

**The Effect of Stem Diameter on the *Brassica napus* (Type:
Canola) (Cultivar: HYHEAR 3) Fiber Quality**

by

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ABSTRACT

A matured canola plant has different types of stems based on the diameter. Therefore, this study was mainly focused on the investigation of the physical and mechanical properties of the canola fibers extracted from 3 different diameter stems (narrow, medium, and wide) and also observed the effects of a chemical softener (Cepreton UN) on the fiber properties. After treating with 2% and 10% Cepreton UN, the fibers were compared with the control fibers. Yield (%), length, diameter, strength, contact angle and also moisture regain (%) of fibers were measured. The stem diameter didn't have any effect on the yield (%) of fibers. ANOVA also showed that stem diameter had effects on all fiber properties except for average length and elongation at break. Fiber diameter had significant effects on the load at break, elongation at break, aspect ratio, tensile stress, and young's modulus. In corrgram, it was found that tensile stress, young's modulus, and aspect ratio were negatively correlated to fiber diameter whereas load at break and tenacity were mostly positively associated. The mean values of fiber diameter, elongation at break, load at break, tenacity, and contact angle were highest for fibers of 7-10 mm diameter stem. On the contrary, the mean values of the tensile stress, young's modulus, aspect ratio, and moisture regain (%) were found to be lowest for fibers of 7-10 mm diameter stem (medium mature). Therefore, 7-10 mm diameter stem fibers found to be less stiff. Moisture regain ability showed that canola fibers isolated from ≥ 8 mm stem diameter were more hydrophobic whereas contact angle measurement showed relatively more hydrophobic nature of 7-10 mm stem fibers. Overall, this study suggested to sort out different qualities of canola fibers for commercial applications. ANOVA showed that fiber diameter had strong effects on elongation at break, load at break, tensile stress, young's modulus, and aspect ratio for all fibers. Corrgram values showed that tensile stress, young's modulus, and aspect ratio were negatively and load at break and tenacity were mostly positively correlated to fiber diameter. In most cases, the fiber diameter was decreased in both 2% and 10% treated 7-10 mm stem fibers. The mean values of elongation at break, load at break, tenacity, and contact angle were decreased for 10% and increased for 2% and the mean values of tensile stress, young's modulus, and aspect ratio were decreased for 2% and increased for 10% treated 7-10 mm stem fibers. Moisture regain (%) mostly decreased for 2% and increased for 10% treated fibers. Low pH (4.5) had almost similar effect on fibers as 2% Cepreton UN. Overall, 2% Cepreton UN treatment was found to be better than 10% to make canola fibers less stiff and low pH was found to be an alternative softener treatment strategy.

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DEDICATION

This thesis is dedicated to my father and my husband

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- 1. Title: The Effect of Stem Diameter on the *Brassica napus* (Type: Canola) (Cultivar: HYHEAR 3) Fiber Quality**

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&

- 2. Title: Observation of a chemical softener's effects on stem-specific lignocellulosic *Brassica napus* (Type: Canola) (Cultivar: HYHEAR 3) fibers**

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CHAPTER 1: INTRODUCTION

1.1 Introduction

Natural fibers, that is, plant and animal fibers have different commercial applications. Bast fibers isolated from plant stems, are of current research interest due to their high availability (Thomas et al., 2011). Though in recent times, the use of synthetic fibers has been higher (Carmichael, 2015; Tortora, 1997), natural fibers are gradually being replaced due to various advantages, such as, better ability to withstand damage, lower cost etc. (Thomas et al., 2011). As a consequence, the raw materials to produce synthetic fibers are rapidly shrinking (Daun, 2011). In addition, synthetic fiber production processes increase environmental pollution (Rana et al., 2014; Shen et al., 2010). On the contrary, cotton processing also requires large quantities of water and pesticides which have detrimental effects on ecosystems (Ebskamp, 2002; Rana et al., 2014). Therefore, researchers are now focusing on alternative resources to develop fibers that are available, sustainable, and cause minimal environmental damage (Thomas et al., 2011).

