flax Fibre (500x mag)

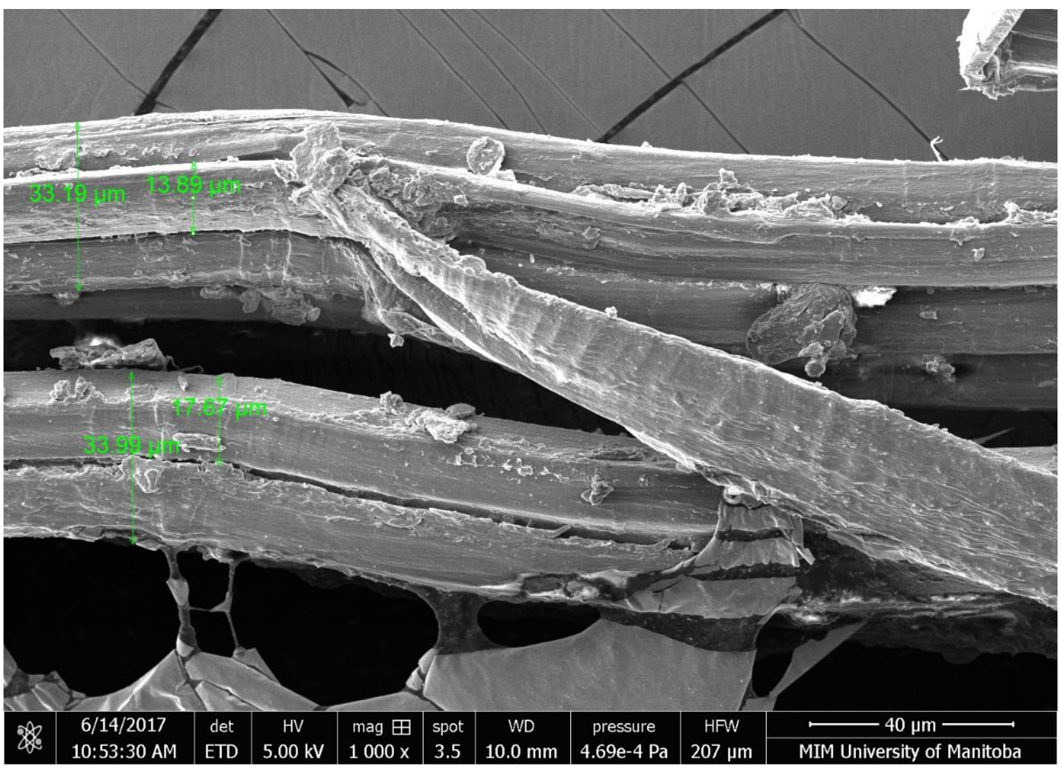

Figure 4.12: Portage Water Retted Flax Fibre (1000x mag)

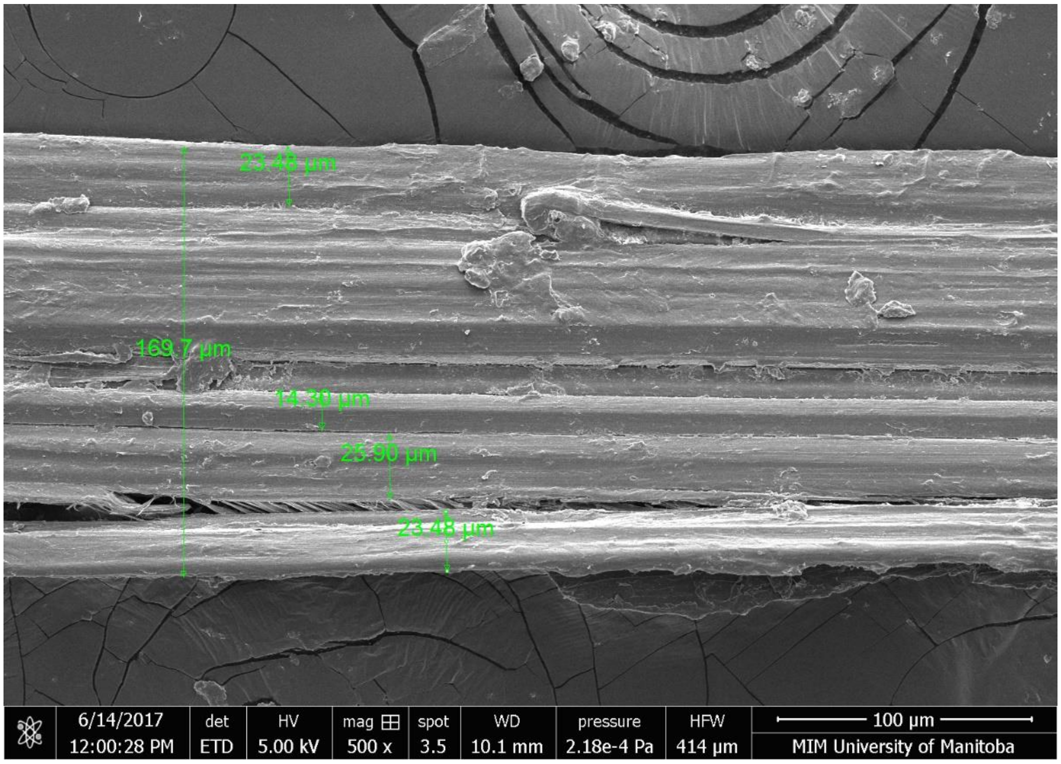

Figure 4.13: Minto Dew Retted Flax Fibre (500x mag)

