Evaluation of *Brassica* Fibre for Textile and Spinning Properties

By

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Abstract

Brassica napus L., which is commonly known as canola, is the largest sources of edible oil in Canada. The remaining plant material, such as the stem, remains unused for any immediate application and is returned to the soil for decomposition. An investigation has been conducted to extract, characterize and modify the fibre materials from *B. napus* stems for textile and apparel applications. In order to find the optimum retting conditions for retting time, four different retting parameters were evaluated including, retting temperature, material liquor ratio, water exchange and the reuse of retted water. It was discovered that the virgin-retted fibres from *Brassica* plants exhibit most of the required textile properties including dye absorbency, strength, and thermal behaviour. However, the virgin-retted fibres do not exhibit the required spinning (yarn transformation) properties (softness, flexibility and individual fibre entity). In order to modify the *Brassica* fibres for spinnability, three treatment methods were applied: 1) alkali, acid and softener treatment; 2) pectinase enzyme treatment; and 3) enhanced enzyme treatment. According to Method 5 of the American Association of Textile Chemists and Colorists (AATCC), *Brassica* fibers obtained from treatments 2 and 3 showed similar spinning properties, and demonstrated superior spinning properties to *Brassica* fibres obtained from treatment one. To determine the variability of the cultivars upon textile and spinning properties, seeds from twenty different *Brassica* cultivars consisting of three different species, *B. napus*, *B. juncea* L. and *B. rapa* L., were collected, planted, and harvested upon reaching physiological maturity. The virgin water-retted fibre samples were then treated with pectinase enzyme, and different spinning properties (stiffness, softness, individual fibre entity) and textile properties (fibre decomposition temperature, tenacity and dye absorbency) of enzyme-treated samples were evaluated. The

current research suggests that producing fibers from canola stubble and stems could be an additional income source for canola growers.

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Chapter 1 Introduction

"Food, clothes and shelter" are the three traditional basic needs for humans (Danton, 1990). Since ancient times, people have been making clothes/apparel from available natural fibres such as cotton, silk and wool. According to American Standard Testing Materials (ASTM), a textile fibre is defined as, "…a generic term for any one of the various types of matter that form the basic elements of a textile and … is characterized by having a length at least 100 times its diameter" (ASTM International, 2015).

Textile fibres are divided into two main sub-groups: natural and man-made. Synthetic fibres, for example polyester, nylon, acrylic have been produced from chemical compounds; whereas viscose, modal, tencel fibres have been modified or regenerated from different natural sources (Mather & Wardman, 2015). Natural fibres include cotton, silk, wool. Natural fibres may be obtained from animal sources such as sheep (wool), or from different portions of plants such as bast (flax, hemp), seed (cotton, kapok) or leaves (sisal, banana) (Kozlowski, Mackiewicz-Talarczyk, Muzyczek, & Barriga-Bedoya, 2012). Of all the natural fibre sources, cotton is the most available and most widely used (B. J. Collier, Bide, & Tortora, 2009).

Naturally-sourced fibres are being replaced gradually by man-made fibres due to their multiple advantages, such as lower cost and better durability (Thomas, Paul, Pothan, & Deepa, 2011). Currently, polyester is the most widely used fibre, superseding cotton in 2002 (Carmichael, 2015). Petroleum resources, such as terephthalic acid and ethylene glycol, are used for producing different synthetic fibres (B. J. Collier et al., 2009). By all accounts, the raw materials for

synthetic fibres are rapidly depleting (Daun, 2011). Production of man-made fibre is responsible for an increase in environmental pollution (Rana, Pichandi, Parveen, & Fangueiro, 2014; Shen, Worrell, & Patel, 2010). Whereas, cotton production uses vast amounts of water, pesticides, herbicides and defoliants that are harmful to ecosystem health (Ebskamp, 2002; Rana et al., 2014). Therefore, scientists and researchers are now evaluating alternative resources to develop fibres that are sustainable and cause minimal environmental damage (Thomas et al., 2011).

Apparel articles require many processing stages, starting with the raw material (fibre), followed by spinning (yarn manufacturing), weaving or knitting (fabric manufacturing), wet processing (scouring, bleaching, dyeing, printing and finishing), and finally, garment production (Collier et al., 2009; Hatch, 1993; Hunter, 2007). High quality finer spun yarn (linear density = 10 tex – 50 tex) is required for apparel end uses, but such yarn can be produced using solely cotton spinning systems, such as ring frame and rotor frame. Increasing demand for polyester fibre is in part due to the fact that cotton spinning systems are used to produce the yarn for apparel end uses. Other fibres such as flax and hemp are falling far behind in apparel applications as these fibres cannot be processed using cotton spinning systems. It is worth mentioning that the global apparel market size is 467 billion USD, which is about two and half times the size of the aeronautics market (187 billion USD) and about one eighth times the size of agrifood sector (3837 billion USD) (based on global sector reports of February 2016 by Euler Hermes) (Euler Hermes, 2016).

Researchers at the University of Manitoba are in the forefront of sustainable fibre development using unused plant material and have discovered that textile fibres can be extracted from the canola plant using a water retting process. The extracted fibres may have the required textile

properties for successful apparel fibres such as breaking strength, dye absorbency, thermal property. However, it was previously found that virgin *B. napus* fibre lacks the needed cotton spinning properties (Sevenhuysen & Rahman, 2015). Nonetheless, the impact of different Brassica species or cultivars on fibre quality is unknown. One of the major limitations of the work (Sevenhuysen & Rahman, 2015) was that the researchers used unknown *Brassica* cultivars.

Different *Brassica* species such as *Brassica oleracea, B. napus, B. juncea* and *B. rapa* are cultivated in different regions of the world. *Brassica oleracea* is commonly used for vegetables, forage and ornamental purposes. In Europe, *B. napus* and *B. rapa* have been used traditionally as oilseed species called "rapeseed" (Diederichsen & McVetty, 2011). In Canada, *B. napus, B. rapa* and *B. juncea* cultivars, named "canola", are used widely for producing edible oils after reducing erucic acid content to < 2 % through breeding (Daun, 2011). *Brassica* plants are mainly cultivated for their seeds and produce the largest source of edible oil. Following harvest, the remaining plant parts, such as stems, are left in the field to decompose. This inexpensive biomaterial has the potential to be a significant naturally sourced fibre.

The primary objective of this study was to extract fibre from known *Brassica* cultivars through retting. The extracted virgin fibre measured for apparel application properties, and modified enzymatically to improve its cotton spinning properties. As the research progressed, a second objective was identified which focused on the effect of different *Brassica* cultivars on textile and cotton spinning properties.

Chapter 2 Literature Review

2.1 Textile Properties and their importance

To become textile fibre, fibre materials need to meet minimum requirements for textile performance, including durability, comfort, aesthetic appeal, maintenance and health and safety protection (Hatch, 1993). In terms of durability, textile materials such as fibre, yarn or fabric must have a certain level mechanical strength to absorb regular wear and tear following regular use (Hatch, 1993). The textiles must have the ability to retain their original position without changing shape, size or color during regular washing (Hatch, 1993).

Fibre possesses a set of numerous physical, chemical, mechanical and thermal properties for textile, apparel and industrial applications. For example, fibre with the ability to regain high moisture can be used as an absorbent for medical textiles such as dressings and hygienic products like diapers (Zhong, 2013). In the case of medical applications, fibres used in wound dressings need to be soft, flexible and anti-bacterial for proper healing and for protection of the wound (Zhong, 2013). For the preparation of composite materials, mechanical properties such as tenacity and extensibility and thermal properties such as melting or decomposition temperature are important for determining processing parameters as well as end uses of the final product (Booth, 1968).

Importance of mechanical properties

Textile fibers are subjected to different stress behaviors during manufacturing (spinning) and during end use (Hatch, 1993). The ability to withstand the tensile force (tenacity) is an important parameter for manufacturing of yarn in different processing stages such as spinning frame,

winding, warping, sizing and fabric formation (weaving and knitting). The strength of yarn also depends on the strength of the fibres (Booth, 1968). When people wear apparel, stress is applied on the apparel due to regular movement of the body portions such as knees, elbows and buttocks. The apparel has to be able to withstand this stress applied due to the regular movement of the human body (Hatch, 1993).

Importance of thermal properties

Textile fibres and fabrics are subjected to coloring for aesthetic appeal. A pretreatment process (scouring) is necessary to dye the cellulosic fibres such as cotton. During scouring, cellulosic fibres (cotton) are treated at 95 ˚C with sodium hydroxide solution (Mather & Wardman, 2015). Dyeing is a process of coloring the textile material at varying temperatures according to the nature of the dyes and fibres (B. J. Collier et al., 2009). For dyeing of cellulosic fibres with reactive dye, the dyeing temperature is raised to 90 ˚C (King, 2007). Fibres and fabrics must retain their original structure at high temperatures during different preparation stages like dyeing (135 ˚C - 140 ˚C for blended fabric) and heat setting (220 ˚C).

Dyeing of cellulosic fibres

Cellulosic fibres can be dyed with direct dyes, vat dyes, sulphur dyes and reactive dyes (B. J. Collier et al., 2009). Direct dyes exhibit poor color fastness following laundering, whereas vat dyes demonstrate good fastness properties (B. J. Collier et al., 2009). However, vat dyes can be costly. On the other hand, sulphur dyes cause light degradation of cellulosic fibres like cotton (B. J. Collier et al., 2009). Among all the dyes, reactive dyes are most widely used for dyeing of cellulosic fibers as they are easy to apply and produce a wide range of colors (B. J. Collier et al.,

2009). The presence of an –OH group in cellulose plays an important role in dyeing by creating a covalent bond with reactive dye molecules (Dye-SO_2-CH_2-CH_2-OSO_3Na) in the presence of NaOH (Mather & Wardman, 2015).

Evaluation of dyeing performance (color fastness)

The AATCC definition of color fastness is:

> "The resistance of a material to change in any of its color characteristics, to transfer of its colorant(s) to adjacent materials, or both, as a result of exposure of the material to any environment that might be encountered during the processing, testing, storage, or use of the material" (AATCC, 2010b).

Textile end products are always subjected to various conditions including moisture, light, heat, abrasion during processing and end uses. During dyeing, all the dyestuffs are not properly fixed with textiles (Trotman, 1975). If the unfixed dyes are not removed properly from the fibres or fabric, they will be easily removed from textiles during end uses and maintenance such as laundering (B. J. Collier & Epps, 1999). These unfixed dyes can be properly removed by washing with soap during processing, and the dyeing performances can be evaluated by the rating of colorfastness, using grey scale and multifibre test fabrics (AATCC, 2010a). Grey scale is used for evaluating the color change and the staining of the fibres, and samples can be evaluated in 9 different grades such as 1, 1-2, 2, 2-3, 3, 3-4, 4, 4-5 and 5. Grade 1 represents the highest staining or color change, and grade 5 represents no staining or color change during washing (B. J. Collier & Epps, 1999). A multifibre test fabric is composed of six different types of fibres containing acetate, cotton, polyamide, silk, viscose and wool (B. J. Collier & Epps,

1999). The purpose of using the multifibre fabric is to evaluate the staining of the color during washing or regular uses (B. J. Collier & Epps, 1999).

2.2 Spinning properties

Fibre spinnability is determined by properties like fibre length, strength, fineness, elongation, trash, stiffness, individuality and twist ability (Gordon, 2007). Table 2.1 summarizes a ranking of the importance of different fibre properties using three cotton spinning methods: ring, rotor and air jet spinning (Gordon, 2007). Long staple fibres are better than short staple fibres because long staples fibres are capable of producing softer yarn when other fibre parameters remain unchanged (B. J. Collier et al., 2009). Fineness, represented by micronaire value, is the number of fibres present in a known cross sectional area (B. J. Collier et al., 2009). Finer fibre produces stronger yarn, as there are more fibres present in a cross section (Gordon, 2007). Fibre stiffness is another important term for yarn preparation (Hearle, 2007). During the spinning process, fibres are twisted at a high rate of speed. A rigid fibre exhibits a higher tendency to break during the application of twist for preparing yarn. Flexural rigidity increases with the increase of the diameter of the fibres; thus the fibres need to be individualized. Bast fibres need additional processing steps to individualize the fibers as they are attached or glued with the adjacent fibers, which is not necessarily the case for cotton (Rana et al., 2014).

Table 2.1. Importance of fibre properties of cotton for different short-staple spinning systems (Developed from Gordon, 2007 and Lord, 2003).

Priority ranking of different fibre properties	Ring	Rotor	Air-jet
1	Flexibility	Flexibility	Flexibility
2	Single fibre entity	Single fibre entity	Single fibre entity
3	Length	Strength	Length
4	Strength	Fineness	Trash
5	Fineness	Length	Fineness
6	N/A	Trash	Strength

Mechanism of cotton spinning

For cotton ring spinning, fibres pass through different sections referred to as blow room, carding, draw frame, simplex or roving frame and ring frame (Hunter, 2007; Lawrence, 2007). In the blow room, fibres tufts are opened from the bale and the heavy particles are separated. A thin layer of fibre films / sliver is produced in the carding machine, and the fibres are also individualized and parallelized by the carding action between the cylinder and the flat of the machine (Lawrence, 2007). A mixing and drawing of the slivers is carried out in the drawframe for reducing the weight per unit length of fibres and making the fibre more parallelized (Hunter, 2007). In the meantime, uniformity of weight per unit length of the fibres is also controlled in the drawframe by a roller drafting system (Figure 2.1) with the support of a modern auto-leveling mechanism (Hunter, 2007). Draft and twist are applied on the slivers for preparing roving that will finally produce the yarn in the ring frame by a technique called ballooning (Hunter, 2007).

In the carding machine, fibre mass enters into the inlet region and moves towards the outlet region. These fibres become individualized and parallelized by the slow moving flats and finally

produce a filmy web of slivers through the carding action between cylinder and flat. The fibres that attached with the flats are removed at the flat cleaning region by brush rollers.

Fibre blending and drafting is carried out in the drafting zone of a draw frame (Hunter, 2007). A variety of drafting roller arrangements such as 4 over 4 rollers or 4 over 3 rollers are available for draw frame machines (Hunter, 2007). Figure 2.1 indicates a setting of a 4 over 3 roller arrangements where four top rollers (1, 2, 3 and 7) are placed on top of three bottom rollers (4, 5 and 6) and sliver passes in between them (Hunter, 2007).

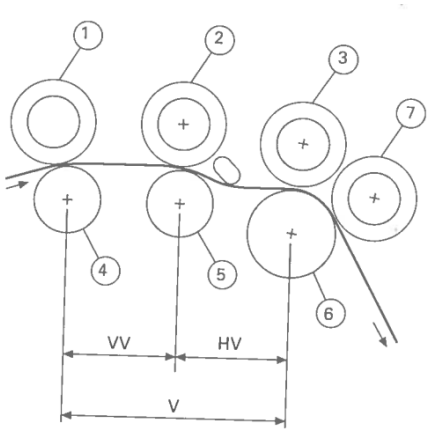

Figure 2.1. A roller drafting system of a draw frame machine with four top rollers (1, 2, 3, and 7) and three (4, 5, and 6) bottom rollers (From Hunter, 2007).

About 15,000 - 25,000 twists/min are applied onto the roving with a delivery speed of 20 - 30 m/min for producing yarn in the ring frame (Hunter, 2007). Slivers are fed through the guide (L) to the space between the ring (R) and traveller (T), which applies twist onto the yarn using circular movements of the traveller onto the edge of the ring forming a balloon on the sliver (Booth, 1975). Finally, the yarns are wound into a suitable package.

2.3 Textile materials

Textile fibres can be obtained from numerous sources. Some fibres are obtained from natural sources whereas others are produced synthetically (Booth, 1968; A. M. Collier, 1970a; B. J. Collier et al., 2009; B. J. Collier & Epps, 1999). Natural fibres can be collected from plants, animals, sea weeds, and mineral sources (Kozlowski, Mackiewicz-Talarczyk, Muzyczek, et al., 2012). A detailed classification of textiles fibres is given in Figure 2.2 (Mather & Wardman, 2015).

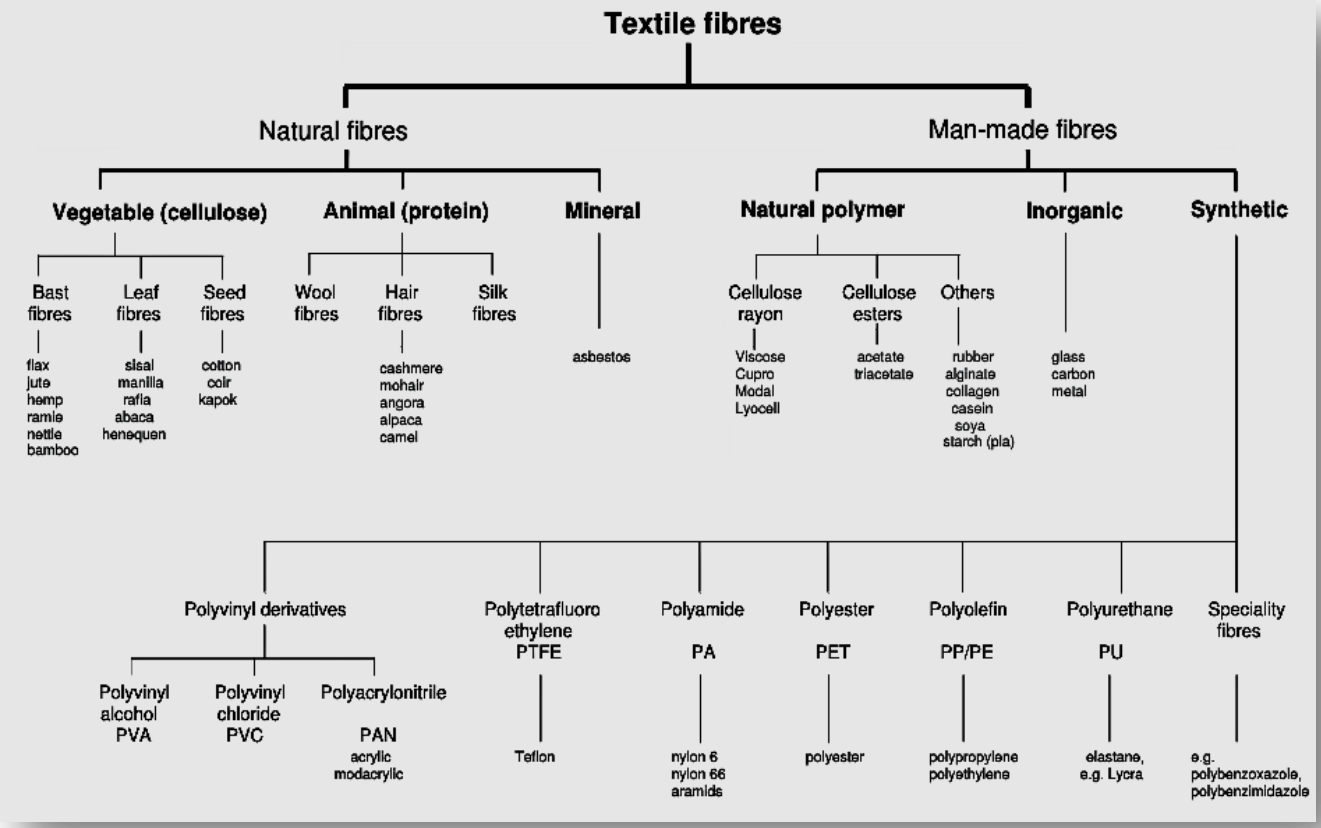

Figure 2.2. The classification of textile fibres (From Mather & Wardman, 2015).

Table 2.2 shows the different production types of natural and man-made fibres produced all over the world in 2009. According to a survey on fibre production in 2009, the relative market positions of natural fibres and manmade fibres were 37.4 % and 62.6 %, respectively (Kozlowski, Mackiewicz-Talarczyk, Muzyczek, et al., 2012). Among all of the natural and man-made fibres, cotton and polyester had the largest share at 35.7 % and 45.3 %, respectively (Figure 2.3).

Table 2.2. The production of textile fibres in 2009 (Kozlowski et al., 2012b).