Natural fibers have received increasing interest from biocomposite manufacturers for use in various applications due to their wide range of properties (Bledzki & Gassan, 1999). A composite is defined as a physical mixture of two or more materials and exhibits properties that are generally better than those of the individual materials. A suitable combination of materials is required to produce a superior composite, as individual materials alone cannot perform well at an acceptable cost. One can produce these composites in different shapes and with varying design structures by placing the structural elements side by side or on top of each other (Hautala et al., 2004). Currently, the primary interest of ongoing research is the development of biocomposites with flax, hemp, jute, coir, palm, and other natural fibers. The properties of natural fibers depend on factors such as fiber length and maturity as well as the processing methods adopted for fiber extraction. Properties such as the ultimate tensile strength, Young's modulus, density, and electrical resistivity, among others, are determined by the

internal structure and chemical composition of the fibers (Mohanty et al., 2001; Reddy & Yang, 2005). To generate biocomposites, mechanical properties such as tenacity and extensibility and thermal properties such as the melting or decomposition temperature are important for determining processing parameters as well as end uses of the final product (Hautala et al., 2004).

Natural fibers can also be used in the textile industries; for example, cotton is the most popular natural textile fiber. Interestingly, not all fibers are suitable for use as textile fibers. A textile fiber must possess certain properties, with given ranges of length, strength, fibrous structure, spinnability, flexibility, cohesiveness, elasticity, fineness, uniformity, luster, color, and the ability to react with acid or alkali. A fabric is a cloth composed of fibers. In general, fabrics are categorized into two types: woven and knitted. Other popular methods for producing fabric include lace-making, felting, net-making, non-woven processes, and tufting (Sayed et al., 2018). Research has clearly demonstrated the properties needed for a fabric to achieve maximum performance in use (Hossain et al., 2019).

Natural fibers must exhibit distinct qualities to be considered for use as textile fibers, including certain levels of durability, comfort, aesthetic appeal, health maintenance, and safety protection. Textiles should retain their original color and structure, without changing size or shape during regular washing. To maintain durability, a fabric must have a certain level of mechanical strength to tolerate the wear and tear that occurs during regular use. For example, when a dress is worn, stress is applied on the textile due to regular movement of the body parts such as the knees, elbows, and buttocks, and this stress must be absorbed by the textile (Hatch, 1993). In addition, textile fibers must tolerate spinning stress and tensile force (tenacity) during different stages, such as the winding, warping, sizing, and fabric formation (weaving and knitting) processes applied during manufacturing (Booth, 1968). Overall, the fabric strength is determined by the fiber strength. Medical textiles such as dressings and hygienic products such as diapers must have a high moisture-absorbance ability; moreover, fibers

used in wound dressings must be flexible and soft and must possess antibacterial properties for proper healing and wound protection (Gluck, 2013).

The quality of a bast fiber may vary due to the intrinsic properties of its natural components, such as the cellulose content, lignin content, fibrous nature, and fiber bundle morphology within plant stems (Bonatti-Chaves et al., 2004; Rowell et al., 2000). Examples of such bast fibers include jute, hemp, flax, ramie, which vary in their chemical compositions and in many physical and chemical properties (Bergfjord & Holst, 2010).

Among natural fibers, Canadian researchers are currently focusing on canola (*Brassica napus*) fiber, which is a plant bast fiber, for use in industrial applications. In Canada, canola contributes \$19.3 billion yearly to the national economy, creating approximately 249,000 jobs and producing \$12.5 billion in wages (Canola Council of Canada, 2015). However, in most cases, after oil production, some parts of these plants are used as animal feed while the remainder is used as waste biomass (Canola Council of Canada, 2010). By utilizing this large amount of waste biomass in textile or composite industries, substantial revenue could be produced. Moreover, using waste materials as raw materials should reduce the cost and energy involved in producing new raw materials.