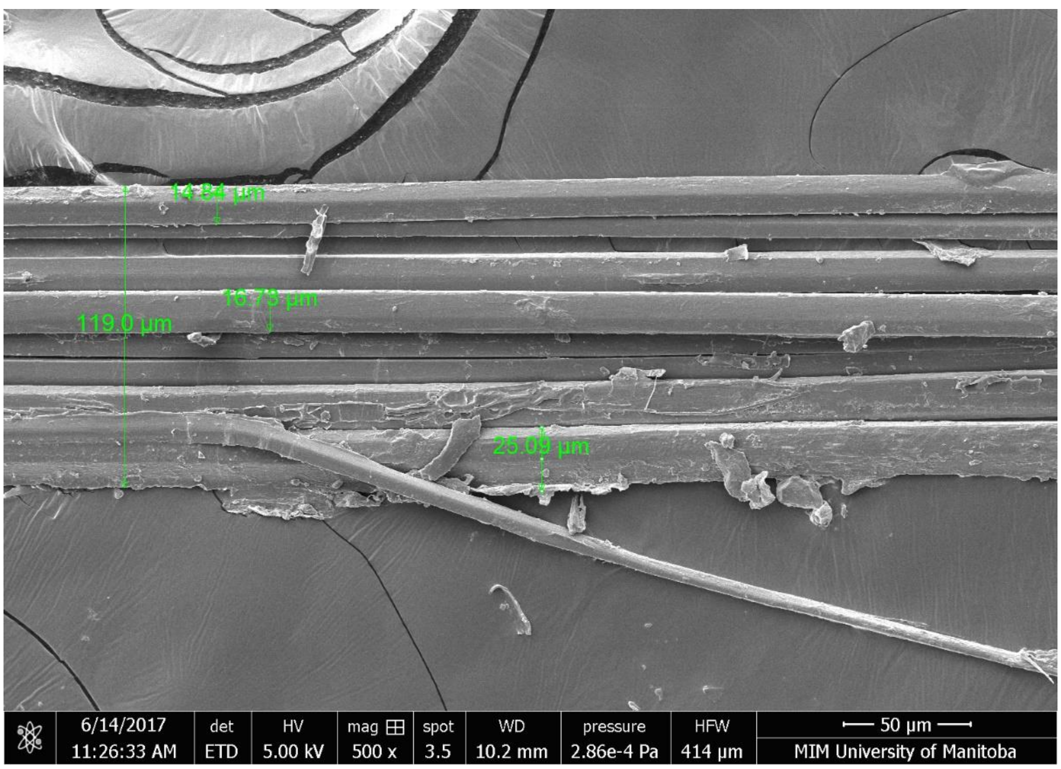

Figure 4.14: Portage Dew Retted Flax Fibre (500x mag)

4.3 FIBRE TENSILE STRENGTH

4.3.1 DATA ANALYSES

For both hemp and flax, the resulting experiment measured the effects of three factors: retting, location and cross-sectional area on the first break tensile stress of fibre samples. Three types of retting were examined: unretted, dew retted and water retted; and two growing locations were compared: Roseisle and Minto Manitoba for hemp and Portage and Minto Manitoba for flax. This results in the following models for hemp and flax:

$$S_{(H)jkl} = \beta_{0(H)} + R_{(H)j} + L_{(H)k} + \beta_1 A_{(H)jkl} + RL_{(H)jk} + e_{(H)jkl}$$

$$S_{(F)jkl} = \beta_{0(F)} + R_{(F)j} + L_{(F)k} + \beta_1 A_{(F)jkl} + RL_{(F)jk} + e_{(F)jkl}$$

Where $S_{(H)jkl}$ is the first break tensile stress of hemp for the l^{th} sample of retting type j and location k; $\beta_{0(H)}$ is the computed intercept of the model of first break tensile stress of hemp, $R_{(H)j}$ is the retting effect of retting type j on the first break tensile stress of hemp, $L_{(H)k}$ is the effect of location k on the first break tensile stress of hemp; β_1 is the amount of change in the first break stress per unit cross-sectional area; $A_{(H)jkl}$ is the measured cross-sectional area of the l^{th} sample of retting type j and location k of hemp; $RL_{(H)jk}$ is the interaction effect between the retting type j and location k on the first break tensile stress of hemp; and $e_{(H)jkl}$ is the random error for the first break tensile stress of hemp for the specific retting type, location, cross-sectional area and sample. The remaining variables refer to (F) flax. The SAS 9.4TS procedure General Linear Model (GLM) with the Tukey-Kramer adjustment for multiple comparisons was used for the statistical analysis of these type III effects and their significance.

4.3.2 HEMP

The Tukey-adjusted least-square means estimate of first break stress of non-macerated hemp fibres for the different growing locations and retting types are presented in **Table 4-13**. The effect significance of retting type, location and cross-sectional area and the GLM equation parameters for first break tensile stress of non-macerated hemp fibres can be found in **Table 4-14** and **Table 4-15** respectively.

Table 4-13: *Least-Square Means Estimate of σ_i of Hemp Fibres*

Group*		Stress LSMEAN	Location	Ret Type
	A	1.11E+09	Minto	Water
	A	1.00E+09	Roseisle	Unretted
B	A	8.99E+08	Minto	Unretted
B	C	7.18E+08	Roseisle	Water
	C	5.40E+08	Roseisle	Dew
	C	5.10E+08	Minto	Dew

*Items with the same group letter are not significantly different with α=0.05

Table 4-14: Effect Significance of Retting Type, Location and Cross-Sectional Area on First Break Tensile Stress of Hemp Fibres

Source	DF	Type III SS	Pr > F
$R_{(H)j}$	2	$6.30x10^{18}$*	<.0001
$L_{(H)k}$	1	$3.23x10^{17}$	0.0614
$A_{(H)jkl}$	1	$2.26x10^{18}$*	<.0001
$RL_{(L)jk}$	2	$1.98x10^{18}$*	<.0001

*Indicates type III effect is significant to p<0.05

Table 4-15: Parameters for Retting Type, Location and Cross-Sectional Area on First Break Tensile Stress of Hemp Fibres

Parameter	First Break Tensile Stress(S)
B_0	$9.34x10^8 \pm 8.00x10^7$Pa*
B_1(Area)	$-4.73x10^{16} \pm 9.48x10^{15}$Pa/m^2*
L_{Minto}	$3.97x10^8 \pm 8.11x10^7$Pa*
R_{Dew}	$-1.78x10^8 \pm 7.98x10^7$Pa*
$R_{Unretted}$	$2.82x10^8 \pm 8.36x10^7$Pa*
$LR_{Minto\ Dew}$	$-4.27x10^8 \pm 1.15x10^8$Pa*
$LR_{Minto\ Unretted}$	$-4.98x10^8 \pm 1.16x10^8$Pa*

*Indicates type III effect is significant to p<0.05
N.B. All unlisted parameters are 0.00 by definition.

The observed effect of cross-sectional area is inversely proportional to the first break tensile stress of non-macerated hemp fibres (see **Table 4-14** and **Table 4-15**). Thus, area increases for a given retting type and growing location as the first break tensile stress decreases (see Figure 4.15). This is in agreement with previous literature that also observed similar results in terms of the effect of area on ultimate tensile stress and Young's Modulus of natural fibres (Baley 2002; Bourmaud and Baley 2009). Duval et al. (2011) reported that fibres had a tendency of breaking at their smallest point; however, they also observed one third of their fibre samples broke at a point with a larger than the average cross-section of that fibre. They attributed this failure to the higher probability of an apparently larger cross-section containing more defects including "kink bands"(Duval et al. 2011). Defects in natural fibres are produced either naturally during plant growth or mechanically during the decortication process and are irreversible (Baley 2002).