Fibre	Production (millions of Tonnes)
Cotton	25.2
Wool	1.1
Silk	0.102
Polyester	31.9
Polyamide	3.5
Polypropylene	2.6
Polyacrylic	1.9

2.4 Man-made fibres

Man-made fibres are divided into two major sub-groups, regenerated fibres and synthetic fibres (B. J. Collier et al., 2009), and produce filament yarn. Regenerated fibres are made from natural polymer materials such as rayon produced from wood pulp, whereas synthetic fibres are made from synthetic polymeric materials such as acrylic fibres produced from acrylonitrile units (B. J. Collier et al., 2009). In 1930, Wallace Carothers first invented synthetic fibres [Nylon (polyamide) and polyesters](Carothers, 1931; Mcintyre, 1998). The first commercial production of polyester was started just after World War II.

Polyester fibre

Polyester fibres are produced from the controlled reaction under vacuum conditions between terephthalic acid and ethylene glycol at 280 ˚C – 290 ˚C (Mcintyre, 1998). The process of producing polyester fibres/filaments is called melt spinning (B. J. Collier et al., 2009). In melt spinning, the basic components, i.e., terephthalic acid and ethylene glycol, are reacted and produce a viscous liquid of polyester (PET), which is pumped through a spinneret under a controlled flow rate (B. J. Collier et al., 2009). A cool air current is applied to the molten polymers emerging from the spinneret hole to solidify the polymers for producing polyester fibres (B. J. Collier et al., 2009).

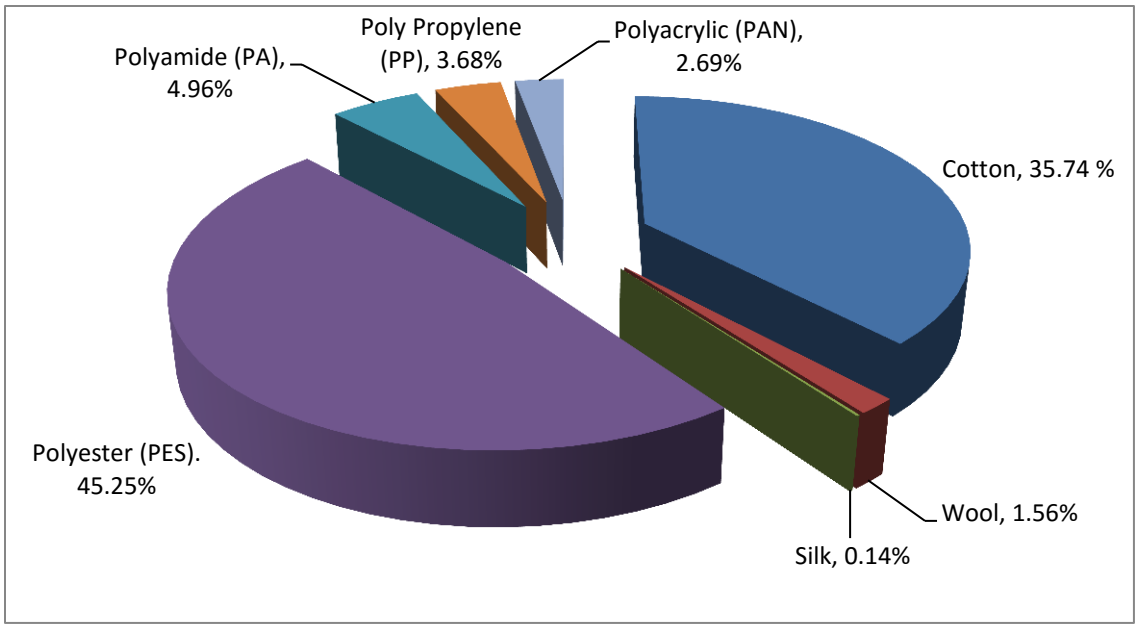

Figure 2.3. The market position of natural and man-made fibres in 2009/2010 (Kozlowski et al., 2012b).

Problems with polyester

The demand of polyester is high (Figure 2.3) because of its low cost, higher dimensional stability and wide range of applications (B. J. Collier et al., 2009). Polyesters are widely used in apparels, home furnishings or in industrial applications, alone or in a blend (B. J. Collier et al., 2009). For blending with cotton, polyester filaments need to be converted to staple form (B. J. Collier et al., 2009). A huge amount of energy is required for producing polyester fibres/filaments. For example, 95 GJ of energy is needed to produce 1 tonne of staple polyester (PET) fibre (Shen et al., 2010) (Figure 2.4).

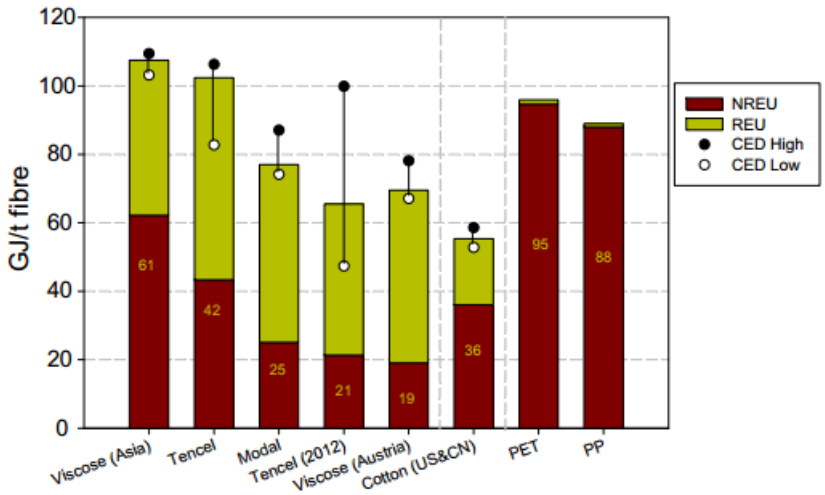

Figure 2.4. The primary energy requirement on the basis of cumulative energy demand (CED) for one tonne of staple fibre from cradle to factory gate [CED = Renewable energy use (REU) + non-renewable energy use (NREU)] (Taken from Shen et al., 2010).

Furthermore, the production of synthetic fibres generates a waste stream resulting in human safety issues (Shen et al., 2010). The production of polyester (PET) fibres produces a higher

amount of human toxicity than cotton (Table 2.3), mainly due to the air emission of polycyclic aromatic hydrocarbons (PAH) (Shen et al., 2010).

Additional concerns arise from the dyeing of blended yarn or fabric that takes two subsequent dyeing steps in union dyeing (Kaliyamoorthi & Thangavelu, 2015). These two steps of dyeing use a lot of water, energy, time and labor compared to the dyeing of non-blends. Whereas 100 % cotton, 100 % nylon or 100 % polyester yarn or fabric only take one step for being colored (Trotman, 1975). Finally, the efficient disposal of man-made fibres may cause a loss of arable lands as they take years to degrade and cause environmental pollution and toxicity (Thomas et al., 2011).

Table 2.3. An environmental assessment for producing one tonne of staple fibre from cradle to factory gate (Shen et al., 2010).

	Cotton	**PET[1]**	**PP[2]**	**Viscose**
Abiotic depletion (kg Sb eq./ t)	17	45	42	40
Ozone layer depletion (× 10^{-4} kg CFC11 eq./t)	2.0	0.7	0.7	2.8
Human toxicity (kg 1,4DB eq./t)	1,700	4,393	369	1,490
Fresh water aquatic ecotoxicity (kg 1,4DB eq./t)	17,310	58	53	160
Terrestrial ecotoxicity (kg 1,4DB eq./t)	1,568	12	12	16
Photochemical oxidant formation (kg C_2H_4 eq./t)	0.7	1.0	0.6	1.8
Acidification (kg SO_2 eq./t)	41	21	11	45
Eutrophication (kg PO_4^{3-} eq./t)	22	1.2	1.0	2.3

[1] PET= Polyethylene terephthalate
[2] PP= Polypropylene

Many researchers and scientists have now begun to rethink the creation of man-made fibre as it is difficult to dispose and degrade. Additionally, they also may contribute to global warming by releasing CO_2 during processing of petroleum-based raw materials (Sun, 2005). The problems of waste management and the current awareness for a cleaner and safer environment diverted scientists to utilize eco-friendly alternatives (natural fibres) that are easily degradable or bio-compatible (Thomas et al., 2011).

2.5 Natural fibres

Research on sustainable technology has increased due to the presence of abundantly accessible agro-waste from natural sources. Thousands of tons of agricultural crop residue, like straw, are produced worldwide and are a cheap and easily available source of lignocellulosic materials (Thomas et al., 2011). The utilization of these inexpensive bio-materials for textile application, industrial products and bio-composites, instead of synthetic fibres, could have an impact on reducing negative impacts on the environment (Thomas et al., 2011).

Various portions of the plants, such as bast, leaf, stalk, stem, seed and fruit, are the available sources of lignocellulosic fibre materials (Table 2.4). These can include cotton, flax, hemp and coir, all for producing spun yarn. Among all of the natural fibres, cotton has the largest world-wide share (35.7 %) for the production of the natural fibres (Figure 2.3).

Table 2.4. Different natural plant fibres and their sources (Collier et al., 2009; Kozlowski et al., 2012b).

Source	Natural plant fibre
Seed	Cotton, kapok
Bast	Flax, hemp, jute, kenaf, ramie
Leaf	Sisal, abaca, henequen
Fruit	Coir, African palm, luffa
Grass	Bamboo, totora
Wood	Hardwood and softwood

Cotton

Cotton, a natural fibre collected from its seed, is very popular because of its versatility and reasonable cost ($0.80 per pound) (B. J. Collier et al., 2009). Cotton has versatility in applications as it can be spun into very fine (200 Ne) to very coarse (4 Ne) yarns that are used to make fabrics that can be used in a wide range of applications (B. J. Collier et al., 2009). It is used for apparel due to its superior dyeability and comfort properties (B. J. Collier et al., 2009). The products made of cotton are also used for household items such as sheets, table cloths, draperies and upholstery fabrics (B. J. Collier et al., 2009). It is also easily blended with other fibre materials such as flax, ramie and polyester (B. J. Collier et al., 2009).

Though the product of cotton has many useful applications, cultivation and processing of cotton has negative impacts on human health and environment (Wakelyn, 2007). Cultivation of cotton fibre leads to soil erosion due to the preparation of soil and use of insecticides and pesticides (Rana et al., 2014). However, production of hemp requires fewer amounts of pesticides (Rana et al., 2014). Cotton requires a lot of water for cultivation and grows in the region with moderate rainfall. However, it is also cultivated in large quantities throughout the regions of low rainfall

relying on irrigation water (Rana et al., 2014). Shen et al. (2010) reported that for cultivation of 1 tonne of cotton fibre, 4300 m^3 and 6800 m^3 of water were used for irrigation purposes in the United States and China, respectively (Table 2.5). Bast fibres such as flax and hemp require comparatively less input for seed production (Ebskamp, 2002). Cotton cultivation also requires significant insecticide, herbicide and defoliant applications (B. J. Collier et al., 2009; Wakelyn, 2007). These chemicals can cause a negative impact on human and environmental safety (Wakelyn, 2007). The application of pesticides also has a significant impact on fresh water eco-toxicity and terrestrial eco-toxicity (Table 2.3). Moreover, the crop production of cotton can also rapidly consume the natural resources and damage the fertility of the soil (Judkins, 2008).

Table 2.5. The amount of natural water used for producing one tonne of staple fibre (Shen et al., 2010).

Type	**Fibre**	**Process water (m^3 / tonne of fibre)**	**Cooling water (m^3 / tonne of fibre)**	**Irrigation water (m^3 / tonne of fibre)**
Petrochemical fibres	PP[3]	<2	74	---
	PET[4]	<5	125	---
Man-made cellulose fibres	Viscose	11	308	---
	Modal	43	429	---
Cotton	Cotton (US and Chinese)	<5	37	5690 (4300-6860)

[3] PP= Polyolefin
[4] PET=Polyester

2.6 Bast fibres

Some commonly known bast fibres are flax, ramie, hemp, kenaf and jute (Kozlowski, Mackiewicz-Talarczyk, Muzyczek, et al., 2012). Plants of bast fibres consist of lumen at the inner layer surrounded by the cell walls (Thomas et al., 2011). In comparison with cotton, production of bast fibres require lesser amounts of water and pesticides (Ebskamp, 2002). They also have some benefits such as better strength (tenacity) and moisture regain than cotton (Nayak, Padhye, & Fergusson, 2012). Table 2.6 describes the physical and mechanical properties of different bast fibres and cotton.

Table 2.6. The physical and mechanical properties of different bast fibres and cotton (Kozlowski et al., 2012a; Nayak et al., 2012).

Fibre properties	**Cotton**	**Jute**	**Flax**	**Ramie**	**Hemp**
Physical properties					
Length of commercial fibre (mm)	15-56	750-1500	700-900	800	2500
Diameter (µm)	14-21	5-25	12-30	16-125	16-50
Density (g/cm^3)	1.52-1.56	1.44-1.50	1.48-1.50	1.51-1.55	1.48-1.49
Standard Moisture regain %	8.5	17	12	8.5	12
Mechanical properties					
Tenacity (g/den)	1.7-6.3	2.0-6.3	2.6-8.0	4.5-8.8	3.0-7.0
Extension at break %	3-12	1-2	1.5-5.0	1.5-5.0	1.5-5.0
Dyeability	Yes	Yes	Yes	Yes	Yes

However, the use of bast fibre is very limited, and only a few percent of the world fibre consumption is made from bast fibres (Ebskamp, 2002). Some of these bast fibre crops are mainly cultivated for the collection of fibres, which is not necessarily economical (Mather &

Wardman, 2015). In addition, bast fibre does not have sufficient spinning and bending properties for preparation of yarns (Mather & Wardman, 2015)

General structure for the plant of bast fibre

Bast fibres consist of several cell walls that are formed from oriented reinforcing semi-crystalline cellulose microfibrils (Thomas et al., 2011). These cellulosic microfibrils are arranged in various manners surrounded by a hemicellulose–lignin matrix (Thomas et al., 2011) (Figure 2.5).

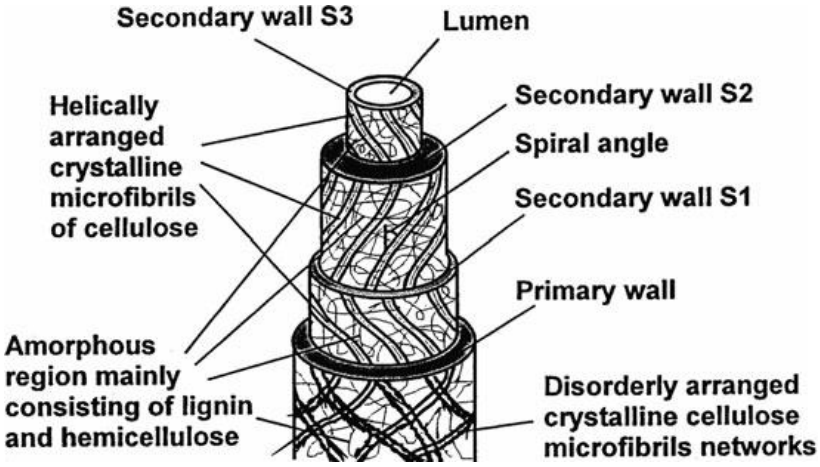

Figure 2.5. A general structure of the plant stems or stalks fibres (Thomas et al., 2011).

The cell walls are distributed in two portions, the primary cell wall and the secondary cell wall (Thomas et al., 2011). Primary cell walls are closely packed loose irregular networks of cellulose microfibrils. The secondary wall is comprised of three different segments: the outer layer, the middle layer and the inner layer (Thomas et al., 2011). The fibres present in the middle layer are responsible for its mechanical properties (Thomas et al., 2011).

2.7 Chemical composition of different fibres

Table 2.7 compares the chemical composition among seed fibre (cotton), bast fibres (jute, flax, ramie and hemp) and leaf fibres (sisal or *Agave sisalana* and abaca or *Musa textilis*). The key component of all the fibres is cellulose (Table 2.7). Cotton has the highest proportion (82.7-90 %) of cellulose content in the fibres, (Table 2.7). Cellulose, a basic component of a plant, is responsible for many physical and chemical properties of the fibre (Thomas et al., 2011). The large fraction of non-cellulosic components in the fibre negatively influences its properties, such as the adhesion between the matrix and the fibre (Kostic, Pejic and Skundric, 2008; Jonoobi et al., 2009).

Table 2.7. The chemical composition (%) of different plant fibres (adapted from Baltazar-Y-Jimenez and Sain, 2012; Nayak et al., 2012).

Fibre	Cellulose (%)	Hemicellulose (%)	Pectin (%)	Lignin (%)	Fat and Wax (%)	Wax (%)	Ash (%)
Cotton	85-90	5.7	< 1.0	----	N/A	0.6	----
Jute	61-75.5	13.6-20.4	0.2	12-13	N/A	0.5	----
Flax	71-75.2	8.6-20.6	2.3	2.2-4.8	N/A	1.7	1.1
Ramie	68-76.2	13-16.7	1.9	< 0.7	N/A	1.7	1.1
Hemp	70-75.1	< 2.0-22.4	0.9	3.5-8.0	N/A	0.8	3.5
Sisal	47.6-78	10-17.8	10	10-14	N/A	2	4.5
Abaca	56-63.7	17.5	1.0	15.1	N/A	----	1.1
Kenaf	45-57	21.5	3.0-5.0	8.0-13.0	N/A	----	----

The mechanical properties of natural fibre depend on its cell geometry upon formation of high ordered crystalline regions or low ordered amorphous regions (Thomas et al., 2011). Cellulose is a linear polymer of 1,4-β-d-glucopyranose units whereas hemicellulose is a branched polymer,

comprised of different sugar units and 1,4-β-d-glucopyranose units (Thomas et al., 2011). Lignin, a complex 3D hydrocarbon polymer, is comprised of aliphatic and aromatic components (Thomas et al., 2011). It is responsible for the strength and rigidity of the plants and hinders chemical, physical and microbial degradation (Harwood & Harwood, 2012; Thomas et al., 2011). Lignin is soluble in alkali at high temperatures whereas cellulose is resistant to strong alkali (Thomas et al., 2011). Pectin is a complex form of hetero polysaccharide and polygalacturon acid, producing flexibility and is soluble in water after a partial neutralization with alkali (Thomas et al., 2011). Waxes are comprised of different alcohols and dissolve in several acids (Thomas et al., 2011).

Scope and limitations of bast plants

The main reason for cultivating different types of bast plants is to collect fibers from the plant stems (Kolodziejczyk, Ozimek, & Kozlowska, 2012). Other portions of the plant often remain unused except for use of the seeds (Kolodziejczyk et al., 2012). Unfortunately, fibres from bast plants often lack spinning properties (Wang, Cai, & Yu, 2008). Due to this disadvantage, other potential crops need to be evaluated for their potential as fibre producing crops. Generating fibre as a bi-product following other commercial crop applications could have huge economic and environmental benefits. Based upon the large acreage of canola (*B. napus* L.) in Canada, it seems prudent to evaluate the spinning and textile properties for preparing yarn from *Brassica* species.

Tofanica et al. (2011) found that rapeseed (*B. napus*) stalk fibers contain 61.3 % cellulose, 5.2 % lignin, 4.8 % ash content and other non-cellulosic materials. They also found that canola (*B. napus*) stems/stalks contained 41 % cellulose, 21.5 % lignin and 5.8 % ash content and other

non-cellulosic materials. Similar results were reported by Hosseinpour et al. (2010) who reported that canola plant stalk fibres contained 48.5 % cellulose, 20 % lignin and 6.6 % ash content and other non-cellulosic materials. The stalk fibre composition of *Brassica* plants is similar to different types of plant fibres (Table 2.7). Therefore, a natural textile fibre extracted from a waste stream should be possible using *Brassica* plants.

2.8 *Brassica* plants

A wide range of various agriculturally important species is organized in a single genus named *Brassica*. It consists of various important oilseed, mustard and cruciferous plant species (Hayward, 2011). Gómez-Campo (1999) provided a detailed taxonomic synopsis of *Brassica* with its subgenera, section and species. *Brassica* species are divided into subgenera with 5 different sections and 27 different species (Table 2.8).

Table 2.8. The taxonomic synopsis of *Brassica* with its subgenera, section and species (Taken from Gómez-Campo, 1999).