Alcock et al. (2018) have investigated the mechanical properties of flax fibers based on stem maturity. However, there are no corresponding data available for canola plant fibers that are primarily used as waste biomass. If one could distinguish canola fiber qualities based on stem maturity, it would be easier to differentiate the canola stems suitable for further fiber extraction in varying commercial uses. In general, stiffer fibers are primarily used for biocomposite production (Rahman et al., 2013; Fanguiero & Rana, 2018) whereas flexible fibers are mostly used by textile industries (Beher, 2005). In this study, we focus on the assumption that stiffer fibers are extracted from more mature stems.

Softeners significantly influence various fabric properties and are a type of conditioner containing cationic, anionic, or non-ionic surfactant or alkoxyalkylamide and can be applied in laundry during the rinse cycle or transferred to the textile during drying from an inert cellulosic or polyurethane substrate (Needles, 1986). Bast fibers are usually stiff because they consist of millions of microfibrils angled inside the cellulosic fibers, with the angles of cellulosic fibers being inversely proportional to stiffness (Tanushree & Chanana, 2016). In canola fibers, the lignin content is higher and the cellulose content is lower than that of cotton and jute, which most likely explains why canola fiber is stiffer than cotton and jute. In our experiments, we applied a cationic softener (Cepreton UN) to observe its effects on stem-specific lignocellulosic canola fibers, as Cepreton UN is primarily used to softens cellulosic (cotton) fibers.

1.2 Hypothesis: Therefore, the hypothesis of our study is “Narrow diameter stem (immature) of canola plant has less stiff fibers and softener makes the fibers even less stiff”.

1.2.1 Objectives: To test this hypothesis, we have the following objectives:

1. Investigation of the physical and mechanical properties of the canola fibers extracting from the three different stem groups, such as narrow (immature), medium (mature), and wide (over mature) and finding the stem-specific effects on the extracted fibers.
2. Investigation of the effects of cationic softener (Cepreton UN) on canola fiber physical and mechanical properties and also establishing a relationship between the narrow, medium, and wide stem extracted fibers after Cepreton UN treatment.

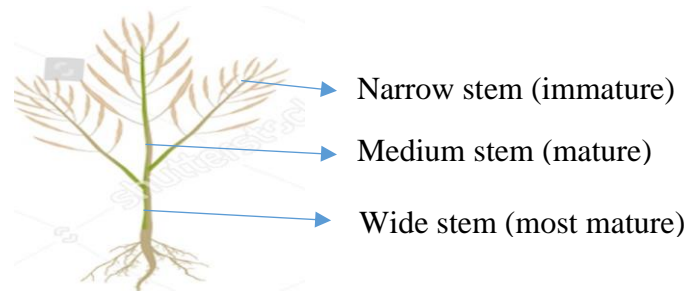


Figure 1.1: A mature canola plant

CHAPTER 2: LITERATURE REVIEW

2.1 Natural fibers: Natural fibers can be obtained from plants, animals, and geological processes (such as asbestos) (John & Thomas, 2008). Most of these fibers can be used as ecofriendly composite components for automobiles. As natural fibers such as cotton rapidly absorb sweat, they can also be used for producing fabric. Fabrics produced from cotton are lightweight and soft (Namvar et al., 2014). Cellulose is the primary chemical component of the plant fiber used to make paper (Fangueiro & Rana, 2017; Sfiligoj et al., 2013).