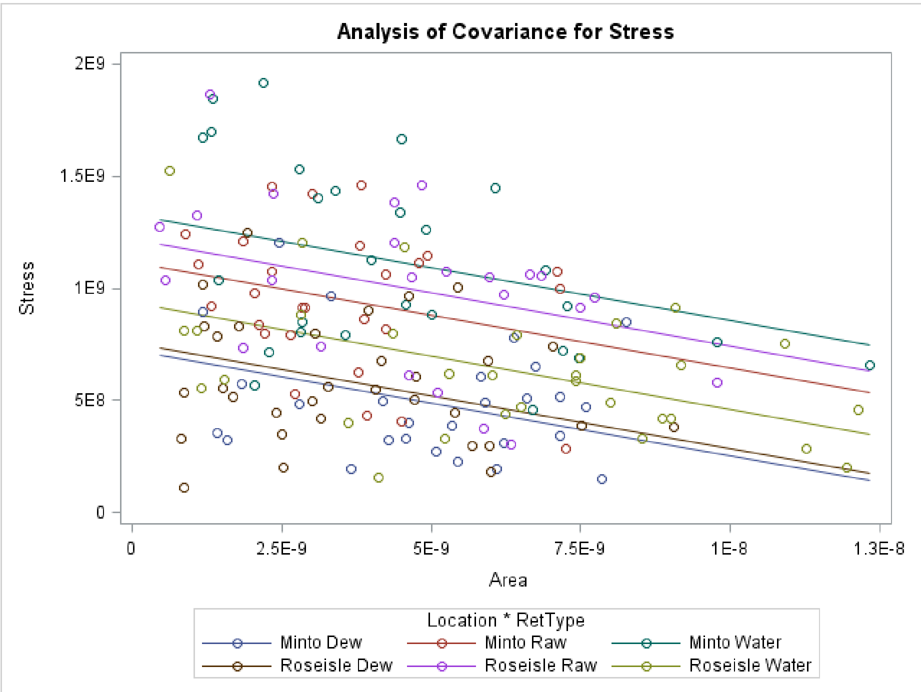

*Figure 4.15: Analysis of Covariance for First Break Stress vs. Area for Location*Retting Type of Hemp Fibres*

The type of retting process had a significant effect on first break tensile stress with unretted hemp fibres being stronger than dew retted hemp fibres (see **Table 4-13** and Figure 4.22). Dew retted fibres from both growing locations were significantly weaker with $\alpha=0.05$. This implies that the dew retting process did occur and had a detrimental effect on fibre strength. No significant difference was found between Minto and Roseisle dew retted fibres. This is in agreement with the visual observation regarding retting quality (see Figure 4.16 & Figure 4.17) wherein both appear to be the same darkish gray colour (D. E. Akin, Epps, et al. 2000).

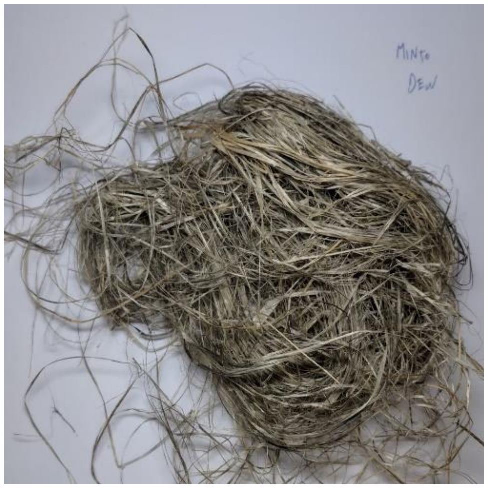

Figure 4.16: Minto Dew Retted Hemp Fibres

Figure 4.17: Roseisle Dew Retted Hemp Fibres

The effect of growing location on first break tensile stress was not significant; however, there was an interaction effect between growing location and retting type as the water retted fibres from Minto were significantly stronger than the water retted fibres from Roseisle (see **Table 4-13** and Figure 4.22). This is in agreement with the visual observation of the water retted hemp fibres (see Figure 4.18 and Figure 4.19), where the hemp fibres from Minto are lighter in colour than the water retted hemp fibres from Roseisle which appear darker and closer to dew retted fibres in colour and are possibly over-retted (D. E. Akin, Epps, et al. 2000). However, the unretted fibres from both growing locations (see Figure 4.20 and Figure 4.21) show little to no difference in colour that might explain the differences in final water retted colour let alone fibre strength. These unretted samples from both locations were water retted for the same number of days (see Section 3.4). This indicates that water retting can result in inherent variation and that tighter control over factors such as water to fibre straw ratio and temperature may be advisable in future works.