Genera	Subgenera	Section	Species
***BRASSICA* L.**	*Brassica*	*Brassica*	• *B. oleracea L.* • *B. montana* Pourret • *B. incana* Ten. • *B. villosa* Biv. • *B. rupestris* Rafin • *B. macrocarpa* Guss • *B. insularis* Moris • *B. cretica* Lam. • *B. botteri* Vis. • *B. hilarionis* Post. • *B. carinata* Braun • *B. balearica* Pers.
		Rapa (Miller) Salmeen	• *B. rapa* L. • *B. napus* L. • *B. juncea* (L.) Czern
		Micropodium DC.	• *B. fruticulosa* Cyr. • *B. nigra* (L.) Koch • *B. cossoniana* Boiss. and Reuter • *B. spinescens* Pomel • *B. maurorum* Durieu • *B. procumbens* (Poiret) O.E. Schulz • *B. cadmea O.E. Schulz* • *B. desertii* Danin and Hedge
		Brassicoides Boiss. *SInapistrum* Willkomm	• *B. deflexa* Boiss • *B. barrelieri* (L.) Janka • *B. oxyrrhina* Coss. • *B. tournefortii* Gouan

Gómez-Campo (1999) also referred to six species, mentioned in "Triangle of U", as "crop brassicas" (Figure 2.6). The rest of the *Brassica* species other than the species mentioned in "Triangle of U" have limited agricultural importance except *B. tournefortii*. *Brassica tournefortii*

is cultivated in India as a source of oil (Gómez-Campo, 1999). According to U (as cited in Gómez-Campo, 1999), three diploid *Brassica* cultivars *B. rapa* (n=10), *B. oleracea* (n=9) and *B. nigra* (n=8) combine to produce three other different amphidiploid species, *B. carinata* (n=17), *B. juncea* (n=18) and *B. napus* (n=19) (Figure: 2.8). Hybridization took place naturally thousands of years ago, but they can also be crossed artificially (Daun, 2011). A, B and C represent the genomes of *B. rapa*, *B. nigra* and *B. oleracea*, respectively (Ostergaard & King, 2008). As a consequence, AB, AC and BC express the resulting amphidiploid haploid genomes of *B. juncea*, *B. napus* and *B. carinata* (Ostergaard & King, 2008).

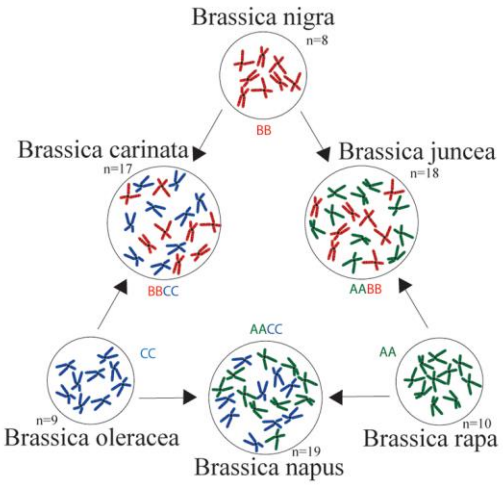

Figure 2.6. The triangle of U depicting the genetic relationship between Brassica species (U, 1935), between the six species, three genomes are shared (A, B and C) and arrows indicate the direction of genome transfer. Image obtained from: http://en.wikipedia.org/wiki/Triangle_of_U.

2.9 Uses of *Brassica* plants with common names

The primary crop brassicas are comprised of oilseed species of *B. napus*, *B. rapa* (syn. *B. campestris*), *B. carinata* and *B. juncea*, cruciferous vegetable species of *B. oleraceae* and herb

species of *B. nigra* (Hayward, 2011). Table 2.9 provides a brief summary of different important *Brassica* cultivars and their common names with the portion of the plants used.

Table 2.9. The end-use of different *Brassica* plants (Taken from Diederichsen and McVetty, 2011).

Species	**Karyotype**	**Usage type**	**Common name**
***B. rapa* L. (syn. *B. campestris* L.)**	n=10, A	Seed oil, forage, green manure	Oilseed turnip, turnip rape, field mustard, canola
		Seed oil, sometimes vegetable	Yellow sarson
		Seed oil, vegetable, forage	Brown sarson, toria (Hindi), Indian rape
		Leaves as vegetable	Mizuna (Japanese)
		Leaves and inflorescence as Vegetable	Cime di rapa (Italian)
		Leaves and petioles as vegetable	Pak-choi
		Leaves and leafy rosettes as vegetable	Pe-tsai, Chinese cabbage
***B. nigra* (L.) Koch**	n=8, B	Seed oil, and condiment, vegetable and forage	Black mustard
***B. oleracea* L.**	n=9, C	Leafy head as vegetable	Cabbage, savoy cabbage
		Stem as vegetable	Kohlrabi
		Leaves as vegetable	Palm kale,curled chicken kale, Russian kale
		Budding inflorescence as vegetable	Cauliflower, broccoli, Chinese kale

Species	Karyotype	Usage type	Common name
***B. juncea* (L.) Czern.**	n=18, AB	seed oil, forage	Indian mustard, oriental mustard
		Leaf vegetable	Leaf mustard
		Root vegetable	Tuberous-rooted mustard
***B. napus* L.**	n=19, AC	Seed oil, forage, green manure	Rapeseed, canola
		leaf vegetable	Nabicol, rape-kale
		Root vegetable and forage	Swede, rutabaga
***B. carinata* A. Braun**	n=17, BC	Seed oil and vegetable use	Abyssinian mustard
***B.fruticulosa* Cyr.**	n=8, F	Wild, weed	Mediterranean Cabbage
***B. tournefortii* Gouan**	n=10, T	Cultivated, seed oil use	Asian mustard

2.10 Rapeseed and canola

All the species, such as *B. juncea, B. nigra* and *B. carinata,* in which seeds are used as spice, are summarized in the general term "mustard," whereas the *Brassica* species used for seed oil are referred to as "rapeseed" (Daun, 2011) or oilseed rape". In Europe, the oilseed species *B. napus* and *B. rapa* are known as "rapeseed" (Diederichsen & McVetty, 2011). Some *Brassica* oilseed species, such as *B. carinata, B. juncea, B. napus,* and *B. rapa*, produce seed oil containing moderate to high in erucic acid content and moderate to high protein content in seed meal (Diederichsen & McVetty, 2011). The ranges of erucic acid content and glucosinolate content in different *Brassica* species are mentioned in Table 2.10 (Diederichsen & McVetty, 2011; Velasco, Goffman, & Becker, 1998).

In 1974, seeds of *B. napus* with low content of erucic acid and low content of glucosinolate were created in Canada and then named "canola" in 1978 (Stefansson & Kondra, 1975). Since then, seeds of *B. juncea* and *B. rapa* also achieved the seed quality of canola (Diederichsen & McVetty, 2011). Now all three species *(B. rapa, B. napus* and *B. juncea)* with low content of erucic acid (< 2 %) and low content of glycosinolates (< 30 µmol g^{-1}) are known as "canola" (Daun, 2011).

Table 2.10. The range of erucic acid and glucosinolate in different *Brassica* oilseed species (Diederichsen and McVetty, 2011; Velasco et al., 1998).

Species	**Erucic Acid Content (%)**	**Glucosinolate content (µmol g^{-1})**
B. carinata	29.6 – 51.0	>75 – 150
B. juncea	15.5 – 52.3	> 100 – 200
B. napus	5.6 – 58.1	> 20 – 200
B. rapa	6.5 – 61.5	> 20 – 200

2.11 Production and uses of rapeseed

The production of rapeseed in Canada between 2006 and 2009 was 10.76 million tons, which was 19.87 % of the total production in the world (Daun, 2011) (Table 2.11). In 2015, the production of canola reached about 17230 thousands of tonnes that are about 20 % increases in production from 2011 (Table 2.12). These plants are mainly cultivated for seed, which is used as a source of vegetable oil, protein meal and spice (Rakow, 2011). The oils are the source of edible oils, fine chemicals, efficient fuels and lubricants (Hayward, 2011), whereas the uses of meal range from fertilizer to high-quality animal feed or functional protein. The seed is also used as a spice or condiment (Daun, 2011). Huge portions of the plant stems remain unused and degrade into the soil prior to the next cultivation.

Table 2.11. Five-year mean production of rapeseed in thousands of tons (modified from Daun, 2011).

Country or region	1996-2000 (thousands of tons)	2001-2005 (thousands of tons)	2005-2009 (thousands of tons)
China	9711.7	11907.6	9210.2
Japan	0.9	0.6	1.1
Australia	1478.2	1466.7	857.5
W. Europe (France, Germany, UK, Austria and other)	8664.6	9832.2	12081.3
Scandinavia (Denmark, Sweden, Finland and Norway)	546.4	592.5	802.6
Eastern Europe (Czech Republic, Slovakia, Hungary, Poland, Russian Fed., Ukraine)	2328.7	3201.8	7334.9
Europe Total	11539.7	13626.5	20218.8
Indian Subcontinent (India, Bangladesh, Pakistan)	6324.6	5837.2	7542.6
Canada	7020.4	6716.7	10761.1
Mexico	4.2	1.0	5.5
United States	574.7	723.4	658.4
North America total	7599.3	7441.1	11425.0
South America	76.0	99.4	300.0
Worlds Total	48327.9	54069.7	54155.5

However, researchers in the field of textiles and agriculture sciences have been searching for a natural fibre from the waste stream for centuries. Due to the large production in Canada, *B. napus* is a potential candidate for producing fibre from plant material remaining in the field

following the harvest of seed. To utilize these stems, they need to be collected and the non-cellulosic materials must remove in order to obtain cellulosic fibres.

Table 2.12. Production of Canola in Canada in thousands of tonnes (Canola Council of Canada, 2016)

Year	Ontario	Manitoba	Saskatchewan	Alberta	British Columbia	Total Canada
2012	61.2	2,100.1	6,486.4	5,097.2	82.8	13,868.5
2013	49.9	3,025.50	9,178.40	6,168.90	88.7	18,551.00
2014	31.3	2,510.60	7,971.90	5,796.90	71.9	16,410.10
2015	34	2,857.60	8,799.70	5,443.10	70.8	17,231.20

2.12 Fibre extraction

Fibres from the plant stems can be extracted by retting (Thomas et al., 2011). Retting is the process of isolating the fibers from the woody core of plant materials by exposing them to moisture, water or chemicals (Thomas et al., 2011). The Textile Institute textile terms and definitions committee (1991) defined the retting of flax as "the subjection of crop or de-seeded straws to chemical or biological treatment to make the fibre bundles more easily separable from the woody part of the stem". The retting process can be accomplished in different ways, such as biological retting, mechanical retting, chemical retting or enzymatic retting (Thomas et al., 2011).

Biological retting or natural retting can occur by dew retting or water retting. Dew retting is the process whereby the crops mown and retained in the field for the separation of the fibres from the straw (Amel *et al*., 2013; Goodman, Ennos & Booth, 2002). Dew retting is limited because the process should be controlled in particular geographical area where the moisture and

temperature are suitable for retting (Kozlowski & Mackiewicz-Talarczyk, 2012). Additionally, the retting process must cease at an appropriate time to prevent over-retting or under-retting (Akin *et al.*, 2000; Akin *et al.*, 2001; Thomas *et al.*, 2011). With respect to water retting, bundles of the mown or cut crops immersed in warm water or canals for several weeks for water retting by anaerobic bacteria (Kozlowski & Mackiewicz-Talarczyk, 2012; Kyung & Obendorf, 2006; Murali & Mohana, 2007). Following completion of the retting, fibres are manually separated from the plants. Water retting produces the best quality fibre, but causes a significant amount of environmentally unacceptable organic fermentation waste (Thomas *et al*., 2011).

Mechanical separation or decortication is the process by which fibres are mechanically separated from the stem (Thomas et al., 2011). Different types of decortication machines are used for fibre separation such as hammer mills (Munder & Fürll, 2004), planetary ball mill (Khan *et al*., 2009), cutter heads (Gratton & Chen, 2004), and crushing rollers (Khan *et al.*, 2013). On the basis of compressing forces, fibres are detached from the plants in a crushing roller (Khan et al., 2013), while both cutting and impact forces are utilized for separating the fibres with cutterheads (Gratton & Chen, 2004). A series of different machines can also be utilized for the decortication process; for example, flax fibres can be extracted over the course of four individual modules: 1) 9- roller calendar, 2) top shaker, 3) scutching wheel, 4) 5-roller calendar in which stems can be passed through over a range of time frames (Akin, Dodd, & Foulk, 2005).

During chemical separation, the straw is immersed in a tank containing a solution of chemicals such as acetic acid, sodium carbonate, or sodium hydroxide (Akin, Dodd, & Foulk, 2005; Amel *et al.,* 2013; Kyung & Obendorf, 2006). Chemical retting has the potential to damage the fibre if the chemicals are not treated in the proper manner. However, chemical retting can also produce a better quality fibre with the proper utilization of chemicals. Enzymatic retting can also be an

efficient alternative for producing an excellent quality fibre (Akin *et al*., 2000, Akin *et al.*, 2001; Akin, Dodd, & Foulk, 2005; Kyung & Obendorf, 2006). In this case, enzymes such as pectinase, catalase, protease can be used to accelerate retting (Konczewich & Kozlowski, 2012).

2.13 Mechanism of retting

The cell wall of a plant consisting of bast fibres has a mixture of different components such as cellulose, hemicellulose, pectin, lignin, fat and waxes with varying ratios (Table 2.7). Cellulose, the main component for textile fibre, is responsible for the textile properties and is surrounded by a complex structure of hemicellulose and lignin in a matrix of varying composition around the woody core of the plant (Figure 2.5). Furthermore, three different types of pectin (water soluble pectin, water insoluble or chelator soluble pectin and Protopectin) are usually seen in plant stems (Walter, 1991). During the retting process, water-soluble pectin is dissolved by micro-organisms, and water insoluble pectin remains within the fibre bundle (Kozlowski & Mackiewicz-Talarczyk, 2012). Chelator soluble pectin and protopectin dissolve in a calcium-chelating agent, such as ethylene diamine tetra acetic acid (EDTA), or an alkali solution, respectively (Walter, 1991). During the retting process of jute, fibre bundles known as reeds are loosened by dissolving or removing the gummy substances like pectin and are separated from the plant core (Figure 2.7) (Roy & Lutfar, 2012a). The removal of the gummy materials is mainly done by microbial activity in the water bath (Batra, 1998; Konczewicz & Kozlowski, 2012). According to Konczewicz and Kozlowski (2012), this removal accelerated by different types of microbes such as *Bacillus amylobacter, B. felsineus, Granulobacter pectinovorum*, *Clostridium felsineum* and *B. comesii rossi*.

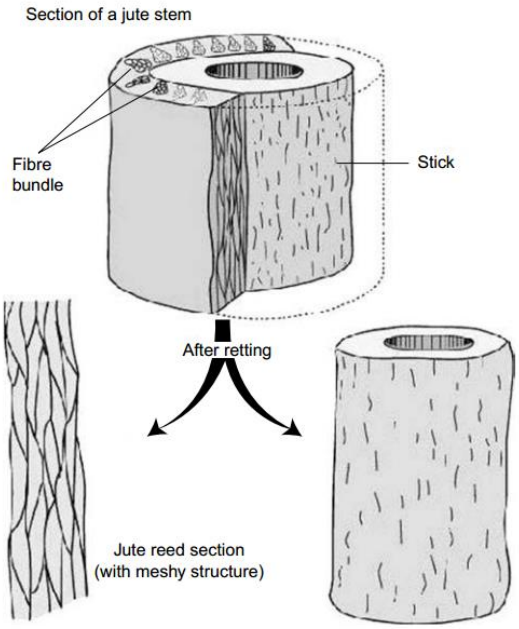

Figure 2.7. The isolation of jute reed from the stick following retting (From Roy & Lutfar, 2012a).

The retting process involves the production of pectic enzymes, which are mainly caused by the micro-organisms (listed above) present in the water. When the plant straw is immersed in water, a pectinolytic bacterial community develops naturally by dissolving the soluble compounds like sugar and nitrogenous substances (Donaghy, Levett, & Haylock, 1990). These bacteria penetrate into the stem and destroy the pectin, causing the separation of fibres from the stem (Donaghy et al., 1990). Aerobic bacteria from the *Bacillus* genus initiate the process, which is then controlled by anaerobic bacteria of the *Clostridium* genus (Donaghy et al., 1990). This bacterial growth reaches a peak and then declines with the reduction of residual oxygen in the water, which prevents and delays the growth of anaerobic *C. felsineum* bacteria (Donaghy et al., 1990). This anaerobic bacterium shows a strong pectinolytic and retting activity (Donaghy et al., 1990).

The objective of this research was to evaluate the suitability of *Brassica* fibres for apparel and textile end uses. In order to study the suitability of *Brassica* fibres, the fibres must be extracted from the plant stems through retting. The extracted fibres are then evaluated for different spinning properties, such as softness, flexibility and individual fibre entity, and textile properties, such as mechanical properties, thermal properties and dyeability. Furthermore, the effect of *Brassica* cultivars on different textile and spinning properties also evaluated. These two objectives will discuss in the following two chapters.

Chapter 3 Characterization of Textile and Spinning Propeties of *Brassica* Fibres

Abstract

Brassica napus L., which is commonly known as oilseed rape and canola, is the largest source of edible oil in Canada. The remaining plant material, such as the stem, remains unused for any immediate application and is returned to the soil for decomposition. An investigation has been conducted to extract, characterize and modify the fibre materials from *B. napus* stems for textile and apparel applications. In order to find the optimum retting conditions for retting time, four different retting parameters were evaluated including, retting temperature, material liquor ratio, water exchange and the reuse of retted water. We have discovered that the virgin-retted fibres from *Brassica* plants exhibit many of the required textile properties including dye absorbency, strength, and thermal behaviour. The thermal decomposition temperature of *Brassica* fibres resulted at 240 ˚C, which is similar to cotton (245 ˚C). The breaking load of virgin-retted *B. napus* fibres was 36 cN. This is higher than the breaking load of cotton fibres (6 cN), and lower than the polyester fibres (47.8 cN). However, the virgin-retted fibres do not exhibit the required spinning (yarn transformation) properties (softness, flexibility and individual fibre entity). In order to modify the *Brassica* fibres for spinnability, three treatment methods were applied: 1) alkali, acid and softener treatment; 2) pectinase enzyme treatment; and 3) enhanced enzyme treatment. According to Method 5 of the American Association of Textile Chemists and Colorists (AATCC), *Brassica* fibers obtained from treatments 2 and 3 showed similar spinning properties to each other and to cotton, and demonstrated superior spinning properties to *Brassica* fibres obtained from treatment one. Treatments two and three are therefore suitable for producing *Brassica* fibres fit for textile and apparel applications. The current research suggests that

producing fibers from canola stubble and stems could be an additional income source for canola growers.

3.1 Introduction

Textile fibre, the raw material for any kind of textile or apparel product, must possess basic requirements for fulfilling textile performance such as durability, comfort, aesthetic appeal, maintenance, and health and safety protection (Hatch, 1993). Textile fibres are broadly divided into two main groups: natural fibres and man-made fibres (Hatch, 1993). In 2009/2010, the relative market positions of natural fibres and manmade fibres were 37.4 % and 62.6 %, respectively (Kozlowski, Mackiewicz-Talarczyk, Muzyczek, et al., 2012). Among all of the natural and man-made fibres, cotton and polyester have the largest market share, with 35.7 % and 45.3 %, respectively (Kozlowski, Mackiewicz-Talarczyk, Muzyczek, et al., 2012).

The demand for polyester has been increasing steadily because of its low production cost, higher dimensional stability and wide range of applications from clothing apparel and home furnishings to industrial products (B. J. Collier et al., 2009). However, the production of polyester fibres consumes a huge amount of energy and generates a significant amount of polycyclic aromatic hydrocarbon (PAH), which is toxic to humans (B. J. Collier et al., 2009). Additionally, polyester takes years to degrade and creates environmental pollution and toxicity during disposal (Thomas et al., 2011). Cotton, a natural fibre, is widely used for apparel and non-apparel applications due to its availability, spinning properties and comfort properties (B. J. Collier et al., 2009; Dochia & Sirghie, 2012). However, cotton is cultivated mainly for fibres, and cotton cultivation requires large amounts of water, pesticides, herbicides and defoliants (B. J. Collier et al., 2009). These chemicals have significant health effects on agricultural workers and the environment as well as on fresh water eco-toxicity and terrestrial eco-toxicity (Shen et al., 2010). Other natural fibres (flax and jute) have very limited apparel uses because they lack spinning properties (Piotrowski & Carus, 2010).