2.1.1 Applications of natural fibers: In addition to their use in textiles, natural fibers can be utilized to create composites for high-tech products, such as automobiles, aircraft, and boat hulls (**Figure 2.1**). Natural fiber composites are superior to synthetic fiber composites, because natural fiber composites have better thermal insulation, result in less skin irritation, and are low in density. Moreover, unlike synthetic fibers, natural fibers are ecofriendly because they can be degraded by bacteria once they are no longer in use. Plant-based fibers such as flax, jute, sisal, hemp, and kenaf have been frequently used in the manufacturing of biocomposites and are already utilized in biomedical applications such as drug/gene delivery, tissue engineering, orthopedics, and cosmetic orthodontics (Frank et al., 2011; Heng et al., 2007; Rajesh & Pitchaimani, 2017; Azizi et al., 2005).



Figure 2.1: Applications of biocomposites (Mishra, 2020)

2.1.2 Types of natural fibers: Cotton fibers produce soft, lightweight fabrics with various sizes. People living in hot and humid climates often prefer cotton fabrics over fabrics composed of synthetic fibers because natural fibers are good sweat absorbents (Frank et al., 2011; Heng et al., 2007; Rajesh & Pitchaimani, 2017; Azizi et al., 2005). Natural fibers can exhibit different textures. Plants fiber are primarily composed of cellulose, which is most commonly used to make paper and cloth (Namvar et al., 2014).

There are two primary types of natural fibers: animal fibers and plant fibers. Mineral fibers includes asbestos and brucite, represent another type of natural fiber (Aplus Topper, 2019) (**Figure 2.2**).

i. Animal fiber: Animal fibers include angora, sinew, silk, mohair, catgut, wool, and alpaca. These fibers are primarily comprised of proteins such as fibroin, keratin, and collagen (Meyers et al., 2008).

ii. Plant fiber: Plant fibers includes the following:

a) Seed fiber: Fibers obtained from plant seeds are called seed fibers.

b) Leaf fiber: Fibers acquired from leaves are known as leaf fibers. Examples include banana (Fuqua et al., 2012) and pineapple (Todkar & Patil, 2019) fiber.

c) Bast fiber: Fibers isolated from the outer bark of a plant's stem are called bast fibers; these fibers includes flax, jute, kenaf, industrial hemp, ramie, rattan, and vine fibers. These fibers are generally used for durable yarn, fabric, packaging, and paper (Summerscales et al., 2010).

d) Fruit fiber: Fruit fibers, such as coconut fiber (coir), are isolated from fruit.

e) Stalk fiber: Stalk fibers are collected from plant stalks, e.g., from stalks of wheat, rice, barley, bamboo, and straw (Fuqua et al., 2012).