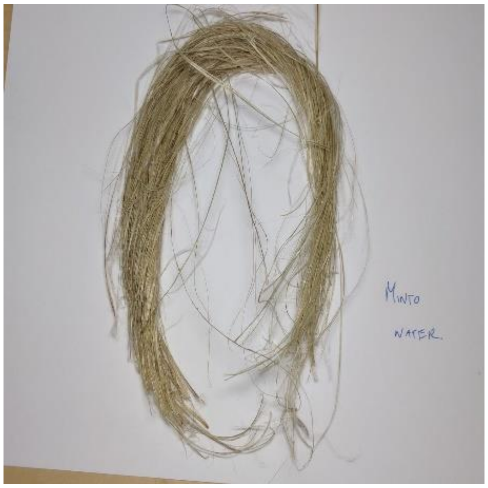

Figure 4.18: Minto Water Retted Hemp Fibres

Figure 4.19: Roseisle Water Retted Hemp Fibres

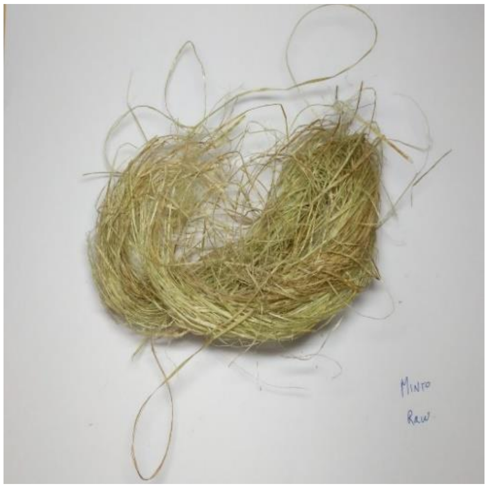

Figure 4.20: Minto Unretted Hemp Fibres

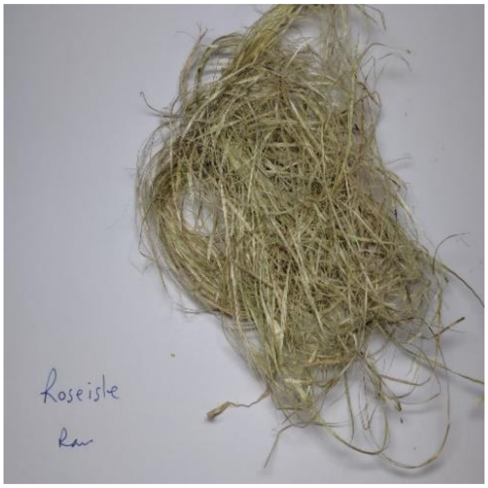

Figure 4.21: Roseisle Unretted Hemp Fibres

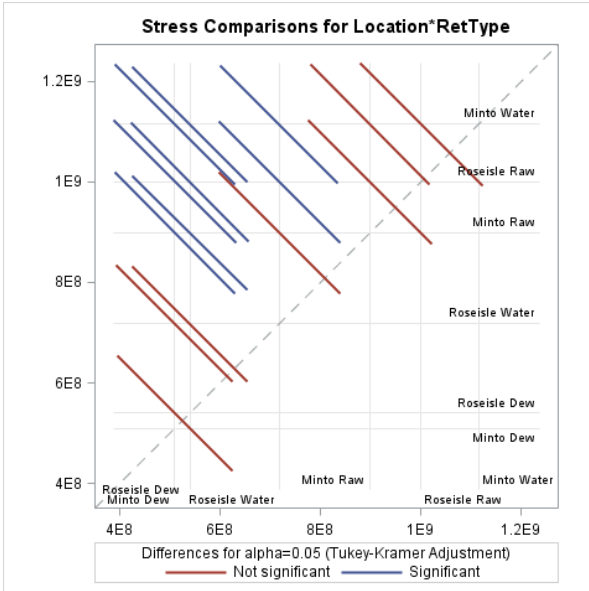

Figure 4.22: Tukey-Kramer Adjustment for First Break Stress Comparisons for Retting Type and Location of Hemp Fibres

4.3.3 FLAX

The Tukey-adjusted least-square means estimate of first break stress of non-macerated flax fibres for the different growing locations and retting types are presented in **Table 4-16**. The effect and significance of retting type, location and cross-sectional area and the GLM equation parameters for first break tensile stress of non-macerated flax fibres can be found in **Table** 4-17 and **Table 4-18** respectively. Similar to the results found regarding hemp fibres (see section 4.3.2) the effect of cross-sectional area had a negative effect on the first break tensile stress of non-macerated flax fibres (see **Table 4-17** and **Table 4-18**). Once again, as area increases for a given retting type and growing location the first break tensile stress decreases (see Figure 4.23). This is once again in agreement with previous literature and possibly caused by the higher probability of defects within the larger fibres (Baley 2002; Bourmaud and Baley 2009; Duval et al. 2011).

Table 4-16: Least-Square Means Estimate of First Break Stress of Flax Fibres

Group*		Stress LSMEAN	Location	RetType
A		7.63E+08	Portage	Unretted
A	B	6.82E+08	Minto	Unretted
A	B	6.55E+08	Portage	Dew
	B	5.68E+08	Minto	Water
	B	5.25E+08	Portage	Water
	B	5.18E+08	Minto	Dew

*Items with the same group letter are not significantly different with α=0.05

Table 4-17: Effect Significance of Retting Type, Location and Cross-Sectional Area on First Break Tensile Stress of Flax Fibres

Source	DF	Type III SS	Pr > F
$R_{(F)j}$	2	1.01×10^{18}*	0.0004
$L_{(F)k}$	1	1.53×10^{17}	0.1146
$A_{(F)jkl}$	1	2.59×10^{18}*	<.0001
$RL_{(F)jk}$	2	2.64×10^{17}	0.1179

*Indicates type III effect is significant to p<0.05

Table 4-18: Parameters for Retting Type, Location and Cross-Sectional Area on First Break Tensile Stress of Flax Fibres

Parameter	First Break Tensile Stress(S)
B_0	$8.29x10^8 \pm 5.65x10^7$Pa*
B_1 (Area)	$-4.65x10^{16} \pm 7.14x10^{15}$Pa/m²*
L_{Minto}	$4.29x10^7 \pm 5.76x10^7$Pa
R_{Dew}	$1.30x10^8 \pm 6.22x10^7$Pa*
$R_{Unretted}$	$2.38x10^8 \pm 6.14x10^7$Pa*
$LR_{Minto\ Dew}$	$-1.79x10^8 \pm 8.91x10^7$Pa*
$LR_{Minto\ Unretted}$	$-1.23x10^8 \pm 8.85x10^7$Pa

*Indicates type III effect is significant to $p<0.05$

N.B. All unlisted parameters are 0.00 by definition.

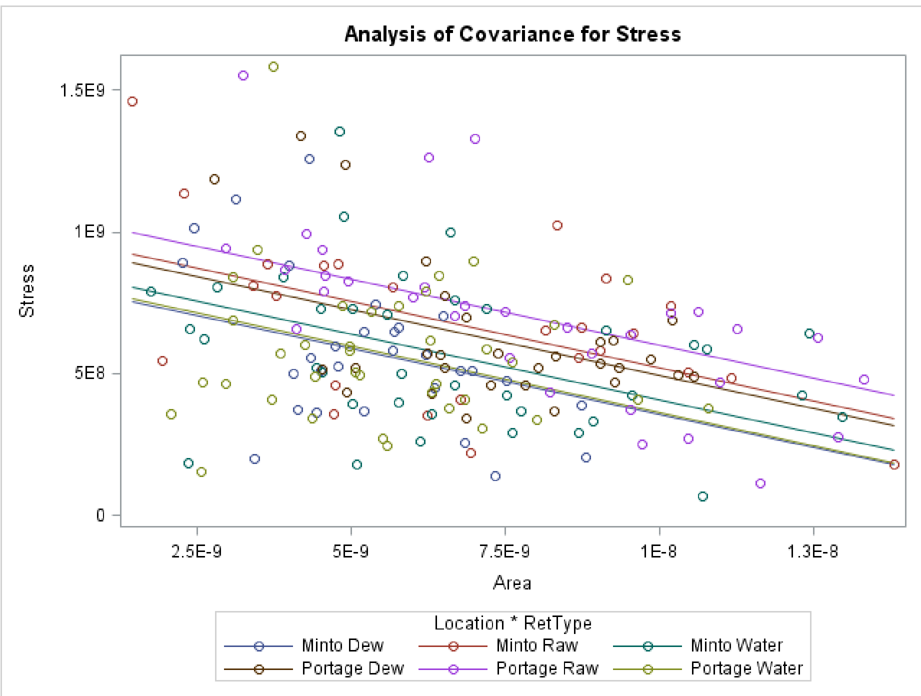

*Figure 4.23: Analysis of Covariance for First Break Stress vs. Area for Location*Retting Type of Flax Fibres*