To this point, no textile fibre has been obtained from the waste stream of agricultural crop residue, although millions of tons of straw/stalks are produced worldwide, and may be available sources for inexpensive textile fibre. Replacing natural and synthetic fibres with these inexpensive bio-materials for textile and apparel applications, industrial products and bio-composites should have a significant positive impact on ecosystem (Thomas et al., 2011). One potential crop is canola or oilseed rape (*Brassica napus* L.), which is mainly cultivated for its seed and the resulting oil and seed meal. Following seed harvest, the remaining plant material (stems, leaves and pods) is left in the field. Previously, it was discovered that *B. napus* stalk fibre contained 61.3 % cellulose, which is similar to that of flax (64.1 %), ramie (68.0 - 76.2 %), hemp (67.0 %) and kenaf (45-57 %) but less than cotton (85-90 %) (Baltazar-Y-Jimenez & Sain, 2012; Nayak et al., 2012; Tofanica et al., 2011). Since natural cellulosic textile fibres are mainly composed of cellulose, it was hypothesized that *Brassica* fibres might have potential as textile fibres. This hypothesis hinges upon the ability to extract fibre from the unused plant stems of *B. napus*.

The first objectives of the current study was to investigate the extraction of Brassica fibres from *B. napus* plant stems using water retting technique, and evaluate different textile properties (thermal decomposition temperature, fibre breaking load, and dyeability) and spinning properties. Secondly, we attempted to optimize the retting time using different retting parameters, including the material liquor ratio, water exchange and the reuse of retting water.

3.2 Materials and methods

3.21 Plant samples

Mature *B. napus* plants were grown in the Crop Technology Centre (CTC) greenhouse, at the University of Manitoba in Winnipeg, Canada. The cultivar Reston (*B. napus*) was grown and harvested when the siliques turned yellow in color. Sunshine® Professional Growing Mix (Sun Gro Horticulture, Canada) was used for growing the plants. The plants were grown in a growth chamber (temperature: day: 22 ˚C, night: 17 ˚C; light cycle: 16 hours light, 8 hours dark) with sufficient air flow and water. At two-leaf stage, plants were transferred to larger plastic pots (14.5 cm × 15 cm) using the same soilless mix used for the flats. The pots remained in the greenhouse at CTC located at the University of Manitoba. The atmospheric conditions of greenhouse (temperature: high 25 ˚C, low 22 ˚C, relative humidity 40-50 %, light cycle: 16 hours light, 8 hours dark) were controlled by Argus Control Systems (Argus Control Systems Ltd., Surrey, BC Canada). Following harvesting, plant samples were transferred to the textile laboratory located in the Department of Textile Sciences at the University of Manitoba. As the fibres are highly susceptible to moisture, all the samples were conditioned at 21 ± 1 ˚C and 65 ± 5 % relative humidity for 48 hours prior to retting, according to the American Society for Testing and Materials (ASTM) standard D1776 (ASTM International, 2008).

3.22 Retting and extraction of fibres

Water retting was carried out at 20 °C, 40 °C and 50 °C in beakers. Twenty-two samples of *Brassica* stems were cut 10 cm (Figure 3.1) in length and immersed into water. The weight of the samples ranged between 4.09 and 8.79 g, depending on the diameter of the stem. Water was added daily to maintain a constant water level in the retting bath. For retting at 20 °C, the retting

bath was kept in a cupboard in the laboratory, while retting was conducted in a hot water bath for retting at 40 °C and 50 °C. Samples were checked daily in order to find the end point of retting. There are no standards for ensuring the end point of retting. In these experiments, the end point of retting was detected when the outer layer of the stem could easily be isolated by gently peeling or rubbing the surface of the plant stems by hand.

Figure 3.1. Cut *Brassica napus* stems undergoing the water retting process.

In order to isolate fibres from retted stems, fibres were peeled manually from the surface of the plant stems by hand. The extracted fibers were then washed, dried at room temperature and kept in a conditioning room for 48 hours. The temperature and the relative humidity of the conditioning room were 21 ± 1 ˚C and 65 ± 5 %, respectively.

3.23 Evaluation of retted waste water

The retted water may have toxic properties that could potentially be harmful to the health and safety of workers or end users. For this reason, analysis of the presence of metal content in the retted waste water was carried out by using argon plasma of Inductively Coupled Plasma-Optical

Emission Spectrometer (ICP-OES) (Model: Varian 725-ES, Australia) located in the Department of Chemistry, University of Manitoba. The metals, cadmium (Cd), chromium (Cr), copper (Cu), mercury (Hg), and lead (Pb), were analyzed with the settings of wavelength in 214.439 nm, 267.716 nm, 327.395 nm, 194.164 nm and 220.353 nm, respectively.

The Nuclear Magnetic Resonance (NMR) analysis for the molecular structure of the chemical substances present in the retted water was conducted using a Bruker Avance III 500 (Bruker, Karlsruhe, Germany) located in the Department of Chemistry, University of Manitoba. The experiment was performed with a 256 scan 1D proton spectrum with pre-saturation of the water signal. Ten % D_2O was added for a lock signal. The sample temperature was 25 ˚C. The pre-saturation time, acquisition time, and a total re-cycle delay time was 3 seconds, 3.28 seconds and 6.28 seconds, respectively.

3.24 Analysis of fibre decomposition temperature

The decomposing point analyzer, used for analyzing thermal decomposition temperature, was comprised of a heating stage controller with link pad (Model: T95 HS, Make: January, 2011), an imaging station (Linkam Scientific Instrument, UK) and a monitor (Brand: Dynax, Model: DX-22L 150A11, Make: August, 2011). The settings of the machine for the rate of increase of temperature and the runtime for the decomposition point of the enzyme-treated *Brassica* samples were adjusted in 11 different ramps as shown in Table 3.1. Single fibres were placed in a glass slide, and the temperature was raised according to the setting of temperature increase rate, maximum temperature and runtime with each ramp. The final decomposition temperature was determined by the visual analysis of the color change on the sample displayed on the monitor.

Fibre decomposition temperature was measured for determining the ability to withstand dyeing or other high temperature manufacturing and laundering procedures.

Table 3.1. Manual settings of 11 different ramps for controlling temperature for the analysis of decomposition point of the enzyme-treated *Brassica* fibres at the University of Manitoba in 2014.

Ramp	Temperature increase rate (^{0}C / min)	Maximum temperature (^{0}C)	Runtime at maximum temperature (hour :min :sec)
01	50	190	00:00:00
02	10	190	00:10:00
03	10	210	00:10:00
04	10	225	00:10:00
05	10	240	00:10:00
06	10	250	00:10:00
07	10	260	00:10:00
08	10	270	00:10:00
09	10	280	00:10:00
10	10	290	00:10:00
11	10	300	00:10:00

3.25 Fibre strength measurement

Fibre breaking load was measured using an Instron Strength Tester (Model: 5965, Massachusetts, USA). The machine was mounted with a 500 N load cell, and fibre samples were

evaluated with an upper crosshead speed of 2 mm/min following the principle of constant rate of extension (Collier & Epps, 1998). The sample length (distance between the end points of the two clamps in Instron 5965 Strength Tester) for measuring fibre strength was considered as one (1) cm. The fibre strength is measured for determining the ability to withstand the spinning procedures and stress related to the end uses.

3.26 Dyeing of the samples

To determine the dyeability of the *Brassica* fibres for commercial textile application, the virgin-retted *Brassica* fibres from Reston cultivar were dyed using an exhaustion dyeing technique, as the samples were fibres and loose in form (Collier, 1970). During exhaustion dyeing, the materials were immersed in a dye solution (Collier, 1970). The dye solution was prepared using a mixture of Reactive blue 4 dyestuff (12 % on the weight of fibre), sodium chloride (80 g/l) and sodium carbonate (20 g/l) solution in a stainless steel lever lock canister at room temperature with a material to liquor (dye solution) ratio of 1:300. The enzyme-treated *Brassica* fibre samples were then added to the canister and placed in the preheated bath of the Atlas Launder-ometer® (SDL, Model M228AA) and firmly attached to the laundering machine shaft using an adapter plate. The bath was preheated to 60 ^{0}C. The Launder-ometer was run for 5 minutes to preheat the fibre samples present inside the canister. The fibre samples were then run at 60 ^{0}C for 60 minutes for the completion of dyeing process (Trotman, 1975). After completion of dyeing, samples were rinsed with running warm water and then treated with water at 70 ^{0}C for 10 minutes. The samples were then dried at room temperature for 48 hours.

3.27 Evaluation of dyeing performance (colourfastness to laundering)

The dyeing performance (Colorfastness to laundering) of the virgin-retted and dyed *Brassica* was evaluated according to test no. 1A of American Association of Textile Chemists and Colorists (AATCC) standard: 61-2009 (AATCC, 2010a). It was conducted in the Atlas Launder-ometer® (SDL, Model M228AA) containing rotating closed canisters. The fibre samples were treated with ten (10) steel balls and one (1) strip of multifibre test fabric in a canister containing 200 ml solution of 0.37 % AATCC 1993 WOB (without optical brightener and without phosphate) standard detergent, at 40 ^{0}C for 45 minutes. The change of color and staining of color were evaluated by using AATCC grey scale (Collier & Epps, 1999).

3.28 Spinning properties of the *Brassica* fibres

For determining the spinnability, physical attributes such as individual fibre entity, bending and surface nature for both virgin-retted fibres and enzyme-treated fibres were evaluated by hand. Bending properties were evaluated onto two subcategories - stiff or supple, and similarly surface attributes also divided into two subdivisions – harsh and soft, using the guideline from AATCC evaluation procedure 5 (AATCC, 2010c).

3.29 Surface modification of fibre

Surface modification was carried out using three different treatment procedures; 1) alkali treatment, acid treatment and softening; 2) enzyme treatment; and 3) enhanced enzyme treatment. All three different treatments were carried out in the Atlas Launder-ometer® (SDL, Model M228AA) for improving fibre spinnability.

In the alkali treatment, acid treatment and softening, fibres were treated in three different steps. First, the virgin-retted *Brassica* samples were treated in a 200 ml solution containing NaOH (5 %) and a wetting agent (0.5 %), at 60 °C for 60 minutes. The samples were then rinsed with warm tap water for 10 minutes. In the second step, alkali-treated samples were processed in 200 ml of 4 % acetic acid solution at 60 °C for 30 minutes, and rinsed with warm tap water for 10 minutes. In the final step, it was treated with 200 ml Tubingal 4748 softener solution (3 %) at 40 ^{0}C with pH 5.5 (controlled by acetic acid) for 30 minutes.

In the enzyme treatment, the virgin-retted *Brassica* fibres were treated with 200 ml solution of 4 % pectinase enzyme from *Aspergillus aculeatus* at 40 ^{0}C with pH 5.5 (controlled by acetic acid) for 90 minutes.

Two different steps were involved in the enhanced-enzyme treatment of virgin-retted *Brassica* fibres. Firstly, the virgin-retted *Brassica* samples were processed with a 200 ml solution of 0.2 % AATCC 1993 WOB (without optical brightener and without phosphate) standard detergent and 0.01% Tx-100 (4-octylphenol polyethoxylate) at 60 ^{0}C for 60 minutes and rinsed with warm tap water for 10 minutes. Secondly, fibre samples were treated with a 200 ml solution of pectinase enzyme from *Aspergillus aculeatus* at 40 ^{0}C with pH 5.5 (controlled by acetic acid) for 150 minutes.

All of the treated fibres from the three different treatments were dried at room temperature for 48 hours in the textile laboratory, and conditioned at 21 ± 1 ˚C and 65 ± 5 % relative humidity for 48 hours according to the standard conditioning procedure of ASTM D1776. The samples were then evaluated for different textile and spinning properties as described above.

3.3 Results and discussions

3.31 Effect of temperature and material to liquor ratio on retting time

The rate of retting was much faster at 40 °C than at room temperature (20 °C). For the *B. napus* (composite of cultivars) plants, the retting was completed in 4 days at 40 °C, while the retting time doubled (8 days) at 20 °C (Table 3.2). To confirm the optimum retting temperature, a set of experiments was conducted at 20 °C, 40 °C and 50 °C with equal amounts (700 ml) of water in each retting bath using the *B. napus* cultivar, Reston. Again, the rate of retting was the fastest (3 days) at 40 °C and the rate was slower below and above this temperature (Table 3.2).

Table 3.2. The effect of retting temperature on retting time on *Brassica napus* stems at the University of Manitoba in 2014.

Retting temperature (°C)	**Volume of water (ml)**	**Retting completion time (day)**	**Species**	**Cultivar**
20	600	8.0	*B. napus*	Mixed
40	600	4.0	*B. napus*	Mixed
20	700	3.5	*B. napus*	Reston
40	700	3.0	*B. napus*	Reston
50	700	4.5	*B. napus*	Reston

The temperature fluctuation of hot water bath quoted by the manufacturer was ±2 °C; therefore, it is reasonable to state that the optimum retting temperature for *B. napus* plant varies between 38 °C and 42 °C. Previous studies have shown that 37.5 °C was the fastest and 45 °C was the slowest retting temperature for hemp fibres (Magnusson & Svennerstedt, 2007). For flax, the optimum retting temperature was between 30 °C and 36 °C (Magnusson & Svennerstedt, 2007). However, for kenaf, the optimum retting temperature was 32 °C, because of maximum microbial

growth, and the rate of retting was slower as less microbial growth occurs below and above 32 °C (Yu & Yu, 2007).

The current research also found that the material to liquor ratio has no effect on retting time. Retting was complete in nine (9) days for both 1:10 and 1:100 material to liquor ratios (Table 3.3). Meanwhile, for jute plants, it was found that at 30 ^{0}C, the shortest (10 days) and the longest (18 days) water retting completion time was obtained for plant material to liquor ratios of 1:20 and 1:5, respectively (Haque, Asaduzzaman, Akhter, Hossain, & Ahmed, 2001). These differences of the material to liquor ratios on retting time may have arisen due to the different retting temperature and plant material as the current study was carried out at 40 °C, whereas the research conducted by Haque et al. (2001) was conducted at 30 °C.

Table 3.3. The effect of material to liquor ratio on retting time using *Brassica napus* stems at the University of Manitoba in 2014.

Retting temperature (°C)	**Ratio of sample to retting water**	**Addition/ change of water**	**Cultivar**	**Retting completion time (day)**
40	1:10	No	Reston	9
40	1:100	No	Reston	9

3.32 Effect of water change on retting time

When examining the impact of water change on retting time, retting time increased by 25 % at 20 °C and 150 % at 40 °C when retting water was changed daily in comparison with the

unchanged retting water (Table 3.4). It is known that the retting of the plant stems is accelerated due to the growth of pectic enzymes by micro-organisms (Donaghy et al., 1990). Daily water change most likely increased retting time because microbial growth needed to resume following each water change. Thus, a probable reason for increasing retting time due to daily water exchange can be illustrated using Figure 3.2. When the water changes, the grown micro-organisms also drain with the water and need to increase their populations when fresh water is added, leading to an increase in time to complete the retting process.

Table 3.4. The effect of water change frequency on retting time of *Brassica napus* stems at the University of Manitoba in 2014.

Retting temperature	**Frequency of water change**	**Retting completion time (days)**
20 °C	Daily[5]	10
20 °C	No change	8
40 °C	Daily[5]	22
40 °C	No change	9

[5] Fresh water was added, old water was replaced.

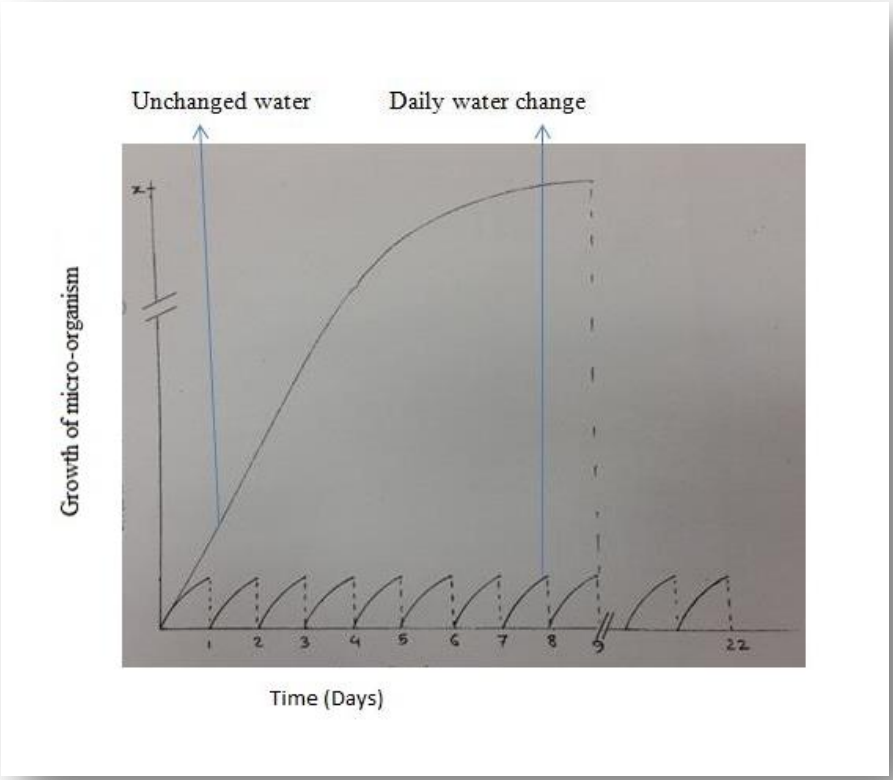

Figure 3.2. A probable growth of micro-organism in unchanged and changed water for *Brassica* plants during retting.

The results indicate that daily water change increased retting time because microbial growth needed to resume following each water change. Based on this result, a logical question to ask was whether there is any effect on retting time if the previous retting water is reused. To determine this, subsequent experiments were conducted using both fresh and previously used retted water in separate retting procedures, keeping all other retting parameters the same.

3.33 Effect of reused water on retting time

At 40 °C, the retting completion time was much faster when previously used retting water was re-used for subsequent retting (4 days), compared to a retting completion time of 24 days in fresh distilled water (Table 3.5).

Table 3.5. The effect of reused water on retting time of *Brassica napus* stems at the University of Manitoba in 2014.

Retting temperature (°C)	**Type of retting water**	**Material to liquor ratio**	**Retting completion time (day)**
40	Distilled water	1:100	24
40	Retting water	1:100	4

A similar reduction in retting time was reported by Di Candilo et al. (2010) when the retting water was inoculated with aerobic bacteria from the *Bacillus* genus and anaerobic bacteria from the *Clostridium* genus. They also found that the retting time was shorter (4 days) for plant samples retted with bacterial inoculated water than the non-inoculated water (6 days).

It appears that the retting process requires more time when fresh water is added due to a decrease in the microorganism populations. In contrast, previously retted water contains microorganism populations produced during the previous retting. Since the current study is the first of its kind that used previously retted water for subsequent retting, further research is needed to further clarify the accelerated rate of retting when reusing retting water.

3.34 Evaluation of retted water

The immediate concern for using the retted water for subsequent retting was whether or not this water is harmful to human health and the environment as the retted water produced an unpleasant odour. Attempts were made to find the odorous and harmful chemical components (metal presence) in this retted water using Inductively Coupled Plasma (ICP) (Table 3.6) and Nuclear Magnetic Resonance (NMR) (Fig. 6) analysis.

Table 3.6. The presence of heavy metals (ppm) in retted water of *Brassica* plant stems compared with drinking water, soil, and clothing standards.