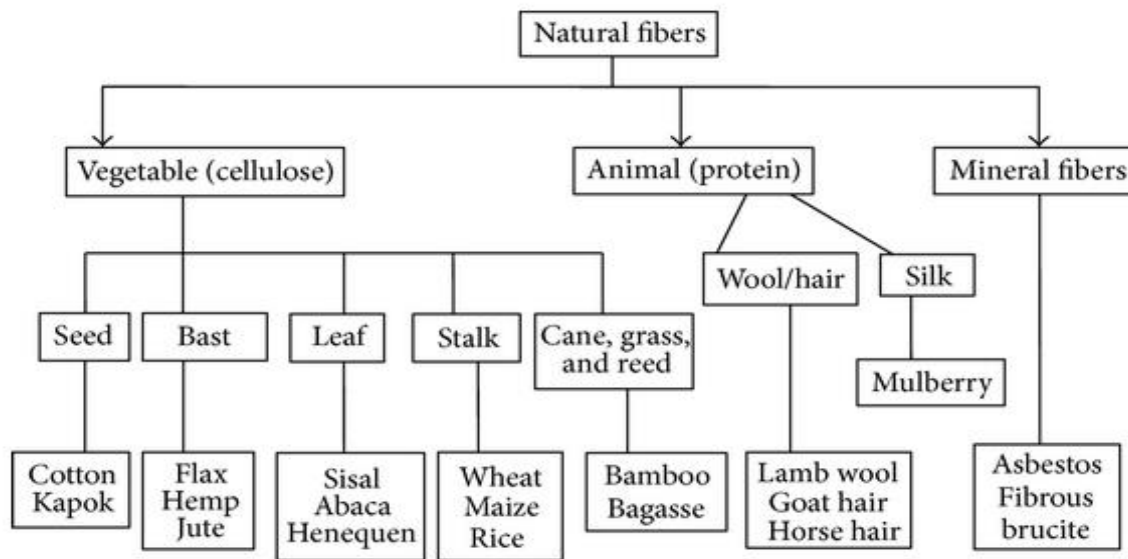


Figure 2.2: Types of natural fibers with examples (Aplus Topper, 2019)

2.2 Plant bast fibers: Scientifically, the term bast is identical to phloem, which is the nutrient-conducting tissue of vascular plants. Bast fibers exist as bundles consisting of elongated thick-walled cells joined together both side by side and end to end. These fibers are arranged in bundles along the length of the stem. Bast fibers are obtained from the stems of various dicotyledonous plants with two

cotyledons. These fibers are more flexible than leaf fibers. The process in which the bast fiber bundles are removed from the parent material is called the decorticating process, which removes the bast and outer bark from the stem. The separated individual fibers are obtained by being washed in water and then dried (Mwaikambo, 2006).

2.2.1 General structure of a bast fiber: Bast fibers have several cell walls composed of oriented semicrystalline cellulose microfibrils that exhibit varying arrangements surrounded by a hemicellulose–lignin matrix. The cell walls are categorized as primary and secondary cell walls. The primary cell wall is the outer wall, which is closely packed with loose irregular networks of cellulose microfibrils. The secondary wall comprises three segments: the outer layer (S1), the middle layer (S2), and the inner layer (S3). The middle layer (S2) determines the mechanical properties of the fiber (Thomas et al., 2011) (**Figure 2.3**).

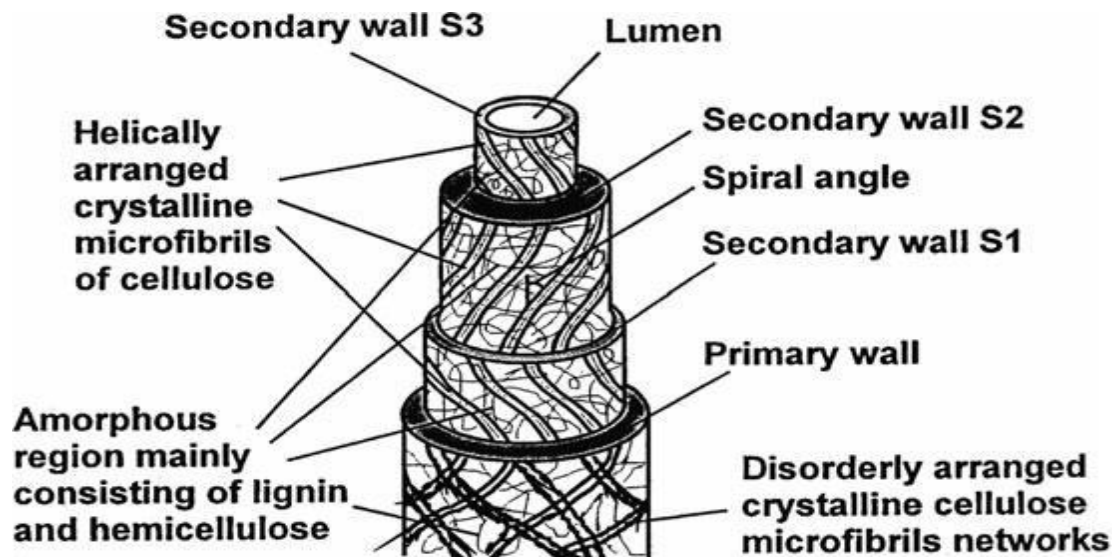


Figure 2.3: General structure of a plant stem or stalk fiber (Thomas et al., 2011)

2.2.2 Mechanical properties of bast fibers: Tensile behavior is an important mechanical property of plant fibers that determines fiber applications. For example, fibers with a higher tensile strength and lower twisting capacity are primarily used for biocomposite production (Rahman et al., 2013;

Fangueiro & Rana, 2018), whereas fibers with low tensile strength and high twisting capacity are used for apparel or textile applications (Beher, 2005).