When observing the effect of retting type there was a significant effect on first break tensile stress with unretted flax fibres being significantly higher than both dew retted and water retted flax fibres (see **Table 4-16** and Figure 4.24). There was no significant difference measured between dew retted and water retted flax fibres. Therefore, dew retting has occurred with results similar to water retting where both yielded weaker fibres than unretted fibres.

Contrary to the results found with hemp fibres (see Section 4.3.2), growing location had no significant effect on first break tensile stress of flax fibres nor was there a significant interaction effect with retting type (see Table 4-17).

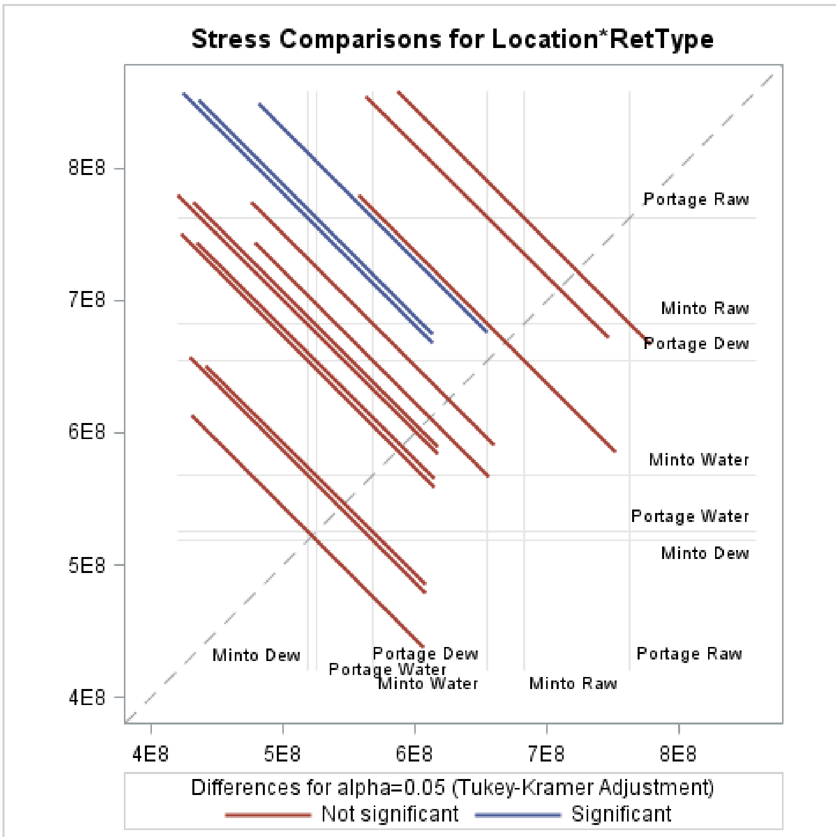

Figure 4.24: Tukey-Kramer Adjustment for First Break Stress Comparisons for Retting Type and Location of Flax Fibres

4.4 Summary and Future Work

4.4.1 Hemp

As described in section 4.1.2, fibre yield results for hemp first showed that the dew retted material from Roseisle was possibly over-retted due to high post-crusher hurd yield and high short fibre yield indicating a higher proportion of brittle fibres within those samples. Unfortunately, no comparison with the dew retted fibres from Minto could be made as those samples had been baled prior to collection of the samples resulting in partially processed material and an artificially high fibre yield. Regardless of the samples from Minto, the results from Roseisle imply that since over-retting of hemp fibres is possible then proper retting may be achievable through more careful monitoring. Soil microflora analysis in future works could also help explain if and why over-retting had occurred. Secondly, water retted samples had significantly higher yields than unretted samples which is in agreement with previous literature (Adamsen, Akin, and Rigsby 2002b; D E Akin 2013) and demonstrates that our fibre decortication method can be used to assess hemp retting efficiency. Finally, maceration using the Macerator 6620 (Les Machineries Pronovost Inc.), which uses flat rollers had no significant effect on hemp fibre yield which is also in agreement with previous literature (Foulk, Akin, and Dodd 2001). In future works, crimping using fluted rollers should be investigated for signs of increased retting efficiency.

SEM scans of hemp fibres from section 4.2.1 showed definite signs of pectinaceous matrix degradation in both water retted and dew retted samples, the former showing more degradation. This demonstrates that dew retting is in fact occurring in Manitoba climate though not as much as when water retting. SEM scans of fibres also revealed that the samples being prepared for single fibre tensile tests were in fact technical fibres composed of many elementary fibres.

Single fibre tensile tests of non-macerated hemp fibres (see section 4.3.2) first revealed that fibre cross-sectional area had a reverse effect on first break tensile stress. Within a given retting type and growing location, as fibre cross-section increases the first break tensile stress decreases. This is in agreement with previous literature (Baley 2002; Bourmaud and Baley 2009) and shows that the hemp fibres in this study are reacting similarly to those tested in past studies. This is possibly caused by the increase in probability of the presence of defects such as “kink-bands” with increased fibre size (Duval et al. 2011). Secondly, retting method had a significant effect on first break tensile stress with unretted hemp fibres being significantly stronger than dew retted hemp fibres. Dew retted fibres from both growing locations were found to be significantly weaker showing once again that dew retting has occurred in Manitoba climate and had a detrimental effect on fibre tensile strength. Lastly, the interaction effect between location and retting method had a significant effect on first break tensile stress with Minto water retted fibres being significantly stronger than Roseisle water retted fibres despite having identical retting types and similar initial states. This demonstrates an inherent variation in water retting results and encourages tighter controls in future works. Microflora analysis of the retting water may also help answer certain questions regarding this variation in future works. It is also worth noting, that in the case of hemp, variety was confounded with location. It is therefore possible that in the cases where location had significant effects, it may in fact be the effect of the difference in variety, the location or both.