Source	**Standard**	**Amount of metal content (ppm)**				
		Cd	**Cr**	**Cu**	**Hg**	**Pb**
Retted water	--	0.041	0.040	0.198	1.464	0.083
Drinking Water[6]	WHO Standard	0.003	0.05	--	0.001	0.01
	Canadian Standard	0.005	0.05	--	0.001	0.01
Allowable limit in soil of Manitoba[7]		2.5	115	113	11.9	126
Baby	Oeko-Tex	0.1	1.0	25	0.02	0.2
Adult (direct contact with skin)	Standard For clothing[8]	0.1	2.0	50	0.02	1.0

[6] (Mohapatra & Mitchell, 2003)
[7] (Canadian Council of Ministers of the Environment, 2010)
[8] (OEKO TEX Association, 2013)

The metal content present in retted water is lower than the allowable limit in the soil of Manitoba (Canadian Council of Ministers of the Environment, 2010). However, the level found in the retted water exceeds the limit in drinking water for both the World Health Organization and Canadian standards (Mohapatra & Mitchell, 2003). The amount of heavy metals present in the retted water is within the acceptable range using the Oeko-Tex Standard (OEKO TEX Association, 2013) for clothing for both children and adults, except for the mercury content (Hg). The amount of mercury (Hg) present in the retted water was 1.464 ppm, compared with the Oeko-Tex Standard for clothing of 0.02 ppm. A sharp peak of acid can be seen in the NMR spectrum in the region between 4.5 and 5.0 ppm (Figure 3.3). Kozlowski, Mackiewicz-Talarczyk, and Allam (2012) stated that the pectin and hemicellulose in the plant materials decompose and form volatile butyric acid (80 %) in water retting under anaerobic conditions. This butyric acid is mainly responsible for the odour resulting from the retted water and dried fibres.

3.35 Thermal properties

The decomposition temperature of the virgin-retted Reston fibres and cotton was determined by the visual change of color at 240 ˚C and 245 ˚C respectively. Figure 3.3(a) illustrates the condition of Reston fibre at the beginning of the experiment at 60.7 ˚C, whereas Figure 3.3(b) indicates a change in the Reston fibres at 240 ˚C. The decomposition temperature of cotton results at 245 ˚C.

(a) 

(b) 

Figure 3.3. The decomposition temperature analysis of virgin-retted *Brassica* napus (cv. Reston) fibre [(a) fibre at 60.7 °C, (b) fibres at 240 °C] at the University of Manitoba in 2014.

3.36 Mechanical properties

The average breaking load of virgin-retted *B. napus* fibres was 36 cN with a standard deviation of 11.6 cN (Table 3.7). A lower breaking load (6 cN) was reported for cotton fibres (Hsieh, 2007) in comparison with *B. napus* fibres. However, polyester fibre has shown a higher breaking load (47.8 cN) compared to *B. napus* fibres (Rahman & East, 2006).

3.37 Dyeing and evaluation of colorfastness of *Brassica napus* fibres

As *B. napus* fibre composed of cellulose and cellulosic fibres can be dyed with reactive dye, virgin-retted fibres (Figure 3.4(a) of Reston dyed with reactive blue dye resulted blue colored fibres (Figure 3.4(b)).

Table 3.7. The breaking load of virgin-retted *Brassica napus* fibre, upland cotton fibres and polyester fibres.

Sample no	*B. napus* fibre			Upland cotton[9]	Polyester[10]
	Breaking load	Average breaking load	Standard deviation (±)		
01	43.0 cN				
02	44.0 cN				
03	47.0 cN	36.0 cN	11.6 cN	6.0 cN	47.8 cN
04	21.0 cN				
05	27.0 cN				

[9] (Hsieh, 2007)
[10] (Rahman & East, 2006)

a)  b) 

Figure 3.4. Dyeing of virgin-retted *Brassica napus* fibres [(a) fibres before dyeing; (b) fibres after dyeing] at the University of Manitoba in 2014.

Figure 3.5 shows the AATCC standard grey scale and the multifiber fabric (both new and washed) for the evaluation of colorfastness to staining of the dyed *Brassica* fibres. Grey scale

rating for both color change and staining for the dyed *B. napus* fibres is mentioned in Table 3.8. Using the grey scale rating for staining, multi-fibre fabric demonstrated a slight stain of the blue color during washing in cotton (4-5) and viscose (4-5) (Figure 3.5). The fastness rating of *B. napus* fibres for shade change (4-5) is much better than acceptable minimum range for 100 % cotton denim fabric (3-4), knit swimwear fabric (4), and woven slipcover fabrics (4) (Table 3.8).

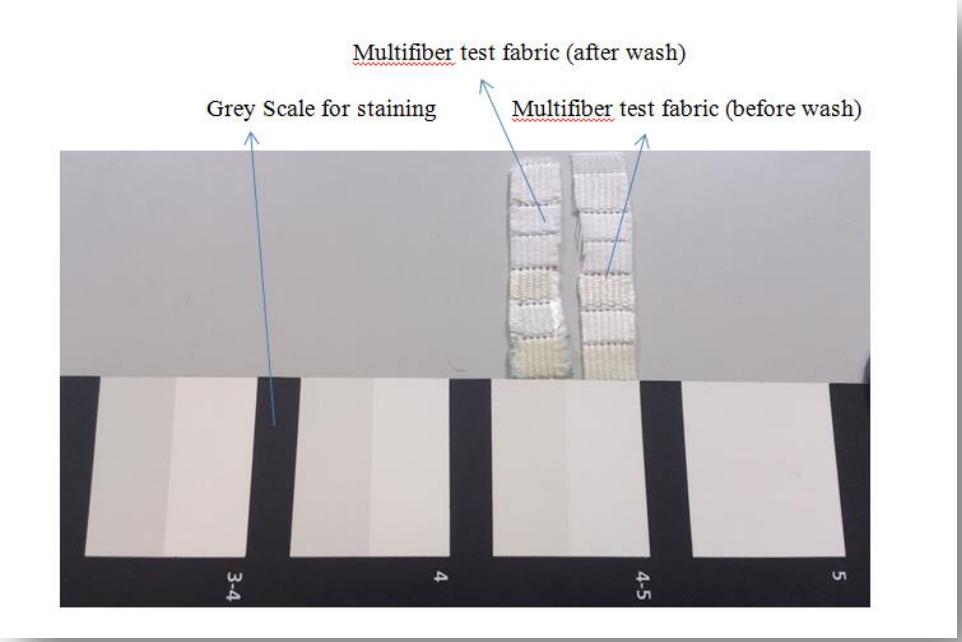

Figure 3.5. The assessment of accelerated colorfastness to laundering (AATCC, 2010a) for staining of retted/dyed *Brassica napus* fibres at the University of Manitoba in 2014.

Table 3.8. The evaluation of colorfastness of the dyed/retted *Brassica napus* fibre (cv. Reston) and minimum industrial requirements for different fabrics.

		Minimum industrial requirements		
	Virgin-retted *Brassica* fibres	**100 % Cotton denim Fabrics[11]**	**Knit swimwear fabrics[12]**	**Woven slipcover fabrics[13]**
Grey Scale rating for shade change				
Rating	4-5	3-4	4	4
	Experimental	Minimum industrial requirements		
Multi-fibre	**Virgin-retted *Brassica* fibres**	**100 % Cotton denim fabrics**	**Knit swimwear fabrics**	**Woven slipcover fabrics**
Grey scale rating for staining				
Acetate	5	3	3	3
Cotton	4-5			
Polyamide	5			
Silk	5			
Viscose	4-5			
Wool	5			

[11] ASTM D 6554-14
[12] ASTM D 3996-14
[13] ASTM D 4113-02

3.38 Spinning properties (stiffness, softness, and individual fibre entity) of virgin-retted fibres

Virgin-retted *B. napus* fibres were stiff and not individualized (Figure 3.6). The fibres are glued together, probably due to the inefficient removal of pectin content during retting process. With this type of fibre, it would be difficult to produce yarn through a spinning machine such as a carding machine. The surface of the fibres was also found to be harsh (Figure 3.6).

Figure 3.6. Virgin water-retted *Brassica napus* fibres at the University of Manitoba in 2014.

3.39 Surface modification of virgin-retted *Brassica napus* fibres

As mentioned in Section 3.38, virgin-retted *B. napus* fibres are stiff, harsh and very difficult to separate as fibres are glued to each other. For preliminary studies to achieve the spinning properties, *B. napus* fibres were treated using three different methods: 1) alkali, acid and softener treatment, 2) pectinase enzyme treatment, and 3) enhanced enzyme treatment with pectinase (scoured + enzyme). Fibres obtained from different treatments were then visually evaluated and compared with each other (Table 3.9).

Table 3.9. A comparative study of three different treatments on *Brassica napus* fibres at the University of Manitoba in 2014.

Fibre properties	**Untreated retted *Brassica* fibres**	**Alkali, acid and softener treated fibre**	**Enzyme-treated fibre**	**Enhanced-enzyme treated fibre**
Fibre length	Not determined	Short	Long	Medium
Strength	Not determined	High	Medium	Low
Single fibre entity	Not individual	Not individual	Individual	Individual
Bending properties	Stiff	Stiff	Supple	Supple
Surface properties	Harsh	Harsh	Soft	Soft

Alkali, acid and softener treated samples (Figure 3.7(a)) were not individualized and seemed stiff after drying of the samples. The opposite is seen for both enzyme- and enhanced-enzyme-treated samples. Both of the samples were more individualized and flexible than alkali, acid and softener treated samples. Besides, fibre length of the enhanced-enzyme-treated samples (Figure 3.7(b)) was higher than alkali, acid and softener-treated samples and lower than enzyme-treated samples (Figure 3.7(c)). In addition, alkali, acid and softener-treated fibre samples demonstrated better strength than enzyme- and enhanced-enzyme-treated samples (Table 3.9). Comparing the enzyme and enhanced-enzyme-treated fibres; the former treatment method produced fibres with better spinning behaviour in terms of fibre length and strength.

Figure 3.7 (a) Alkali, acid and softener-treated, (b) enhanced enzyme-treated, and (c) enzyme-treated *Brassica napus* fibres at the University of Manitoba in 2014.

3.4 Conclusions

Fibres were successfully extracted from the *B. napus* stems using water retting at varying conditions. To determine the optimum retting conditions, *B. napus* stems were retted in different retting conditions of varying temperature, material liquor ratio, water change, and reuse of previous retted water. It was found that ~40 °C is the optimum retting temperature for extracting fibres from *B. napus* stems. Among other retting variables, material to liquor ratio did not have any impact on the completion of retting time while the use of retted water in subsequent retting accelerated the retting rate significantly. However, retted water could have a negative impact as it contains larger amounts of mercury and does not conform to the OEKO-Tex Standard for both adult and children clothing. The thermal decomposition temperature and breaking load of virgin-retted *B. napus* fibres was 240 ˚C and 36 cN, respectively.

It appears that the virgin-retted fibre obtained from the retting process is not sufficient to undergo the spinning processing steps as the fibres are not flexible and are attached to neighbouring fibres. Surface modification is therefore necessary for improving the spinning properties of virgin-retted fibres. It can be seen that both enzyme and enhanced-enzyme-treated fibres exhibit better spinning properties than alkali, acid and softener-treated fibres. Further, when comparing the enzyme and enhanced-enzyme-treated fibres; the former treatment method

produced fibres with better spinning properties in terms of fibre length and strength. During the research work it also seemed important to find the effect of different cultivars on the fibre properties.

Chapter 4 Analysis of different *Brassica* cultivars for fibre properties in textile and apparel applications

Abstract

Brassica plant consists of different specices: *Brassica napus* L., *B. juncea* L. and *B. rapa* L. Modern breeding programs developed a lot of cultivars incorporating different properties such as herbicide tolerance and pesticide tolerance in these species. Seeds from twenty different *Brassica* cultivars consisting of three different species, *Brassica napus* L., *B. juncea* L. and *B. rapa* L., were collected, planted and then harvested upon reaching physiological maturity. The stems were retted in water in order to obtain fibre. Retting time, fibre yield and spinning properties of virgin-retted fibres were evaluated. The virgin-retted fibre samples were then treated with pectinase enzyme, and different spinning properties (stiffness, softness, individual fibre entity) and textile properties (fibre decomposition temperature, tenacity and dye absorbency) of enzyme-treated samples were evaluated and compared among the twenty different cultivars. Cultivars were reached their end point of retting at 5 to 11 days when treated at room temperature. The thermal decomposition temperature of enzyme-treated fibres ranged from 220 ºC to 260 ºC among the cultivars. The tenacity of the enzyme-treated *Brassica* fibres also ranged between 0.012 N/tex and 0.085 N/tex. The current research suggests that the cultivars of *Brassica* plants had an impact on different spinning and textile properties of the fibre.

4.1 Introduction

Plant breeders develop new cultivars on the basis of the yield, quality and productivity, as well as agronomic characteristics such as the time required for maturity and resistance to lodging and disease, among many other traits (Allard, 1999; Salmon-Minotte & Franck, 2005). Cultivars in all types of crops (seed or fibre) can vary for all of the traits listed above. For example, the properties of cotton fibres vary with cotton type (Pima, American Upland, DCH-32, Suvin and many more), cultivar, geographical location and cotton color (Cotton Incorporated, 2015; Gordon, 2007). Furthermore, the fibres obtained from the same ball of the cotton plant may vary in length, diameter, and even in strength (Farag & Elmogahzy, 2009). Farag and Elmogahzy (2009) found that Pima cotton fibre has more strength (4.14 ± 0.59 N/tex) than the Upland cotton (3.19 ± 0.87 N/tex). Salmon-Minotte and Franck (2005) found the rate of growth, flowering and maturation of the fibre and seed productivity for ten different cultivars and conveyed that the productivity depends on the flax cultivar. For example, the Ariane cultivar has 'very good' productivity compared to the Nynke cultivar, which has 'medium' productivity (Salmon-Minotte & Franck, 2005).

In another study, Cromack (1998) found that the hemp cultivars and seeding rate affect the hemp fibre yield (t ha^{-1}) and hemp fibre yield (%). The highest yield of hemp fibres (3.48 t ha^{-1}) was obtained from the cultivar 'Komopoliti', with a seeding rate of 400 seeds/m^2 among 5 different cultivars named Fedora 19, Felina 34, Uniko B, Futura 77 and Komopoliti, whereas the lowest yield (1.34 t ha^{-1}) was obtained from Felina 34 with a seeding rate of 200 seeds/m^2. These studies clearly demonstrate that plant cultivar has an impact on the fibre yield and quality. It is

hypothesized that fibres from different cultivars of *Brassica* plants may also result in different fibre yield (%), textile properties and spinning properties.

Sevenhuysen and Rahman (2015) patented the major textile (strength, moisture regain, decomposition temperature, moisture regain and dye absorbency) and spinning (fibre length and length variation, strength, and flexibility) properties of the fibres from the plant sources obtained from the *Brassica* genus. In the patent, *Brassica* fibres were obtained by water retting. The inventors also mentioned that the textile fibres produced from the *Brassica* plants originated from *B. napus* species, but they didn't mentioned the name of the cultivar used in retting process. In the present study, seeds from canola and rapeseed of *Brassica* plants were planted and harvested for obtaining the fibres. Canola is a modified *Brassica* type, used as edible oil, that contains low erucic acid (< 2 %) and low glycosinolate (< 30 µmol g^{-1}) content (Daun, 2011). The cultivars of low erucic acid and low glucosinolate were developed in Canada in 1970s and renamed "canola" in 1978 (McVetty & Duncan, 2015). Rapeseed is another type of *Brassica* that contains high erucic acid (> 40 %) and is cultivated for producing industrial oil.

In the present study, twenty known *Brassica* cultivars (Table 4.1) of three different species *(B. napus, B. juncea and B. rapa)* were investigated to find the impact of species and cultivar on retting time, yield (%), fibre spinning properties (surface smoothness, bending property), and textile properties (fibre decomposition temperature, tenacity and dye ability).

4.2 Materials and methods

4.21 Seeds of different *Brassica* cultivars

Seeds of the twenty different cultivars were collected from the *Brassica* breeding program in the Department of Plant Science at the University of Manitoba. The cultivars are comprised of three different plant species of *B. napus*, *B. juncea* and *B. rapa* (Table 4.1).

Agronomic information such as plant height and plant lodging on the twenty cultivars of *Brassica* plants is shown in Table 4.2. Lodging is a term used to describe the displacement of vertical positioning of plant stems as a result of various external factors such as wind, rain, disease and so forth (Podolska, 2011). Lodging is expressed numerically, ranging from 1 to 5, where 1 indicates no lodging of the plant (almost all plants erect) and 5 represents complete lodging of the plants (all plants down) (McVetty, Duncan, Fernando, Li, & Zelmer, 2012). The erucic acid and glucosinolate content of different *Brassica* seeds are also found in Table 4.2.

Table 4.1. Twenty different *Brassica* cultivars and their species collected from *Brassica* Breeding Program in the Department of Plant Science at the University of Manitoba in 2014.

Type of cultivar	Cultivar	Species
Rapeseed	Hero	*B. napus*
Rapeseed	Reston	*B. napus*
Rapeseed	Mercury	*B. napus*
Rapeseed	Venus	*B. napus*
Rapeseed	Neptune	*B. napus*
Rapeseed	Red River 1861	*B. napus*
Rapeseed	HYHEAR 1	*B. napus*
Canola	Global	*B. napus*
Canola	Westar	*B. napus*
Canola	O2R276	*B. napus*
Canola	Sentry	*B. napus*
Canola	UM no. 2407	*B. napus*
Canola	UM no. 2257	*B. napus*
Canola	Defender	*B. napus*
Canola	Stellar	*B. napus*
Canola	Apollo	*B. napus*
Canola	Polo	*B. napus*
Canola	Topas	*B. napus*
Mustard	Arid	*B. juncea*
Rapeseed	Reward	*B. rapa*

Table 4.2. Registration and agronomic information for twenty *Brassica* cultivars used in the retting experiments in 2014 at the University of Manitoba.

Cultivar	Developer	Plant height (cm)	Erucic acid content (%)	Glucosinolate Content (μmol g^{-1})	Lodging (1-5)	Reference
Hero	University of Manitoba	107	50.2	15	2.2	(McVetty, Rimmer, Scarth, & van den Berg, 1996; Scarth, McVetty, Rimmer, & Stefansson, 1991)
Reston	University of Manitoba	103	40-45	<30	1.8	(McVetty & Scarth, 2012)
Mercury	University of Manitoba	109	54.1	12.9	2.4	(Scarth, McVetty, & Rimmer, 1995)
Venus	University of Manitoba	106	~53	11.3	2.9	(McVetty, Scarth, Rimmer, & Van Den Berg, 1996)
Neptune	University of Manitoba	108	~53.5	8.3	3.1	(McVetty, Rimmer, et al., 1996)
Red River	University of Manitoba	102	52.6	9.3	2.4	(McVetty et al., 2012)

Cultivar	Developer	Plant height (cm)	Erucic acid content (%)	Glucosinolate Content (µmol g^{-1})	Lodging (1-5)	Reference
1861						
HYHEAR 1	(Collaboration) DL Seeds/ Norddeutsche Pflanzenzucht (NPZ) Lembke and University of Manitoba	107	52.2	11.7	2.3	(McVetty et al., 2014)
Global	Svalof AB, Svalov, Sweden	127	0.2	15	1.0	(Food Production and Inspection, 1985)
Westar	Agriculture and Agrifood Canada	102	0.2	15	2.0	(Klassen, Downey, & Capcara, 1987)
O2R276	University of Manitoba	N/A[14]	N/A	N/A	N/A	---
Sentry	University of Manitoba	102	0.1	10.1	2.3	(Rimmer, Scarth, & McVetty, 1998)
UM no. 2407	U of M Accession acquired from Semundo, Germany	109	0.7	17.0	1.0	In house data from 2012
UM no. 2257	U of M Accession acquired from Svalof AB, Sweden	107	2.8	20.3	1.3	In house data from 2012
Defender	Svalof Weibull AB, Sweden	108	0.1	8.1	2.2	(Proven Seed, 1994)

[14] Not available

Cultivar	Developer	Plant height (cm)	Erucic acid content (%)	Glucosinolate Content (μmol g^{-1})	Lodging (1-5)	Reference
Stellar	University of Manitoba	116	0.1	8	2.9	(Scarth, McVetty, Rimmer, & Stefansson, 1988)
Apollo	University of Manitoba	115	0.1	7	2.8	(Scarth, Rimmer, & McVetty, 1995)
Polo	Mycogen Canada Inc	114	0.2	6.4	2.3	In house data from 1994
Topas	Svalof AB, Svalof, Sweden	116	0.1	9.7	1.1	(Food Production and Inspection, 1987)
Arid	Saskatchewan Wheat Pool (SWP)	N/A	N/A	N/A	N/A	(Canola Council of Canada, 2014)
Reward	University of Manitoba	85	0.1	16.7	2.7	(Scarth, Rimmer, & McVetty, 1992)

4.22 Plant production in greenhouse

Twenty-four seeds of each cultivar were planted at 1 cm depth in flats during the spring of 2014. Sunshine® Professional Growing Mix (Sun Gro Horticulture, Canada) was used in flats. The plants were grown in a growth chamber (temperature: day: 22 ˚C, night: 17 ˚C; light cycle: 16 hours light, 8 hours dark) and were watered daily. Thirteen days after seeding, at the two-leaf stage, each plant was transferred to the larger plastic growers pot (14.5 cm × 15 cm) using the same soilless mix used for the flats. The pots were kept into the greenhouse of Crop Technology Centre (CTC) located at the University of Manitoba. The atmospheric condition of greenhouse (temperature: high 25 ˚C, low 22 ˚C, relative humidity 40-50 %, light cycle: 16 hours light, 8 hours dark) was controlled by Argus Control System (Argus Control System Ltd., Surrey, BC Canada) and the plants were watered daily. Plant Prod 20-20-20 fertilizer were applied twice (once during transplantation and again after 40 days of seeding) at a concentration of 15 gm / 3.78 L. Seed pods developed and became physiologically mature 94 days after seeding, and were harvested by hand. Physiological maturity was visually determined by analyzing the 60 % of color change of the seeds on the main raceme (Canola Council of Canada, 2015). At the point, the stem of each plant was cut and was labeled.