Most mechanical properties have been well documented for natural fibers in recent studies. Perhaps the most extensive study on the mechanical properties of hemp fibers was reported by Prasad and Sain (2003), who used hemp fibers with diameters ranging from 4 μm to 800 μm for tensile testing (Prasad & Sain, 2003). The mechanical properties were found to depend on the fiber diameter, with the property values decreasing with increasing fiber diameter. This trend is consistent with general observations for synthetic fibers, where the amount of flaws decreases as the fiber diameter decreases, thus improving the tensile/mechanical properties of the fiber. The mean tensile strength and modulus values were 4200 MPa and 180 GPa, respectively, for 4- μm -diameter fibers. For fibers with a diameter of 66 μm , these values decreased to 250 MPa and 11 GPa, respectively. For 800- μm -diameter fibers, the values decreased to 10 MPa for tensile strength and 2 GPa for tensile modulus (Shahzad, 2013). **Table 2.1** shows the mechanical properties of different plant fibers.

2.3 Canola plant varieties or cultivars: Canola is a modified form of rapeseed or brown mustard, obtained via traditional plant breeding methods from three species:

- 1) Polish canola or *Brassica rapa*,
- 2) Argentine canola or *Brassica napus*, and
- 3) Canola-quality brown mustard or *Brassica juncea*.

Different types of canola plant varieties or cultivars include Golden, Arlo, HYHEAR 1, HYHEAR 3, Nugget, Tanka, Echo, Target, Polar, Turret, and Arid (Canola Council of Canada, 2020). Among others, HYHEAR 1 and HYHEAR 3 are cultivars of *Brassica napus*.

Table 2.1: Mechanical properties of major plant fibers (Müssig et al., 2010)

Fiber type	Mechanical properties			
	Density	Tensile strength	Young's modulus	Extension at break
Units	g/cm ³	MPa	GPa	%
Cotton	1.54	400	4.8	4-8%
Flax	1.27-1.55	500-900	50-70	2.70-3.6
Jute	1.30-1.45	300-700	20-50	1.69-1.83
Kenaf	0.15-0.55	295-955	23.1-27.1	1.56-1.78
Sisal	1.45-1.50	300-500	10-30	4.10-4.3
Abaca	1.42-1.50	879-980	38-45	9-11
Kapok	0.68-1.47	80.3-111.5	4.56-5.12	1.20-1.75
Coconut (Coir)	0.67-1.15	173.5-175	4.0-6.0	27.21-32.32
Oil Palm	0.7-1.55	227.5-278.4	2.7-3.2	2.13-5.00
Sugarcane	0.31-1.25	257.3-290.5	15-18	6.20-8.2
Banana	0.65-1.36	51.6-55.2	3.00-3.78	1.21-3.55
Corn Stalks	0.21-0.38	33.40-34.80	4.10-4.50	1.90-2.30
Pineapple	1.25-1.60	166-175	5.51-6.76	2.78-3.34
Rice Straw	0.86-0.87	435-450	24.67-26.33	2.11-2.25