4.4.2 FLAX

As described in section 4.1.4, fibre yield results for flax demonstrated an increase in yield when comparing water retting to unretted flax straw. This shows that the decortication method used in this study can be used to assess retting efficiency for both flax and hemp. Secondly, since the dew retted samples from Minto and Portage were both baled prior to sample collection they could only be compared to each other. This analysis showed a significantly higher fibre yield for the dew retted samples from Minto, implying that Minto was more retted than Portage. Pictures of the dew retted samples (see Figure 4.1 and Figure 4.2) confirm this as the dew retted fibres from Minto are darker in colour demonstrating a higher degree of retting (D. E. Akin, Epps, et al. 2000). This demonstrates that dew retting did occur at one of the locations, and therefore dew retting is possible in Manitoba climate and requires optimization. Finally, similarly to the results with hemp fibre, the flax fibre yield study showed that maceration using the Macerator 6620 (Les Machineries Pronovost Inc.) which uses flat rollers had no significant effect on fibre yield.

SEM scans of flax fibres from section 4.2.2 showed definite signs of degradation of the pectinaceous matrix enveloping the lignocellulosic fibres. Water retted fibres showed the most signs of degradation followed by the dew retted fibres. This demonstrates that dew retting in Manitoba does help degrade pectin and promote fibre separation. Resembling the SEM scans of hemp, SEM scans of flax fibres also demonstrate that fibres used in the single fibre tensile tests of this project are technical fibres composed of multiple elementary fibres.

Single fibre tensile tests of non-macerated flax fibres (see section 4.3.3) once again showed a significant negative relationship between fibre cross-sectional area and first break tensile stress. As cross-section of fibres increased within a given retting method and growing location, the first break tensile stress decreased. Secondly retting type had a significant effect on first break tensile stress with unretted fibres being stronger than both dew and water retted fibres.

There was no significant difference in first break tensile stress between dew and water retted fibres. This demonstrates that dew retting of flax in Manitoba climate is possible and has a detrimental effect on fibre strength that is comparable to water retted flax fibres.

4.4.3 FUTURE WORK

As stated in section 3.6, the sample preparation of fibres used for single fibre tensile tests using the Diastron single fibre testing equipment requires the samples to be free of frays, kinks and loose threads. As such, fibres stemming from macerated samples were not tested as damaged fibres from the maceration process would not be suitable for testing according to the Diastron SOP. Future works should focus on developing a method, possibly fibre bundle tensile testing, in order to study the effect of maceration on fibre tensile strength. Secondly, it has been stated by past literature that the large variation in natural fibre tensile strength and Young's Modulus is affected by many possible factors including the difficulty in accurately estimating fibre cross-sectional area (Shah, Nag, and Clifford 2016; Baley 2002). Future works should attempt to more accurately estimate fibre cross-sectional area by better accounting for the presence of lumens. Also, as stated in sections 4.1.2 and 4.1.4, maceration using the Macerator 6620 (Les Machineries Pronovost Inc.), which had flat rollers, produced no significant effect on fibre yield nor did it increase retting speed during macerated hemp retting trials. However, Foulk et al. saw a significant increase in retting efficiency when crimping with fluted rollers (Foulk, Akin, and Dodd 2001) and therefore crimping with fluted rollers in future works may be worth investigating to optimize dew retting in Manitoba. Finally, as stated in sections 4.4.1 and 4.4.2, microflora analysis of soil during dew retting and retting water during water retting in future works may help clarify certain questions regarding possible over-retting.

5 CONCLUSIONS

Hemp and flax are both important crops grown primarily for seed in Manitoba. Both these crops are referred to as bast fibre plants because of the cellulosic fibres that are found within the phloem region of the plant stem. However, for the most part, the straw containing these fibres are currently being burned or chopped and spread over fields making this waste stream a largely untapped renewable and sustainable resource. In order to extract fibre from bast fibre plants, the straw needs to undergo a process referred to as retting in which bacteria, fungi, enzymes and/or chemicals break down a matrix composed of pectins, hemicelluloses and lignin in which the fibre bundles are embedded. The retting process therefore releases these fibre bundles from the pectinaceous matrix and also separates the individual fibres within the bundles from each other. Numerous types of retting methods exist including: water retting, dew retting and enzymatic/chemical retting. Dew retting is a method of retting bast fibre straw whereby straw is laid in the field in swathes while rain and morning dew grant the necessary moisture for indigenous fungi and bacteria to degrade the pectinaceous matrix. Dew retting is an environmentally friendly and low-cost method of retting bast fibre straw however it is highly dependent on climate and has not been rigorously studied in Manitoba. Since the retting process is a critical component to extracting fibre from bast fibre straw methods such as maceration have been employed in past works to accelerate and increase retting efficiency. Maceration is a mechanical pre-treatment applied to the bast fibre straw prior to retting with the goal of disrupting the outer cuticle layer of the stem thereby granting micro-organisms easier access to the straw interior. The goal of this project was to study the effect of dew retting in Manitoba and maceration using flat-steel rollers on hemp and flax fibre properties. Water retted and unretted flax and hemp fibres were used a means of comparison. Results from the fibre yield study, SEM scans and single fibre tensile tests as well as visual observations showed that dew retting is possible in Manitoba climate. Dew retted fibres showed signs of over-retting using the custom decortication method developed for this project, SEM scans of dew retted fibres showed definite signs of degradation of the pectinaceous matrix enclosing the natural fibres and single fibre tensile tests showed a significant decrease in fibre tensile stress when compared to unretted fibres. Additionally, maceration using flat rollers had no significant effect on both hemp and flax fibre yields. Future works should focus on better estimating fibre cross-sectional area to more accurately calculate fibre strength, develop methods of testing damaged fibres for tensile strength and investigate the potential of crimping using fluted rollers for increased retting efficiency.

6 REFERENCES

Adamsen, A. P. S., D. E. Akin, and L. L. Rigsby. 2002a. “Chelating Agents and Enzyme Retting of Flax.” *Textile Research Journal* 72 (4): 296–302. doi:10.1177/004051750207200404.

———. 2002b. “Chemical Retting of Flax Straw Under Alkaline Conditions.” *Textile Research Journal* 72 (9): 789–94. doi:10.1177/004051750207200907.