4.23 Retting methodology

The harvested plant stems were then transferred to the textile laboratory of the Department of Textile Sciences at University of Manitoba. To avoid any external influences such as variation of moisture content, all plant samples were conditioned for 48 hours prior to retting according to ASTM D1776 (ASTM International, 2008). Retting was carried out using water at 20 °C in beakers. Twenty-two stems from each cultivar were cut into pieces 10.1 cm in length and

immersed into water (800 ml) using dead weight. Water was added daily to maintain the water level (800 ml) in the retting bath. Samples were checked daily in order to find the end point of retting.

In order to extract fibres from retting, fibres were peeled manually from the surface of the plant stems, when they reached at the end point of retting. There are no standards for ensuring the end point of retting. In this research, the end point of retting was detected when the outer layer of the stem could be easily isolated by gently peeling or rubbing the surface of the plant stems by hand. The extracted fibers were then washed, dried at room temperature and kept in the conditioning room for 48 hours at 21 ± 1 ˚C and 65 ± 5 % relative humidity. Fibre yield (%) of the extracted fibres was calculated using the formula:

$$\text{Fibre yield (\%)} = \frac{\text{Weight of the conditioned fibres after extraction}}{\text{Weight of conditioned plant stems before retting}} \times 100\ \%$$

4.24 Surface modification of *Brassica* fibres

A surface modification was carried out using an enzyme treatment procedure described by Sevenhuysen and Rahman (2015). In short, the fibres were treated with a solution of 4 % Pectinase from *Aspergillus aculeatus* in acidic condition (pH 5.5) at 40 ^{0}C for 90 minutes. The pH was adjusted using Acetic acid and measured by pH meter (Hanna HI 98127 waterproof pH and temperature meter). The treatments were carried out in an Atlas Launder-ometer® at 40 ± 2 rpm located at Department of Textile Sciences in University of Manitoba. The samples were washed with cold water, dried at room temperature in the laboratory for 48 hours and conditioned at 21 °C and 65 % relative humidity according to the standard procedure of ASTM D1776 (ASTM International, 2008).

4.25 Physical/spinning properties of the *Brassica* fibres

Physical attributes such as individual fibre entity, bending properties (stiff / supple) and surface properties (harsh / soft), individual fibre entity of both retted and enzyme-treated samples were evaluated by hand according to the AATCC evaluation procedure 5 (AATCC, 2010c). Bending properties were evaluated into two subcategories - stiff or supple. Surface attributes were also divided into two subdivisions – harsh or soft. A standard sample of cotton and flax was used to develop a method for ranking for the treated samples. The samples were collected from the lab manual written by Hatch (1993). Cotton fibres are considered as soft or supple whereas flax fibres represented as a standard of stiff or harsh fibres.

4.26 Fibre decomposition point analysis

The decomposing point analyzer, was comprised of a heating stage controller with link pad (Model: T95 HS), an imaging station (Linkam Scientific Instrument, UK) and a monitor (Brand: Dynax, Model: DX- 22L 150A11), located in the Department of Textile Sciences at University of Manitoba. The settings of the machine for the rate of increase of temperature and the runtime for the decomposition point of the enzyme-treated *Brassica* samples is adjusted in eleven different ramps according to Table 4.3 starting from ramp 1 to 11. Single fibres were placed in a glass slide, and the temperature was raised according to the setting such as ramp, temperature increase rate, maximum temperature and runtime. The final decomposition temperature was determined by the visual analysis of the color change on the sample displayed on the monitor.

Table 4.3. Manual settings of eleven different ramps for controlling temperature for the analysis of decomposition point of the enzyme-treated *Brassica* fibres at the University of Manitoba in 2014.

Ramp	Temperature increase rate (^{0}C/min)	Maximum temperature (^{0}C)	Runtime at maximum temperature (hour:min:sec)
01	50	190	00:00:00
02	10	190	00:10:00
03	10	210	00:10:00
04	10	225	00:10:00
05	10	240	00:10:00
06	10	250	00:10:00
07	10	260	00:10:00
08	10	270	00:10:00
09	10	280	00:10:00
10	10	290	00:10:00
11	10	300	00:10:00

4.27 Fibre strength (tenacity) measurement

Fibre breaking load was measured using an Instron Strength Tester (Model: 5965, Norwood, Massachusetts, USA) located in the Department of Textile Sciences at the University of Manitoba. The machine was mounted with a 500 N load cell and fibre samples were evaluated with an upper crosshead speed of 2 mm/min following the principle of constant rate of extension (Collier & Epps, 1998). The sample length (distance between the end points of the two clamps in Instron 5965 Strength Tester) for measuring fibre strength was considered as one (1) cm.

The diameter of the breaking point was measured by the Bioquant Image Analyzer (BIOQUANT Image Analysis Corporation, USA). Three readings of the diameter were taken on each side of

the breaking point of the fibre that was used to measure the breaking point in Instron 5965 Strength Tester. The mean of these six values indicated the final average diameter of the sample. Tex is a measuring unit of the fibre or yarn count indicating the fibre weight in gm per km of yarn. The count of the *Brassica* fibre was measured by using the formula (Booth, 1968):

$$\text{Tex} = \text{fiber Density} \times \frac{\pi d^2}{4} \times 10^5$$

Where, *d* = fibre diameter was calculated in centimeters and density was assumed as similar to cotton = 1.50 gm/cm^3. The exact density value for *Brassica* fibre is not available.

Finally, tenacity ($\text{Tenacity} = \frac{\text{Breaking Load}}{\text{Tex}}$) was calculated by using the breaking load and fibre count obtained from the *Brassica* fibres (Booth, 1975).

4.28 Dyeing of the enzyme-treated *Brassica* fibres

The enzyme-treated *Brassica* fibre samples were dyed using an exhaustion dyeing technique, as the samples were fibres and loose in form (Collier, 1970). During exhaustion dyeing, the materials are immersed in dye solution (Collier, 1970). The dye solution was prepared using a mixture of Reactive blue 4 dyestuff (12 % on the weight of fibre), sodium chloride (80 g/l) and sodium carbonate (20 g/l) solution in a stainless steel lever lock canister at room temperature with a material liquor (dye solution) ratio of 1:1000. The enzyme-treated *Brassica* fibre samples were then added to the canister and placed in the preheated bath of the Atlas Launder-ometer® and firmly attached to the laundering machine shaft using an adapter plate. The bath was preheated to 60 ^{0}C. The Launder-ometer was run for 5 minutes to preheat the fibre samples present inside the canister. The fibre samples were then run at 60 ^{0}C for 60 minutes for the completion of dyeing process (Trotman, 1975). After completion of dyeing, samples were rinsed

with running warm water and then treated with water at 70 ^{0}C for 10 minutes. The samples were then dried at room temperature.

The leftover solutions following dyeing of the fibre samples were evaluated by the Hunterlab Labscan XE spectrophotometer (Make, Location) by using D65 illuminant at 10^{0} observer angle at the Department of Textile Sciences, University of Manitoba. The L, a, and b value of the solutions were measured using easy match® QC spectrophotometer software. Scale "L" ranges from 0 to 100, where 0 represents black and 100 represents perfect white. Scale "b" represents negative infinity of blue to positive infinity of yellow color. Similarly, scale "a" represents negative infinity of green to positive infinity of red color (B. J. Collier & Epps, 1999). The higher the (–ve) b hue, the darker the dye solution, the lower the exhaustion of dyes in the fibres.

4.3 Results and Discussion

4.31 Physiological maturity of *Brassica* cultivars from seeding to harvesting

Maturity for the twenty *Brassica* cultivars ranged from 94 to 102 days (Table 4.4). The earliest physiological maturity of the *Brassica* plants was obtained within 94 days for Hero, Apollo, Polo, and Topas cultivars (Table 4.4). The longest maturity (102 days) was required for the cultivar Global (*B. napus*).

Table 4.4. Time require for maturity for twenty *Brassica* cultivars grown in Crop Technology Centre (CTC) at the University of Manitoba in 2014.

Cultivar	Species	Maturity (days)	Cultivar	Species	Maturity (days)
Hero	*B. napus*	94	**Sentry**	*B. napus*	98
Reston	*B. napus*	95	**UM no. 2407**	*B. napus*	98
Mercury	*B. napus*	95	**UM no. 2257**	*B. napus*	98
Venus	*B. napus*	95	**Defender**	*B. napus*	98
Neptune	*B. napus*	95	**Stellar**	*B. napus*	94
Red River 1861	*B. napus*	95	**Apollo**	*B. napus*	94
HYHEAR 1	*B. napus*	95	**Polo**	*B. napus*	94
Global	*B. napus*	102	**Topas**	*B. napus*	94
Westar	*B. napus*	98	**Arid**	*B. juncea*	95
O2R276	*B. napus*	98	**Reward**	*B. rapa*	98

4.32 Retting Time

The retting time of the eighteen different cultivars of *B. napus* ranged between nine to twelve days, whereas *B. juncea* stems required nine days of retting (Table 4.5). The retting time of the *B. rapa* (Reward) fibres was much quicker (5 days) than cultivars of the other two *Brassica* species.

Table 4.5. Retting time for twenty *Brassica* cultivars at room temperature in the textile laboratory at the University of Manitoba in 2014.

Cultivar	Retting time (d)	Cultivar	Retting time (d)
Hero	11	**Sentry**	10
Reston	09	**UM no. 2407**	09
Mercury	09	**UM no. 2257**	09
Venus	09	**Defender**	10
Neptune	10	**Stellar**	09
Red River 1861	09	**Apollo**	11
HYHEAR 1	09	**Polo**	09
Global	12	**Topas**	11
Westar	10	**Arid**	09
O2R276	12	**Reward**	05

Amel et al. (2013) reported that water retting of *Hibiscus cannabinus* L. (kenaf) required a total of 24 days (Amel, Paridah, Sudin, Anwar, & Hussein, 2013), whereas water retting of vakka [*Roystonea regia (Oreodoxa regia* Kunth*)*] fibre needed 15 days to extract fibre (Rao & Rao, 2007). Furthermore, different retting time was reported for different cultivars. For example, the retting time for hemp cultivars ranges between 7 to 14 days at room temperature (Horne, 2012),

and for different flax cultivars, the retting time was 3 to 7 days (Salmon-Minotte & Franck, 2005).

4.33 Fibre yield

The fibre yield (%) varied between 6.23 % and 13.82 % (Table 4.6). Red River 1861, HYHEAR 1, Stellar, Apollo and Global had fibre yields > 12.0 %. Defender (8.70 %), Arid (6.23 %) and Reward (8.73 %) produced the least amount of fibre (Table 4.6). Variation in fibre yield (19.3 % - 27.4 %) was also reported for hemp cultivars (Horne, 2012).

Table 4.6. Fibre yield for retting of twenty different *Brassica* cultivars in the textile laboratory at University of Manitoba in 2014.

Type	Cultivar	Unretted stalk weight (g)	Extracted fibre weight (g)	Fibre yield (%)	Average fibre yield (±) Standard deviation
Rapeseed	**Hero**	6.84	0.73	10.71	
Rapeseed	**Reston**	5.47	0.59	10.84	
Rapeseed	**Mercury**	4.65	0.42	9.08	
Rapeseed	**Venus**	5.14	0.56	10.80	
Rapeseed	**Neptune**	4.09	0.43	10.61	**10.90 ± 1.57**
Rapeseed	**Red River 1861**	5.62	0.78	13.82	
Rapeseed	**HYHEAR 1**	7.08	0.90	12.66	
Canola	**Global**	6.17	0.83	13.50	
Canola	**Westar**	5.64	0.59	10.41	
Canola	**O2R276**	6.20	0.69	11.08	
Canola	**Sentry**	7.26	0.84	11.61	
Canola	**UM No. 2407**	5.02	0.54	10.74	
Canola	**UM No. 2257**	6.68	0.78	11.64	**10.86 ± 1.81**
Canola	**Defender**	6.47	0.56	8.70	
Canola	**Stellar**	5.50	0.70	12.64	
Canola	**Apollo**	6.19	0.75	12.05	
Canola	**Polo**	6.65	0.74	11.14	
Canola	**Topas**	8.79	0.94	10.63	
Canola	**Arid**	4.70	0.29	6.23	
Rapeseed	**Reward**	5.44	0.48	8.73	
Mean		**5.98**	**0.66**	**10.88**	

4.34 Spinning properties of virgin-retted fibres

To determine the spinnability of the *Brassica* fibres for spun yarn preparation, different spinning properties were evaluated and compared with cotton according to the procedures mentioned in section 4.25. The data indicates that the virgin-retted *Brassica* fibres from all cultivars were stiff and not individualized, and the majority of them were harsh except Defender (Table 4.7).

Table 4.7. Spinning properties for virgin-retted fibres from twenty different *Brassica* cultivars in the textile laboratory at the University of Manitoba in 2014.

Cultivar	**Spinning properties**[15]			**Spinning properties**	
	Bending properties	**Surface properties**	**Name of the cultivar**	**Bending properties**	**Surface properties**
Hero	Stiff	Harsh	**Sentry**	Stiff	Harsh
Reston	Stiff	Harsh	**UM No. 2407**	Stiff	Harsh
Mercury	Stiff	Harsh	**UM No. 2257**	Stiff	Harsh
Venus	Stiff	Harsh	**Defender**	Stiff	Soft
Neptune	Stiff	Harsh	**Stellar**	Stiff	Harsh
Red River 1861	Stiff	Harsh	**Apollo**	Stiff	Harsh
HYHEAR 1	Stiff	Harsh	**Polo**	Stiff	Harsh
Global	Stiff	Harsh	**Topas**	Stiff	Harsh
Westar	Stiff	Harsh	**Arid**	Stiff	Harsh
O2R276	Stiff	Harsh	**Reward**	Stiff	Harsh

[15] The fibres were evaluated using the guideline of AATCC evaluation procedure 5 (AATCC, 2010c)

Although cotton fibres are attached with each other, the fibres are easily separated using beating and carding actions in spinning (Lawrence, 2007), whereas the *Brassica* fibres were difficult to individualize from the fibre bundles. Since the virgin-retted *Brassica* fibres were stiff, attached to each other and very difficult to individualize, the fibres were not suitable for spinning. To impart the spinning properties, a surface modification seemed necessary.

4.35 Spinning properties of enzyme-treated *Brassica* fibres

A visual evaluation of the bending and surface attribute of the enzyme-treated fibres demonstrated a considerable improvement in comparison with the virgin-retted fibres (Table 4.8). Following enzyme treatment, fibre from all cultivars became more supple and soft than the virgin-retted fibres (Table 4.8). The fibre surface of Polo, Topas and Reward cultivars exhibited a harsh feeling following enzyme treatment. When compared with cotton bending and surface properties, these results indicate that enzyme-treated *Brassica* fibres may be used in cotton spinning systems as the cotton fibres are also soft, individualized and easily bend.

Table 4.8. Spinning properties for retted and enzyme-treated fibres from twenty *Brassica* cultivars in the textile laboratory at the University of Manitoba in 2014.

Cultivar	Retted fibres		Enzyme-treated fibres	
	Bending properties[16]	**Surface properties**[17]	**Bending Properties**	**Surface properties**
Hero	Stiff	Harsh	Supple	Soft
Reston	Stiff	Harsh	Supple	Soft
Mercury	Stiff	Harsh	Supple	Soft
Venus	Stiff	Harsh	Supple	Soft
Neptune	Stiff	Harsh	Supple	Soft
Red River 1861	Stiff	Harsh	Supple	Soft
HYHEAR 1	Stiff	Harsh	Supple	Soft
Global	Stiff	Harsh	Supple	Soft
Westar	Stiff	Harsh	Supple	Soft
O2R276	Stiff	Harsh	Supple	Soft
Sentry	Stiff	Harsh	Supple	Soft
UM No. 2407	Stiff	Harsh	Supple	Soft
UM No. 2257	Stiff	Harsh	Supple	Soft
Defender	Stiff	Soft	Supple	Soft
Stellar	Stiff	Harsh	Supple	Soft
Apollo	Stiff	Harsh	Supple	Soft
Polo	Stiff	Harsh	Supple	Harsh
Topas	Stiff	Harsh	Supple	Harsh
Arid	Stiff	Harsh	Supple	Soft
Reward	Stiff	Harsh	Supple	Harsh

[16] Evaluated according to AATCC evaluation procedure 5 (AATCC, 2010c)
[17] Evaluated according to AATCC evaluation procedure 5 (AATCC, 2010c)

4.36 Thermal properties of enzyme-treated *Brassica* fibres

The decomposition temperature of different *Brassica* fibres ranged between 225 ^{0}C and 260 ^{0}C (Figure 4.1). The highest decomposition temperature (260 ^{0}C) was recorded for Apollo and the lowest (225 ^{0}C) was found for Hero and Reston (Figure 4.1). The average decomposition temperature of cotton (245 ± 5.77 ˚C) was also found by the same procedure used for analyzing *Brassica* fibres in this research.

4.37 Mechanical properties

Table 4.9 illustrates the average diameter, average breaking load, fibre count (tex) and tenacity of corresponding fibre samples. The tenacity of the fibres ranged between 0.012 N/tex and 0.085 N/tex (Table 4.9). The highest tenacity of the fibres was obtained for HYHEAR 1 (0.085 N/tex), and the lowest was found for Red River 1861 (0.012 N/tex). The tenacity of cotton fibres ranged from 0.26 N/tex to 0.44 N/tex in standard conditions (Collier & Epps, 1999), whereas bundle strength of cotton was recorded between 0.22 N/tex and 0.45 N/tex by USTER STATISTICS® 2007 (Uster Technologies AG., 2006). Steinmann (1998) mentioned that the tenacity of cotton ranged between 0.18 N/tex and 0.44 N/tex, whereas ramie ranged from 0.39 N/tex to 0.64 N/tex (Roy & Lutfar, 2012b) (Table 4.10). Figure 4.2 shows the comparison of average tenacity of virgin-retted *Brassica* (0.91 N/tex), enzyme-treated rapeseed (0.04 N/tex) and canola (0.029 N/tex) fibre, as well as other available natural fibres such as, cotton (0.34 N/tex), flax (0.52 N/tex), hemp (0.53 N/tex) and ramie (0.54 N/tex). Unlike other natural fibres mentioned, *Brassica* fibers demonstrated variation (Table 4.9 and Table 4.10) in tenacity, probably due to cultivar variation.

Table 4.9. Mechanical properties of enzyme-treated fibres from twenty *Brassica* cultivars in the textile laboratory at the University of Manitoba in 2014.