NB: g/cm³ = Gram/Centimeter³, % = Percent, MPa = Mega Pascal, GPa = Giga Pascal

2.3.1 History of canola: The earliest evidence of humans using fibers dates back to 36,000 BP, with the discovery of dyed flax fibers and wool (Balter, 2009). In the early 1970s, Keith Downey and Baldur R. Stefansson were the first to breed canola from rapeseed cultivars of *Brassica napus* and *Brassica rapa* at the University of Manitoba, Canada (Downey, 2007). The oil of this new cultivar has much less erucic acid (Barthet, 2015). Canola was originally a trademark name of the Rapeseed Association of Canada, a combination of "Can" from Canada and "ola", meaning "oil, low erucic acid" (Wrigley et al., 2016). However, the term 'canola' is now used as a generic term for edible varieties of rapeseed oil in North America and Australia (AAFC, 2016). The name

distinguishes canola from the natural rapeseed oil, which has a much higher erucic acid content. Although wild rapeseed oil contains high levels of erucic acid (Sahasrabudhe, 1977), cultivars are used to produce human-consumable commercial canola oil containing less than 2% erucic acid (CFR - Code of Federal Regulations Title 21 2010). This level of erucic acid is not considered as a health risk and results in a milder taste (Knutsen et al., 2016; WHO, 1974). Thus far, no cases of severe detrimental health issues have been reported in association with dietary consumption of erucic acid by humans (Stöckler et al., 1997). However, significant amounts of erucic acid may be poisonous in other species, as implied by erucic acid metabolism tests (Hayes, 2008).

2.3.2 Canola production in 2018/19: In Canada, the canola production in year 2018/19 was 20.3 million metric tons (MMT), representing a decrease of 5% from the previous year. This decrease was attributed to prolonged periods of dryness throughout July and August and a reduction in cultivation area from 9.3 million hectares to 9.2 million hectares (1%). The western provinces of Canada, such as Manitoba, Saskatchewan, and Alberta, are the highest canola producers (**Table 2.2**). However, small amounts of canola are grown in Ontario and the southernmost reaches of Quebec. Although yields were lower overall in Canada, Manitoba canola production rose 5% in year 2018/19 to 3.3 MMT, with an 8% growth in cultivation land. The best canola yields (2.72–3.26 MT/ha (metric tones/hectare)) came from the Parkland region, between the western shores of Lake Manitoba and the Saskatchewan border. Reportedly, farmers that managed to harvest before early fall rain and snowfall averaged approximately 2.72 MT/ha, while those who harvested afterward had yields of approximately 2.43 MT/ha (Danielson, 2019).

Table 2.2: Canola production total and by province (metric tons) in Canada (Danielson, 2019)

	2016	2017	2018	% Change from 2017/18
Canada	19,599,200	21,328,000	20,342,500	-5%
Manitoba	2,608,200	3,147,900	3,318,400	5%
Saskatchewan	10,682,100	11,181,100	10,927,100	-2%
Alberta	6,157,500	6,826,600	5,870,600	-14%

2.3.3 Canola fibers: *Brassica napus* (commonly known as canola) is widely cultivated throughout the world and is used as a major oilseed crop for vegetable oils and biodiesel production (González et al., 2013; Yousefi, 2009). After oil production, the remaining plant material goes unused and is returned to the soil for decomposition (waste biomass) or utilized to feed animal stock. Thus, several recent studies have investigated the use of this bast fiber for biocomposite production as well as textile applications. Among these studies, researchers have characterized the canola biomass (fibers) of four cultivars (Shuvo et al., 2019) and determined the wicking properties of canola biomass for threeCanola Encyclopedia of the canola plant (Soriano & Rahman, 2017). Researchers have also optimized the retting time and temperature and have treated canola fiber with alkali, softener, and enzymes for textile applications (Khan, 2016). Thus, at this point, it is important to determine the chemical composition of canola fiber in comparison with cotton and jute (ideal fiber for textile industries). Jute is considered the second most important natural bast fiber behind cotton (seed fiber). Jute is primarily used to manufacture cloth, bags and coarse fabrics. Besides being woven into curtains, chair coverings, carpets, area rugs, hessian cloth, backing for linoleum, and paper, these fibers are also used in rural road constructions. With low density, less abrasive behavior, and good dimensional stability, jute fibers have superior durability and moisture-retention capacity (Mia et al., 2017; Anwar, 2007).