Akin, D. E., R. Dodd, W. Perkins, G. Henriksson, and K.-E. L. Eriksson. 2000. “Spray Enzymatic Retting: A New Method for Processing Flax Fibers.” *Textile Research Journal* 70 (6): 486–94. doi:10.1177/004051750007000604.

Akin, D. E., H. H. Epps, D. D. Archibald, and H.S. S. Sharma. 2000. “Color Measurement of Flax Retted by Various Means.” *Textile Research Journal* 70 (10): 852–58. doi:10.1177/004051750007001002.

Akin, D. E., D. Slomczynski, L. L. Rigsby, and K.-E. L. Eriksson. 2002. “Retting Flax with Endopolygalacturonase from Rhizopus Oryzae.” *Textile Research Journal* 72 (1): 27–34. doi:10.1177/004051750207200105.

Akin, D E. 2013. “Linen Most Useful: Perspectives on Structure, Chemistry, and Enzymes for Retting Flax.” *ISRN Biotechnology* 2013: 23. doi:doi:10.5402/2013/186534.

Akin, Danny E., Brian Condon, Miryeong Sohn, Jonn A. Foulk, Roy B. Dodd, and Luanne L. Rigsby. 2007. “Optimization for Enzyme-Retting of Flax with Pectate Lyase.” *Industrial Crops and Products* 25 (2): 136–46. doi:10.1016/j.indcrop.2006.08.003.

Andresen, Stina Troldtoft, and Sabine Karg. 2011. “Retting Pits for Textile Fibre Plants at Danish Prehistoric Sites Dated between 800 B.C. and A.D. 1050.” *Vegetation History and Archaeobotany* 20 (6): 517–26. doi:10.1007/s00334-011-0324-0.

Baley, C. 2002. “Analysis of the Flax Fibres Tensile Behaviour and Analysis of the Tensile Stiffness Increase” 33: 939–48.

Bennett, Sarita J., Rebecca Snell, and David Wright. 2006. “Effect of Variety, Seed Rate and Time of Cutting on Fibre Yield of Dew-Retted Hemp.” *Industrial Crops and Products* 24

(1): 79–86. doi:10.1016/j.indcrop.2006.03.007.

Booth, I., A. M. Goodman, S. A. Grishanov, and R. J. Harwood. 2004. “A Mechanical Investigation of the Retting Process in Dew-Retted Hemp (Cannabis Sativa).” *Annals of Applied Biology* 145 (1): 51–58. doi:10.1111/j.1744-7348.2004.tb00358.x.

Bourmaud, Alain, and Christophe Baley. 2009. “Rigidity Analysis of Polypropylene / Vegetal Fibre Composites after Recycling.” *Polymer Degradation and Stability* 94 (3). Elsevier Ltd: 297–305. doi:10.1016/j.polymdegradstab.2008.12.010.

Candilo, M. Di, P. M. Bonatti, C. Guidetti, B. Focher, C. Grippo, E. Tamburini, and G. Mastromei. 2010. “Effects of Selected Pectinolytic Bacterial Strains on Water-Retting of Hemp and Fibre Properties.” *Journal of Applied Microbiology* 108 (1): 194–203. doi:10.1111/j.1365-2672.2009.04409.x.

CIC. 2017. “Composites Innovation Centre.” http://www.compositesinnovation.ca/?doing_wp_cron=1499812771.1729369163513183593750.

Duval, Antoine, Alain Bourmaud, Laurent Augier, and Christophe Baley. 2011. “Influence of the Sampling Area of the Stem on the Mechanical Properties of Hemp Fibers” 65: 797–800. doi:10.1016/j.matlet.2010.11.053.

Environment Canada. 2017. “Historical Data - Climate - Environment and Climate Change Canada.” http://climate.weather.gc.ca/historical_data/search_historic_data_e.html.

FCC. 2017. “Flax Council Of Canada.” https://flaxcouncil.ca/.

Fila, G, Lm Manici, and F Caputo. 2001. “In Vitro Evaluation of Dew Retting of Flax by Fungi from Southern Europe.” *Annals of Applied Biology* 138: 343–51. http://onlinelibrary.wiley.com/doi/10.1111/j.1744-7348.2001.tb00119.x/abstract.

Foulk, Jonn A., Danny E. Akin, and Roy B. Dodd. 2001. “Processing Techniques for Improving Enzyme-Retting of Flax.” *Industrial Crops and Products* 13 (3): 239–48. doi:10.1016/S0926-6690(00)00081-9.

———. 2008. “Processability of Flax Plant Stalks into Functional Bast Fibers.” *Composite*

Interfaces 15 (2–3): 147–68. doi:10.1163/156855408783810902.

Haag, Katharina, and J Mussig. 2016. "Scatter in Tensile Properties of Flax Fibre Bundles: Influence of Determination and Calculation of the Cross-Sectional Area." *Journal of Materials Science* 51 (17): 7907–17. doi:10.1007/s10853-016-0052-z.

Henriksson, G, D E Akin, R T Hanlin, C Rodriguez, D D Archibald, L L Rigsby, and K L Eriksson. 1997. "Identification and Retting Efficiencies of Fungi Isolated from Dew-Retted Flax in the United States and Europe." *Applied and Environmental Microbiology* 63 (10): 3950–56.

Henriksson, Gunnar, Danny E Akin, Luanne L Rigsby, Nirav Patel, and Karl-Erik L Eriksson. 1997. "Influence of Chelating Agents and Mechanical Pretreatment on Enzymatic Retting of Flax." *Textile Research Journal* 67 (11): 829–36. doi:10.1177/004051759706701107.

Hu, Wei, Minh-Tan Ton-That, Johanne Denault, Denis Rho, Jianzhong Yang, and Peter C.K. Lau. 2012. "Comparison Between Dew-Retted and Enzyme-Retted Flax Fibers as Reinforcing Material for Composites." *Polymer Engineering and Science* 52: 165–71. doi:10.1002/pen.

Jankauskiene, Zofija, Bronislava Butkute, Elvyra Gruzdeviene, Jurgita Cesevičiene, and Ana Luisa Fernando. 2015. "Chemical Composition and Physical Properties of Dew- and Water-Retted Hemp Fibers." *Industrial Crops and Products* 75: 206–11. doi:10.1016/j.indcrop.2015.06.044.

Kaczmar, Jacek W., Joanna Pach, and Christoph Burgstaller. 2011. "The Chemically Treated Hemp Fibres to Reinforce Polymers." *Polimery/Polymers* 56 (11–12): 817–22.