Cultivar	Average diameter (µm)	Average fibre breaking load (N)	Tex	Tenacity (N/tex)
Hero	133.19 ± (70.42)[18]	0.49 ± (0.41)[19]	20.90	0.023
Reston	116.79 ± (52.58)	0.92 ± (0.30)	16.07	0.057
Mercury	84.07 ± (42.60)	0.40 ± (0.36)	8.33	0.048
Venus	151.22 ± (79.83)	0.67 ± (0.39)	26.94	0.025
Neptune	123.30 ± (61.99)	0.54 ± (0.29)	17.91	0.030
Red River 1861	222.61 ± (53.86)	0.70 ± (0.61)	58.39	0.012
HYHEAR 1	124.67 ± (65.82)	1.55 ± (0.86)	18.31	0.085
Global	132.80 ± (38.64)	0.62 ± (0.39)	20.78	0.030
Westar	130.05 ± (50.10)	0.48 ± (0.26)	19.93	0.024
O2R276	173.13 ± (63.53)	0.79 ± (0.87)	35.31	0.022
Sentry	114.73 ± (54.67)	0.66 ± (0.31)	15.51	0.043
UM No. 2407	100.77 ± (21.20)	0.50 ± (0.27)	11.96	0.042
UM No. 2257	145.47 ± (48.59)	0.60 ± (0.21)	24.93	0.024
Defender	127.51 ± (53.03)	0.45 ± (0.30)	19.16	0.023
Stellar	144.51 ± (36.93)	0.58 ± (0.21)	24.61	0.024
Apollo	123.78 ± (29.96)	0.54 ± (0.34)	18.05	0.030
Polo	139.41 ± (64.85)	0.69 ± (0.59)	22.90	0.030
Topas	194.52 ± (114.32)	1.30 ± (0.47)	44.58	0.029
Arid	133.46 ± (81.07)	0.72 ± (0.52)	20.99	0.034
Reward	194.15 ± (95.48)	0.78 ± (0.77)	44.41	0.018

[18] Standard deviation
[19] Standard deviation

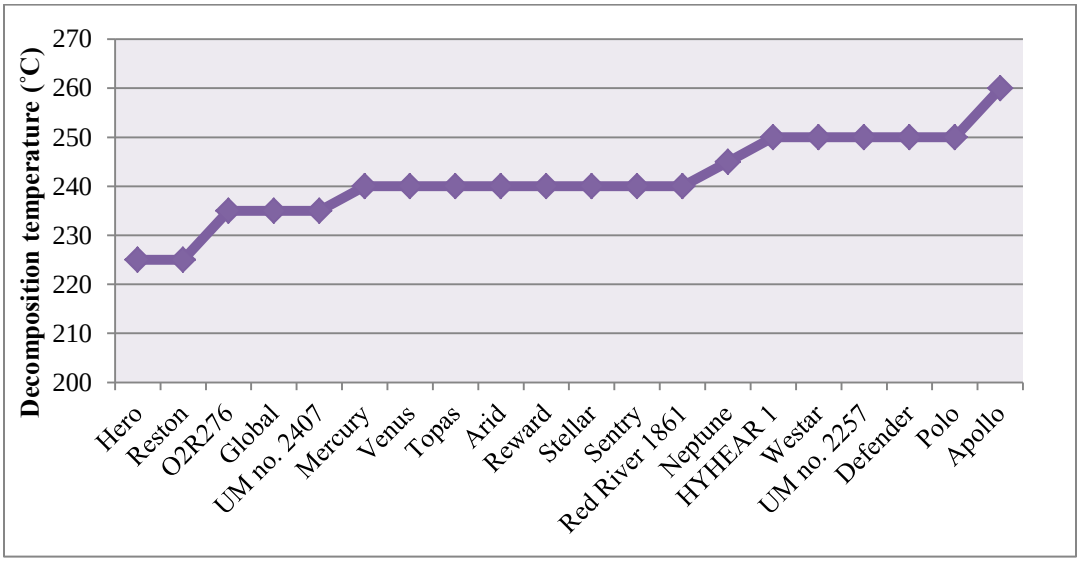

Figure 4.1. Decomposition temperature of enzyme-treated fibres from twenty different *Brassica* cultivars in the textile laboratory at the University of Manitoba in 2014.

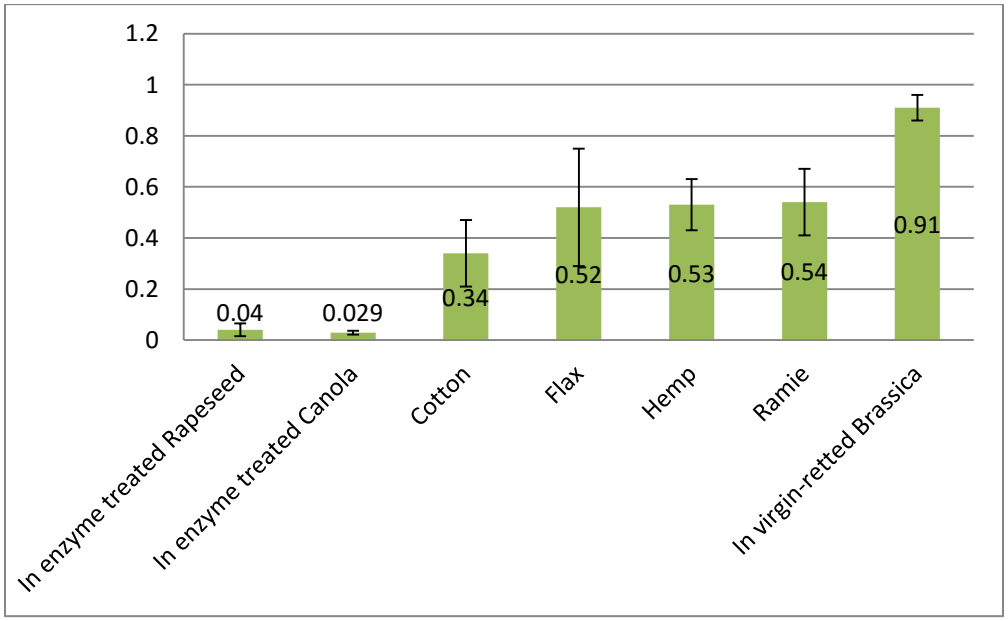

Figure 4.2. Tenacity of virgin-retted *Brassica* fibres, enzyme-treated rapeseed and canola fibre, as well as other natural fibre: cotton, flax, hemp and ramie with standard deviation from Table 4.8 and Table 4.9.

Table 4.10. Tenacity of different natural fibres compiled from previous literature.

Fibre	Tenacity (minimum)	Tenacity (maximum)	Tenacity (mean ± standard deviation)	Reference
Cotton	0.26 N/tex	0.44 N/tex		(Collier & Epps, 1999)
Cotton	0.18 N/tex	0.44 N/tex		(Steinmann, 1998)
Cotton	0.19 N/tex	0.45 N/tex		(Morton & Hearle, 2008)
Cotton	0.27 N/tex	0.44 N/tex	0.34 ± 0.13 N/tex	(Hudson, Clapp, & Kness, 1993)
Cotton	0.19 N/tex	0.56 N/tex		(Foulk, Akin, Dodd, & Ulven, 2011)
Cotton (bundle)	0.22 N/tex	0.45 N/tex		(Uster Technologies AG., 2006)
Flax	0.44 N/tex	0.95 N/tex		(Foulk et al., 2011)
Flax	0.51 N/tex	0.55 N/tex		(Foulk et al., 2011)
Flax	0.23 N/tex	0.68 N/tex	0.52 ± 0.23 N/tex	(Hudson et al., 1993)
Flax	0.26 N/tex	0.54 N/tex		(Foulk et al., 2011)
Hemp	0.51 N/tex	0.60 N/tex	0.53 ± 0.10 N/tex	(Foulk et al., 2011)
Hemp	0.39 N/tex	0.60 N/tex		(Foulk et al., 2011)
Ramie	0.39 N/tex	0.64 N/tex	0.54 ± 0.13 N/tex	(Roy & Lutfar, 2012b)
Ramie	0.47 N/tex	0.65 N/tex		(Hudson et al., 1993)

Cotton, flax, hemp and ramie demonstrate better tenacity compared to enzyme-treated *Brassica* fibres (Table 4.9). However, virgin-retted *Brassica* fibres demonstrated better tenacity (0.91 N/tex) (Sevenhuysen & Rahman, 2015) than other available natural fibres such as cotton (0.34 N/tex), flax (0.52 N/tex), hemp (0.53 N/tex) and ramie (0.54 N/tex). A probable reason for the deterioration of tenacity could be the treatment of the *Brassica* fibre samples with enzyme. The purpose of the enzyme treatment was the individualization of the fibre, which was achieved, but the treatment may have damaged the fibre strength by damaging the bonds between cellulosic

chains. Despite the enzyme-treated fibres demonstrating low tenacity, they are still spinnable for yarn preparation. For example, rayon and acetate are a regenerated cellulosic fibre widely used as yarn or fabric with lower tenacity values of 0.07 N/tex and 0.11 N/tex, respectively (Collier & Epps, 1999).

4.38 Colorimetric value

Table 4.11 denoted the L, a and b values for the left over dye solution of different *Brassica* fibres. As the reactive Blue 4 dye was used for the dyeing of the samples, all of the solutions resulted in a higher bluish (-ve b) hue than reddish (+ve a) and greenish (- ve a) hue. The range of leftover dye solution was -5.41 and -7.50 for Topas and O2R276, respectively. This means Topas demonstrated the highest dye absorbency among the twenty cultivars. The variation of dye absorbency may be due to the presence of crystalline and amorphous regions in the fibre forming cellulosic polymer chain as the dye absorbency increase with the increase of amorphous region (Collier, Bide, & Tortora, 2009).

Table 4.11. L, a, b values of the waste dyed solution from dyeing of enzyme-treated fibres of twenty *Brassica* cultivars in the textile laboratory at University of Manitoba in 2014.

Cultivar	Species	L[20]	a[21]	b[22]
Hero	*B. napus*	14.41	-2.70	-5.88
Reston	*B. napus*	14.99	-2.74	-6.63
Mercury	*B. napus*	---	---	---
Venus	*B. napus*	14.78	-2.60	-5.87
Neptune	*B. napus*	15.14	-2.94	-7.02
Red River 1861	*B. napus*	---	---	---
HYHEAR 1	*B. napus*	12.09	-3.00	-6.62
Global	*B. napus*	12.28	-3.11	-6.96
Westar	*B. napus*	15.09	-3.00	-7.36
O2R276	*B. napus*	13.68	-3.00	-7.50
Sentry	*B. napus*	---	---	---
UM no. 2407	*B. napus*	13.61	-2.90	-6.84
UM no. 2257	*B. napus*	12.77	-3.08	-7.12
Defender	*B. napus*	13.23	-2.62	-5.69
Stellar	*B. napus*	12.94	-2.58	-5.45
Apollo	*B. napus*	---	---	---
Polo	*B. napus*	12.97	-2.74	-5.68
Topas	*B. napus*	12.59	-2.72	-5.41
Arid	*B. juncea*	15.58	-2.97	-6.94
Reward	*B. rapa*	13.84	-3.03	-7.01

[20] L = lightness of color (0 = black, 100 = white)
[21] a = +ve value indicates red and –ve value indicated green
[22] b = +ve value indicates yellow and –ve value indicates blue

4.4 Conclusion

Prior to this research, it was hypothesized that *Brassica* cultivars may have an impact on fibre properties. During processing of the fibres from *Brassica* plant stems, it was observed that lowest retting time (5 days) and highest fibre yield (13.82 %) resulted from Reward and Red River 1861 cultivars. Virgin-retted *Brassica* fibres were stiff, whereas the bending and surface properties were improved by treating the fibre samples with pectinase enzyme. Apollo demonstrated the best performance in terms of decomposition temperature (260 ˚C). In this research, different fibre processing parameters and the fibre properties of twenty cultivars were evaluated, and were found that HYHEAR 1 and Reston demonstrated the better fibre properties, considering all fibre properties (retting time, tenacity, fiber surface properties). The above results indicate that the cultivars have an impact on different textile and spinning properties for retted and enzyme-treated *Brassica* fibres.

Chapter 5 General Conclusions

Brassica plants are mainly harvested for their seeds to produce industrial oil, edible oil and protein meal. These plants are already proven for the economic contributions (Daun, 2011). However, a large portion of the *Brassica* plant stems are left in the field following harvest. This research investigated the suitability of extracted fibres for apparel applications using these unused stems to create another income stream for farmers. Plant stems were collected and retted for isolating the fibres using the water-retting technique. The textile and spinning properties of the extracted fibres were evaluated and compared with the most used natural fibre, cotton. It was observed that retted fibres (virgin) have multiple required textile properties (strength, decomposition temperature, dyeability); however, virgin fibres are rigid and attached to one another by non-cellulosic materials, which may create difficulties during spinning. To overcome this problem, fibres were treated with three different treatment procedures: 1) alkali, acid and softener treatment, 2) enzyme treatment, and 3) enhanced enzyme treatment. It was found that both enzyme- and enhanced-enzyme-treated fibre samples had sufficient flexibility and single fibre entity required for processing in spinning machines. As for the textile properties of the enzyme-treated fibres, although the strength was reduced drastically compared to virgin fibre, the strength was still high enough for spinning.

To understand the effect of *Brassica* cultivars on the textile and spinning properties, seeds from twenty different cultivars were collected from three different species. The plants were grown and harvested in the greenhouse under standard growing conditions. The stems were water-retted and treated with enzyme. The textile and spinning properties of the twenty cultivars had significant variation.

Based on the results of the study, *Brassica* fibres have the potential to be used as a raw material for the textile and apparel applications, though the virgin fibres need to be modified through enzyme treatments. A further detailed study is needed to ensure the suitability of the fibre for spinning such as fibre length and diameter, and frictional rigidity or torsional rigidity. However, the challenge of utilizing *Brassica* fibres will occur during scale-up (i.e., transferring the lab processes to an industrial scale to have a realistic economic benefit).

This research was carried out in the lab and the fibre was extracted manually from regular shaped precut stems. To transfer these results into an industrial scalable process, the retting steps should be carried out mechanically. The plant stems also grow with irregularly branched structure and have irregular nodes. The major challenge will be isolating the fibres mechanically from the plant stems and maintaining the spinning properties of the fibre due to the presence of the irregular branches and nodes. Additionally, the chemical composition of the plant stems may vary due to the variable environmental growth conditions. This may create difficulty to develop a consistent protocol for extracting and individualizing the fibres by retting and enzyme treatment, respectively.

Chapter 6 Future Research

Micro-organisms are responsible for the acceleration of the retting process (Di Candilo et al., 2010; Donaghy et al., 1990). In this research, retting for the isolation of the fibres from the plant stem was carried out, but the micro-organisms, responsible for the isolation, were not identified. It may be a great discovery if the micro-organisms can be detected and produced industrially, which will significantly save retting time and improve the quality of the retted fibres as the retting process will be controlled according to the required fibre properties.

While conducting this research, it was discovered that retted water produces a sharp odor. An immediate concern was whether this liquid is harmful for both human health and natural ecosystem. For this reason, the presence of different metal contents was evaluated. The impact of this water on the ecosystem (following drainage) was not evaluated. It will also be beneficial to determine whether retted water produces gram negative bacteria (e.g., *Klebsiella pneumonia*) that may cause harmful effects on human health, during processing or within the final product.

The fibres obtained from water retting were stiff and attached to each other. Pectin is responsible for this attachment of the bast fibres. Fibre composition of the plant stems were assessed for *Brassica* plants from the literature review (Hosseinpour et al., 2010; Tofanica et al., 2011). Tofanica et al. (2011) mentioned the composition of fibres and confirmed the presence of pectin, though the results were for the evaluation of fibre properties related to the pulping process. As a consequence, fibre samples were treated with pectinase enzyme resulting in individualized fibres. It will be useful for researchers to determine the compositions of *Brassica* fibres before and after enzyme treatment for the textile processing.

In this experiment some important fibre properties (hand feel, individualization, flexibility, tenacity, decomposition temperature and dyeability) were evaluated. But there are many other fibre properties, such as fibre length and breaking twist angle, which are also important for the processing of fibres in different machines. This research needs to be conducted to fully understand the potential of *Brassica* fibres.

In this research, the fibre yield (%) of twenty different fibres was evaluated. However, the variation of seeding rate affected the fibre yield of hemp (*Cannabis sativa* L.) (Cromack, 1998). The hypothesis is that seeding rate and other agronomic practices might also have an impact on the fibre yield for the *Brassica* cultivars. As a consequence, it will be important to analyze the productivity of the fibres on the basis of seeding rate for rapeseed and canola.

References

AATCC. (2010a). AATCC Test Method 61-2009: Colorfastness to Laundering: Accelerated. In *AATCC Technical Manual* (pp. 86–90). NC, USA: American Association of Textile Chemists and Colorists.

AATCC. (2010b). AATCC Test Method 8-2007: Colorfastness to Crocking: Crockmeter Method. In *AATCC Technical Manual* (pp. 19–21). NC, USA: American Association of Textile Chemists and Colorists.

AATCC. (2010c). Fabric Hand: Guidelines for the Subjective Evaluation of. In *AATCC Technical Manual* (Vol. 85, pp. 371–373). NC, USA: American Association of Textile Chemists and Colorists.

Akin, D. E., Dodd, R. B., & Foulk, J. a. (2005). Pilot plant for processing flax fiber. *Industrial Crops and Products*, *21*(3), 369–378. http://doi.org/10.1016/j.indcrop.2004.06.001

Allard, R. W. (1999). *Principles of Plant Breeding* (2nd ed.). New York: John Wiley & Sons, Ltd.

ASTM International. (2008). *Standard Practice for Conditioning and Testing Textiles*. West Conshohocken, United States. http://doi.org/10.1520/D1776-08E01.2

ASTM International. (2015). *ASTM D123-15b Standard Terminology Relating to Textiles*. West Conshohocken, United States. http://doi.org/10.1520/D0123-13AE01.1.4.3

Baltazar-Y-Jimenez, A., & Sain, M. (2012). Natural Fibres for automotive applications. In R. M. Kozłowski (Ed.), *Handbook of Natural Fibres: Volume 2 Processing and Applications* (pp. 219–253). Cambridge, UK: Woodhead Publishing Limited. http://doi.org/10.1533/9780857095510.2.219

Booth, J. E. (1968). *Principles of textile testing: an introduction to physical methods of testing textile fibres, yarns and fabrics* (3rd ed.). London: Heywood Books.

Booth, J. E. (1975). *Textile Mathematics, Volume Two*. Manchester: The Textile Institute.

Canadian Council of Ministers of the Environment. (2010). *A Review of the Current Canadian Legislative Framework for Wastewater Biosolids*. Retrieved from http://www.ccme.ca/files/Resources/waste/biosolids/pn_1446_biosolids_leg_review_eng.pdf

Canola Council of Canada. (2014). Canola Varieties. Retrieved November 30, 2015, from http://www.canolacouncil.org/crop-production/canola-grower's-manual-contents/chapter-2-canola-varieties/canola-varieties

Canola Council of Canada. (2015). Harvet Management. Retrieved June 11, 2016, from http://www.canolacouncil.org/canola-encyclopedia/managing-harvest/harvest-management/

Canola Council of Canada. (2016). Canadian canola Production - Updated January 13, 2016. Retrieved August 15, 2016, from http://www.canolacouncil.org/markets-stats/statistics/tonnes/

Carmichael, A. (2015). Man-Made Fibers Continue To Grow. Retrieved April 5, 2015, from http://www.textileworld.com/textile-world/fiber-world/2015/02/man-made-fibers-continue-to-grow/

Carothers, W. H. (1931). Polymerization. *Chemical Reviews*, *8*(3), 353–426. http://doi.org/10.1021/cr60031a001

Collier, A. M. (1970a). *A Handbook of Textiles* (1st ed.). New York: Pergamon Press.

Collier, A. M. (Ed.). (1970b). *A Handbook of Textiles* (1st ed.). New York: Pergamon Press.

Collier, B. J., Bide, M. J., & Tortora, P. G. (2009). *Understanding Textiles* (7th ed.). Upper Saddle River, New Jersey: Pearson Prentice Hall.

Collier, B. J., & Epps, H. H. (1999). *Textile Testing and Analysis*. Upper Saddle River, New Jersey: Prentice Hall.

Cotton Incorporated. (2015). Classification of Cotton. Retrieved from http://www.cottoninc.com/fiber/quality/Classification-Of-Cotton/

Cromack, H. T. H. (1998). The effect of cultivar and seed density on the production and fibre content of Cannabis sativa in southern England. *Industrial Crops and Products*, *7*(2-3), 205–210. http://doi.org/10.1016/S0926-6690(97)00049-6

Danton, J. A. (1990). *Society and the official world: a reintroduction to sociology*. New York: General Hall.