Liu, Ming, Dinesh Fernando, Geoffrey Daniel, Bo Madsen, Anne S. Meyer, Marcel Tutor Ale, and Anders Thygesen. 2015. "Effect of Harvest Time and Field Retting Duration on the Chemical Composition, Morphology and Mechanical Properties of Hemp Fibers." *Industrial Crops and Products* 69. Elsevier B.V.: 29–39. doi:10.1016/j.indcrop.2015.02.010.

Liu, Ming, Diogo Alexandre Santos Silva, Dinesh Fernando, Anne S. Meyer, Bo Madsen, Geoffrey Daniel, and Anders Thygesen. 2016. "Controlled Retting of Hemp Fibres: Effect of Hydrothermal Pre-Treatment and Enzymatic Retting on the Mechanical Properties of Unidirectional Hemp/epoxy Composites." *Composites Part A: Applied Science and Manufacturing* 88. Elsevier Ltd: 253–62. doi:10.1016/j.compositesa.2016.06.003.

MAFRI. 2017. "Industrial Hemp Production and Management | Manitoba Agriculture | Province of Manitoba." *Manitoba Agricultural, Food and Rural Initiatives*. http://www.gov.mb.ca/agriculture/crops/production/print,hemp-production.html.

Martin, Nicolas, Nicolas Mouret, Peter Davies, and Christophe Baley. 2013. "Influence of the Degree of Retting of Flax Fibers on the Tensile Properties of Single Fibers and Short Fiber/polypropylene Composites." *Industrial Crops and Products* 49. Elsevier B.V.: 755–67. doi:10.1016/j.indcrop.2013.06.012.

Masto, A. Del, F. Trivaudey, V. Guicheret-Retel, V. Placet, and L. Boubakar. 2017. "Nonlinear Tensile Behaviour of Elementary Hemp Fibres: A Numerical Investigation of the Relationships between 3D Geometry and Tensile Behaviour." *Journal of Materials Science* 52 (11). Springer US: 6591–6610. doi:10.1007/s10853-017-0896-x.

Moriana, Rosana, Francisco Vilaplana, Sigbritt Karlsson, and Amparo Ribes. 2014. "Correlation of Chemical, Structural and Thermal Properties of Natural Fibres for Their Sustainable Exploitation." *Carbohydrate Polymers* 112. Elsevier Ltd.: 422–31. doi:10.1016/j.carbpol.2014.06.009.

Musialak, Magdalena, Magdalena Wróbel-Kwiatkowska, Anna Kulma, Eligia Starzycka, and Jan Szopa. 2008. "Improving Retting of Fibre through Genetic Modification of Flax to Express Pectinases." *Transgenic Research* 17 (1): 133–47. doi:10.1007/s11248-007-9080-4.

Nair, Gopu, Raveendran, Denis Rho, Varoujan Yaylayan, and Vijaya Raghavan. 2013. "Microwave Assisted Retting – A Novel Method of Processing of Flax Stems." *Biosystems Engineering* 116 (4). IAgrE: 427–35. doi:10.1016/j.biosystemseng.2013.10.004.

Nair, Gopu Raveendran, Darwin Lyew, V. Yaylayan, and Vijaya Raghavan. 2015. "Application of Microwave Energy in Degumming of Hemp Stems for the Processing of Fibres." *Biosystems Engineering* 131. Elsevier Ltd: 23–31. doi:10.1016/j.biosystemseng.2014.12.012.

Paridah, M. T., A. B. Basher, S. SaifulAzry, and Z Ahmed. 2011. “Retting Process of Some Bast Plant Fibers and Its Effect on Fiber Quality: A Review.” *Bio Resources* 6 (Sur 2005): 5260–5281. doi:10.15376/biores.6.4.5260-5281.

PIHP. 2017. “About Company - Plains Hemp | Plains Industrial Hemp Processing Ltd.” http://plainshemp.com/about-company/.

Ribeiro, Alexandra, Philippe Pochart, Arnaud Day, Sarah Mennuni, Pierre Bono, Jean Luc Baret, Jean Louis Spadoni, and Irène Mangin. 2015. “Microbial Diversity Observed during Hemp Retting.” *Applied Microbiology and Biotechnology* 99 (10): 4471–84. doi:10.1007/s00253-014-6356-5.

Ruan, Peiying, Vijaya Raghavan, Yvan Gariepy, and Jianmin Du. 2015. “Characterization of Flax Water Retting of Different Durations in Laboratory Condition and Evaluation of Its Fiber Properties.” *BioResources* 10 (2): 3553–63. doi:10.15376/biores.10.2.3553-3563.

Seed Manitoba. 2017. “Seed Solutions.” http://www.seedmb.ca/.

Shah, Darshil U., Ranajit K. Nag, and Mike J. Clifford. 2016. “Why Do We Observe Significant Differences between Measured and ‘back-Calculated’ Properties of Natural Fibres?” *Cellulose* 23 (3). Springer Netherlands: 1481–90. doi:10.1007/s10570-016-0926-x.

Sisti, Laura, Grazia Totaro, Micaela Vannini, Paola Fabbri, Susheel Kalia, Alessandro Zatta, and Annamaria Celli. 2016. “Evaluation of the Retting Process as a Pre-Treatment of Vegetable Fibers for the Preparation of High-Performance Polymer Biocomposites.” *Industrial Crops and Products* 81. Elsevier B.V.: 56–65. doi:10.1016/j.indcrop.2015.11.045.

Virk, A S, W Hall, and J Summerscales. 2012. “Modulus and Strength Prediction for Natural Fibre Composites.” *Materials Science and Technology* 28 (7): 864–71. doi:10.1179/1743284712Y.0000000022.

Wilcox, Rand. 2012. *Introduction to Robust Estimation & Hypothesis Testing*. Third. Waltham, MA, USA: Elsevier Inc.

Zeng, Xuesen, Sacha J. Mooney, and Craig J. Sturrock. 2015. “Assessing the Effect of Fibre Extraction Processes on the Strength of Flax Fibre Reinforcement.” *Composites Part A: Applied Science and Manufacturing* 70. Elsevier Ltd: 1–7.

doi:10.1016/j.compositesa.2014.12.004.

Zuccarello, B., and M. Zingales. 2017. “Toward High Performance Renewable Agave Reinforced Biocomposites: Optimization of Fiber Performance and Fiber-Matrix Adhesion Analyses.” *Composites Part B: Engineering*. Elsevier Ltd. doi:10.1016/j.compositesb.2017.04.011.