Daun, J. K. (2011). Origin, DIstribution, and Production. In *Canola - Chemistry, Production, Processing and Utilization* (pp. 1–27). Urbana, Illinois, USA: AOCS Press.

Di Candilo, M., Bonatti, P. M., Guidetti, C., Focher, B., Grippo, C., Tamburini, E., & Mastromei, G. (2010). Effects of selected pectinolytic bacterial strains on water-retting of hemp and fibre properties. *Journal of Applied Microbiology*, *108*(1), 194–203. http://doi.org/10.1111/j.1365-2672.2009.04409.x

Diederichsen, A., & McVetty, P. B. E. (2011). Botany and Plant Breeding. In J. K. Daun, A. M. Neskin, & D. Hickling (Eds.), *Canola - Chemistry, Production, Processing and Utilization* (pp. 29–56). Urbana, Illinois, USA: AOCS Press.

Dochia, M., & Sirghie, C. (2012). Cotton fibres. In R. M. Kozlowski (Ed.), *Handbook of Natural Fibres: Volume 1 Types, Properties and Factors Affecting Breeding and Cultivation* (1st ed., pp. 11–23). Cambridge, UK: Woodhead Publishing Limited.

Donaghy, J. A., Levett, P. N., & Haylock, R. W. (1990). Changes in microbial populations during anaerobic flax retting. *Journal of Applied Bacteriology*, *69*(5), 634–641. http://doi.org/10.1111/j.1365-2672.1990.tb01556.x

Ebskamp, M. J. . (2002). Engineering flax and hemp for an alternative to cotton. *Trends in Biotechnology*, *20*(6), 229–230. http://doi.org/10.1016/S0167-7799(02)01953-4

Euler Hermes. (2016). Sector Risks. Retrieved July 13, 2016, from http://www.eulerhermes.com/economic-research/sector-risks/Pages/default.aspx

Farag, R., & Elmogahzy, Y. (2009). Tensile properties of cotton fibers. In A. R. Bunsell (Ed.), *Handbook of tensile properties of textile and technical fibres* (pp. 51–72). Cambridge, UK: Woodhead Publishing in association with the Textile Institute.

Food Production and Inspection. (1985). *Description of Global variety*. *Agriculture Canada*.

Food Production and Inspection. (1987). *Description of Topas variety*. *Agriculture Canada*.

Foulk, J. A., Akin, D. E., Dodd, R. B., & Ulven, C. (2011). Production of Flax Fibers for Biocomposites. In S. Kalia, B. S. Kaith, & I. Kaur (Eds.), *Cellulose Fibers: Bio- and Nano-Polymer Composites, Green Chemistry and Technology* (pp. 61–96). London, New York: Springer.

Gómez-Campo, C. (1999). Taxonomy. In C. Gómez-Campo (Ed.), *Biology of Brassica coenospecies* (pp. 3–32). Amsterdam, The Netharlands: Elsevier Science. Retrieved from https://books.google.ca/books?hl=en&lr=&id=bA0-GcScHYgC&oi=fnd&pg=PP2&dq=Biology+of+Brassica+Coenospecies&ots=X97uNqpPkS&sig=Uoa1BqCPhBX-s0Jzjs282nWGez0

Gordon, S. (2007). Cotton fibre quality. In S. Gordon & Y.-L. Hsieh (Eds.), *Cotton: Science and Technology* (1st ed., pp. 68–102). Boca Raton, USA: Woodhead Publishing Limited.

Gratton, J.-L., & Chen, Y. (2004). Development of a field-going unit to separate fiber from Hemp (Cannabis Sativa) stalk. *Applied Engineering in Agriculture*, *20*(2), 139–145. http://doi.org/10.13031/2013.15882

Haque, M. S., Asaduzzaman, M., Akhter, F., Hossain, M. M., & Ahmed, Z. (2001). Impact of Stem-water Ratio and Separately Retting the Top and Basal Parts of Jute on the Quality of Fibre. *Pakistan Journal of Biological Sciences*. http://doi.org/10.3923/pjbs.2001.1191.1193

Harwood, J., & Harwood, R. (2012). Testing of natural textile fibres. In R. M. Kozlowski (Ed.), *Handbook of Natural Fibres Types, Volume 1- Properties And Factors Affecting Breeding And Cultivation* (pp. 345–390). Cambridge, UK: Woodhead Publishing in association with the Textile Institute.

Hatch, K. L. (1993). *Textile science*. Saint Paul, Minneapolis: West Publishing Company.

Hayward, A. (2011). Introduction—Oilseed Brassica. In D. Edwards, J. Batley, I. Parkin, & C. Kole (Eds.), *Genetics, Genomics and Breeding of Oilseed Brassicas* (pp. 1–13). Hoboken: Science Publishers.

Hearle, J. W. S. (2007). Physical Structure and properties of cotton. In S. Gordon & Y.-L. Hsieh (Eds.), *Cotton: Science and Technology* (1st ed., pp. 35–67). Boca Raton, USA: Woodhead Publishing in association with the Textile Institute.

Horne, M. R. L. (2012). Bast fibres: hemp cultivation and production. In R. M. Kozlowski (Ed.), *Handbook of Natural Fibres: Volume 1 Types, Properties and Factors Affecting Breeding and Cultivation* (pp. 114–145). Cambridge, UK: Woodhead Publishing in association with the Textile Institute.

Hosseinpour, R., Fatehi, P., Latibari, A. J., Ni, Y., & Sepiddehdam, S. J. (2010). Canola straw chemimechanical pulping for pulp and paper production. *Bioresource Technology*, *101*(11), 4193–7. http://doi.org/10.1016/j.biortech.2010.01.055

Hsieh, Y.-L. (2007). Chemical structure and properties of cotton. In S. Gordon & Y.-L. Hsieh (Eds.), *Cotton: Science and Technology* (1st ed., pp. 3–34). Boca Raton, USA: Woodhead Publishing Limited.

Hudson, P. B., Clapp, A. C., & Kness, D. (1993). *Joseph's introductory textile science*. Fort Worth: Harcourt Brace Jovanovich College Publishers.

Hunter, L. (2007). Cotton spinning technology. In S. Gordon & Y.-L. Hsieh (Eds.), *Cotton: Science and Technology* (1st ed., pp. 240–274). Boca Raton, USA: Woodhead Publishing Limited.

Judkins, G. (2008). Declining cotton cultivation in Maricopa County, Arizona: An examination of macro-and micro-scale driving forces. *Yearbook of the Association of Pacific Coast Geographers*, *70*(1), 70–95.

Kaliyamoorthi, K., & Thangavelu, R. (2015). Union dyeing of cotton/nylon blended fabric by plasma-nano chitosan treatment. *Fashion and Textiles*, *2*(1), 10. http://doi.org/10.1186/s40691-015-0035-8

Khan, M. M. R., Chen, Y., Laguë, C., Landry, H., Peng, Q., & Zhong, W. (2013). Hemp (Cannabis sativa L.) Decortication Using the Drop Weight Method. *Applied Engineering in Agriculture*, *29*(1), 79–87. http://doi.org/10.13031/2013.42521

King, D. (2007). Dyeing cotton and cotton products. In S. Gordon & Y.-L. Hsieh (Eds.), *Cotton: Science and Technology* (1st ed., pp. 353–380). Boca Raton, USA: Woodhead Publishing Limited.

Klassen, A. J., Downey, R. K., & Capcara, J. J. (1987). Westar Summer Rape. *Canadian Journal of Plant Science*, *67*(2), 491–493. http://doi.org/10.4141/cjps87-068

Kolodziejczyk, P., Ozimek, L., & Kozlowska, J. (2012). The application of flax and hemp seeds in food, animal feed and cosmetics production. In R. M. Kozlowski (Ed.), *Handbook of Natural Fibres: Volume 2 Processing and Applications* (1st ed., pp. 329–366). Cambridge, UK: Woodhead Publishing Limited.

Konczewich, W., & Kozlowski, R. M. (2012). Enzymatic treatment of natural fibres. In R. M. Kozlowski (Ed.), *Handbook of Natural Fibres: Volume 2 Processing and Applications* (1st ed., pp. 168–184). Cambridge, England: Woodhead Publishing Limited.

Kozlowski, R. M., Mackiewicz-Talarczyk, M., & Allam, A. M. (2012). Bast Fibres: Flax. In R. M. Kozłowski (Ed.), *Handbook of Natural Fibres: Volume 1 Types, Properties and Factors*

Affecting Breeding and Cultivation (pp. 56–113). Cambridge, UK: Woodhead Publishing Limited. http://doi.org/10.1533/9780857095503.1.56

Kozlowski, R. M., Mackiewicz-Talarczyk, M., Muzyczek, M., & Barriga-Bedoya, J. (2012). Future of Natural Fibers, Their Coexistence and Competition with Man-Made Fibers in 21st Century. *Molecular Crystals and Liquid Crystals*, *556*(1), 200–222. http://doi.org/10.1080/15421406.2011.635962

Lawrence, C. (2007). The opening, blending, cleaning, and carding of cotton. In S. Gordon & Y.-L. Hsieh (Eds.), *Cotton: Science and Technology* (1st ed., pp. 203–239). Boca Raton, USA: Woodhead Publishing in association with the Textile Institute.

Magnusson, K., & Svennerstedt, B. (2007). Influence of temperature on the water retting process of hemp (Cannabis sativa L.) cultivated under Swedish climate conditions. *Journal of Industrial Hemp*, *12*(2), 3–17. http://doi.org/10.1300/J237v12n02_02

Mather, R. R., & Wardman, R. H. (2015). *The Chemistry of Textile Fibres* (2nd ed.). Cambridge, UK: The Royal Society of Chemistry.

Mcintyre, J. E. (1998). Polyester fibers. In M. Lewin & E. M. Pearce (Eds.), *Handbook of Fiber Chemistry* (2nd ed., pp. 1–66). New York, USA: Marcel Dekker, Inc.

McVetty, P. B. E., Cuthbert, J. L., Marwede, V., Paulmann, W., Sass, O., Duncan, R. W., … Zelmer, C. D. (2014). HYHEAR 1 hybrid Roundup Ready™ high erucic acid, low glucosinolate summer rape. *Canadian Journal of Plant Science*, *94*(2), 453–455. http://doi.org/10.4141/cjps2013-169

McVetty, P. B. E., Duncan, R. W., Fernando, W. G. D., Li, G., & Zelmer, C. D. (2012). Red River 1861 Roundup Ready™ high erucic acid, low glucosinolate summer rape. *Canadian Journal of Plant Science*, *92*(7), 1407–1409. http://doi.org/10.4141/cjps2012-126

McVetty, P. B. E., Rimmer, S. R., Scarth, R., & van den Berg, C. G. J. (1996). Neptune high erucic acid, low glucosinolate summer rape. *Canadian Journal of Plant Science*, *76*(2), 343–344. http://doi.org/10.4141/cjps96-061

McVetty, P. B. E., & Scarth, R. (2012). Breeding Oil Quality for Edible and Industrial Applications in Oilseed Rape and Linseed. In S. K. Gupta (Ed.), *Technological Innovations in Major World Oil Crops, Volume 2* (pp. 105–122). New York, NY: Springer New York. http://doi.org/10.1007/978-1-4614-0827-7

McVetty, P. B. E., Scarth, R., Rimmer, S. R., & Van Den Berg, C. G. J. (1996). Venus high erucic acid, low glucosinolate summer rape. *Canadian Journal of Plant Science*, *76*(2), 341–342. http://doi.org/10.4141/cjps96-060

Mohapatra, S. P., & Mitchell, A. (2003). *Drinking Water Quality Standards in Ontario – Are They Tough ? Canadian Institute for Environmental Law and Policy*. Toronto, Ontario.

Morton, W. E., & Hearle, J. W. S. (2008). *Physical Properties of Textile Fibres* (4th ed.). Cambridge, England: Woodhead Publishing in association with the Textile Institute. http://doi.org/10.1533/9781845694425.163

Munder, F., & Fürll, C. (2004). Effective processing of bast fiber plants and mechanical properties of the fibers. In *ASAE Annual International Meeting* (pp. 6803–6811). http://doi.org/10.13031/2013.16960

Nayak, R. K., Padhye, R., & Fergusson, S. (2012). Identification of natural textile fibres. In R. M. Kozłowski (Ed.), *Handbook of Natural Fibres: Volume 1 Types, Properties and Factors Affecting Breeding and Cultivation* (pp. 314–344). Cambridge, UK: Woodhead Publishing Limited. http://doi.org/10.1533/9780857095503.1.314

OEKO TEX Association. (2013). Limit values and fastness. Retrieved July 26, 2015, from https://www.oeko-tex.com/en/manufacturers/test_criteria/limit_values/limit_values.html

Ostergaard, L., & King, G. J. (2008). Standardized gene nomenclature for the Brassica genus. *Plant Methods*, *4*, 10. http://doi.org/10.1186/1746-4811-4-10

Piotrowski, S., & Carus, M. (2010). Natural Fibres in Technical Applications: Market and Trends. In J. Müssig (Ed.), *Industrial Applications of Natural Fibres* (pp. 73–86). Chichester, UK: John Wiley & Sons, Ltd. http://doi.org/10.1002/9780470660324

Podolska, G. (2011). Plant Lodging, Effects, and Control. In J. Gliński, J. Horabik, & J. Lipiec (Eds.), *Encyclopedia of Agrophysics* (pp. 609–610). Dordrecht: Springer Netherlands. http://doi.org/10.1007/978-90-481-3585-1

Proven Seed. (1994). *Defender-- Argentine (B. napus) Canola, Everything you want in a canola variety*. Lindsay, Ontario.

Rahman, M., & East, G. C. (2006). Effect of applied stress on the alkaline hydrolysis of poly(ethylene terephthalate) at 40°C: Relevance to medical textiles. *Journal of Applied Polymer Science*, *102*(5), 4814–4822. http://doi.org/10.1002/app.24684

Rakow, G. (2011). Classical Genetics and Traditional Breeding. In C. Kole (Ed.), *Genetics, Genomics and Breeding of Oilseed Brassicas* (pp. 73–84). Hoboken: Science Publishers.

Rana, S., Pichandi, S., Parveen, S., & Fangueiro, R. (2014). Natural Plant Fibers: Production, Processing, Properties and Their Sustainability Parameters. In S. S. Muthu (Ed.), *Roadmap to Sustainable Textiles and Clothing: Eco-friendly Raw Materials, Technologies, and Processing Methods* (pp. 1–36). New York: Springer.

Rao, K. M. M., & Rao, K. M. (2007). Extraction and tensile properties of natural fibers: Vakka, date and bamboo. *Composite Structures*, *77*(3), 288–295. http://doi.org/10.1016/j.compstruct.2005.07.023

Rimmer, S. R., Scarth, R., & McVetty, P. B. E. (1998). Sentry summer rape. *Canadian Journal of Plant Science*, *78*(4), 615–616.

Roy, S., & Lutfar, L. B. (2012a). Bast fibres: jute. In Ryszard M. Kozlowski (Ed.), *Handbook of Natural Fibres: Volume 1 Types, Properties and Factors Affecting Breeding and Cultivation* (1st ed., pp. 24–46). Cambridge, UK: Woodhead Publishing Limited.

Roy, S., & Lutfar, L. B. (2012b). Bast fibres: ramie. In Ryszard M. Kozlowski (Ed.), *Handbook*

of Natural Fibres: Volume 1 Types, Properties and Factors Affecting Breeding and Cultivation (pp. 47–55). Cambridge, UK: Woodhead Publishing in association with the Textile Institute.

Salmon-Minotte, J., & Franck, R. R. (2005). Flax. In R. R. Franck (Ed.), *Bast and other plant fibres* (pp. 94–175). Cambridge, England: Woodhead Publishing in association with the Textile Institute.

Scarth, R., McVetty, P. B. E., & Rimmer, S. R. (1995). Mercury high erucic low glucosinolate summer rape. *Canadian Journal of Plant Science*, *75*(1), 205–206.

Scarth, R., McVetty, P. B. E., Rimmer, S. R., & Stefansson, B. R. (1988). Stellar Low Linolenic-High Linolenic Acid Summer Rape. *Canadian Journal of Plant Science*, *68*(2), 509–511. http://doi.org/10.4141/cjps88-061

Scarth, R., McVetty, P. B. E., Rimmer, S. R., & Stefansson, B. R. (1991). Hero summer rape. *Canadian Journal of Plant Science*, *71*(3), 865–866. http://doi.org/10.4141/cjps91-124

Scarth, R., Rimmer, S. R., & McVetty, P. B. E. (1992). Reward summer turnip rape. *Canadian Journal of Plant Science*, *72*(3), 839–840. http://doi.org/10.4141/cjps92-102

Scarth, R., Rimmer, S. R., & McVetty, P. B. E. (1995). Apollo low linolenic summer rape. *Canadian Journal of Plant Science*, *75*(1), 203–204. http://doi.org/10.4141/cjps95-037

Sevenhuysen, G., & Rahman, M. (2015). Textile fibres and Textiles from Brassica plants. Canada.

Shen, L., Worrell, E., & Patel, M. K. (2010). Environmental impact assessment of man-made cellulose fibres. *Resources, Conservation and Recycling*, *55*(2), 260–274. http://doi.org/10.1016/j.resconrec.2010.10.001

Shree Sanjay Trading Co. (n.d.). Indian cotton info. Retrieved from http://www.cottonin.com/ind_cot_inf.htm

Stefansson, B. R., & Kondra, Z. P. (1975). Tower Summer Rape. *Canadian Journal of Plant Science*, *55*(1), 343–344. http://doi.org/10.4141/cjps75-053

Steinmann, H. W. (1998). Cellulose Acetate and Triacetate Fibers. In M. Lewin & E. M. Pearce (Eds.), *Handbook of Fiber Chemistry* (2nd ed., pp. 803–868). New York: Marcel Dekker, Inc.

Sun, X. S. (2005). Overview of plant polymers: resources, demands, and sustainability. In R. P. Wool & X. S. Sun (Eds.), *Bio-Based Polymers and Composites* (pp. 1–14). Amsterdam: Elsevier Academic Press. http://doi.org/10.1016/B978-012763952-9/50002-2

The Textile Institute textile terms and definitions committee. (1991). *Textile terms and definitions*. (M. C. Tubbs & P. N. Daniels, Eds.) (9th ed.). Manchester, UK: The Textile Institute.

Thomas, S., Paul, S. A., Pothan, L. A., & Deepa, B. (2011). Natural Fibres: Structure, Properties

and Applications. In S. Kalia, B. S. Kaith, & I. Kaur (Eds.), *Cellulose Fibers: Bio- and Nano-Polymer Composites; Green Chemistry and Technology* (pp. 3–42). Newyork: Springer. http://doi.org/10.1007/978-3-642-17370-7

Tofanica, B. M., Cappelletto, E., Gavrilescu, D., & Mueller, K. (2011). Properties of Rapeseed (*Brassica napus*) Stalks Fibers. *Journal of Natural Fibers*, *8*(4), 241–262. http://doi.org/10.1080/15440478.2011.626189

Trotman, E. R. (1975). *Dyeing and Chemical Technology of Textile Fibres* (5th ed.). London: Charles Griffin & Company Ltd.

Uster Technologies AG. (2006). *Uster® Statistics 2007 The Quality Benchmarks for the Textile Industry*. Uster, Switzerland: Uster Technologies.

Velasco, L., Goffman, F., & Becker, H. (1998). Variability for the fatty acid composition of the seed oil in a germplasm collection of the genus Brassica. *Genetic Resources and Crop Evolution*, *45*, 371–382.

Wakelyn, P. J. (2007). Health and safety issues in cotton production and rocessing. In S. Gordon & Y.-L. Hsieh (Eds.), *Cotton: Science and Technology* (1st ed., pp. 460–483). Boca Raton, USA: Woodhead Publishing in association with the Textile Institute.

Wang, W., Cai, Z., & Yu, J. (2008). Study on the Chemical Modification Process of Jute Fiber. *Journal of Engineered Fibers and Fabrics*, *3*(2), 1–11.

Yu, H., & Yu, C. (2007). Study on microbe retting of kenaf fiber. *Enzyme and Microbial Technology*, *40*(7), 1806–1809. http://doi.org/10.1016/j.enzmictec.2007.02.018

Zhong, W. (2013). *An introduction to healthcare and medical textiles*. Pennsylvania, USA: DEStech Publications, Inc.