The percentages of the dominant chemicals in cotton, jute, and canola fibers are given in **Table 2.3**. Considering the types and composition of canola fiber, it is reasonable to find that canola is a lignocellulosic bast fiber (Tofanica et al., 2011) because the lignin content (wood fiber content) is much higher and cellulose content is much lower in canola fibers than in cotton and jute fibers, indication that canola fibers will likely be less flexible than cotton and jute.

Table 2.3: Dominant chemical components of canola, jute, and cotton fiber (percentage based on optical density) (Kiaei et al., 2014; Kozasowski et al., 2012; Macmillan & Birke, 2016)

Component	Cotton fiber	Jute fiber	Canola fiber
	%	%	%
Cellulose	88-96	61-75.5	44
Lignin	0.4-1	12-13	19.21

Table 2.4: Chemical composition of different plant fibers, including cotton, jute, and canola

Fiber	Chemical composition						References
	Cellulose	Hemicellulose	Pectin	Lignin	Wax	Ash	
	%	%	%	%	%	%	
Cotton	88-96	5.7	<1.0	0.4-1	0.6	---	(Kiaei et al., 2014; M, 2012; Macmillan & Birke, 2016)
Canola	44	--	--	19.21	--	13	(Kiaei et al., 2014; Macmillan & Birke, 2016)
Jute	61-75.5	13.6-20.4	0.2	12-13	0.5	---	(Kozasowski et al., 2012)
Flax	71-75.2	8.6-20.6	2.3	2.2-4.8	1.7	1.1	(Kozasowski et al., 2012)
Ramie	68-76.2	13-16.7	1.9	<0.7	1.7	1.1	(Nayak et al., 2012)
Hemp	70-75.1	<2.0 – 22.4	0.9	3.5-8.0	0.8	3.5	(Kozasowski et al., 2012)
Sisal	47.6-78	10-17.8	10	10-14	2	4.5	(Nayak et al., 2012)
Abaca	56-63.7	17.5	1.0	15.1	---	1.1	(Kozasowski et al., 2012)
Kenaf	45-57	21.5	3.0-5.0	8.0-13.0	---	---	(Nayak et al., 2012)
Cattail	37-63	8.7-21	4.0-23.90	5.6-13	1.4	2-12.56	(Sridach, 2014)

The chemical composition and morphological microstructure of a plant fiber are extremely complex due to the hierarchical organization of different compounds in various compositions. **Table 2.4**

compares the chemical composition of seed fiber (cotton), bast fiber (jute, canola, flax, hemp), and leaf fiber (sisal, abaca). The core component of all fibers is cellulose.

Among the fibers considered, cotton has the highest cellulosic and lowest lignin content and thus exhibits highest flexibility and twisting capacity. Similarly, jute contains less cellulose than cotton but more than canola and contains more lignin than cotton but less than canola showing less flexibility than cotton but more flexibility than canola (**Table 2.4**).

2.3.4 Growth stages of the canola plant: As growth and development are continuous processes for the canola plant, the roots grow deeper as the stems elongate. Depending on the date of seeding and growth conditions, the vegetative stages can range from 40 to 60 days in *Brassica napus* from seeding to first flower. Different growth factors, such as variety, moisture, seeding date, soil fertility, and plant population, influence the stem diameter and height (Canola Encyclopedia, 2020). Considering the above facts, a mature canola plant has stems of varying diameter, including thicker (over-matured), thinner (less matured), and medium (matured) stems. It is reasonable to assume that thinner stem fibers may have higher cellulose and lower lignin contents than thicker stem fibers and are hence softer or more flexible. However, thus far, no data have been available to address this query. Moreover, no studies have been conducted to assess the quality of extracted canola fibers with respect to different stem diameters.

Although the growth and development of canola plants are continuous, these processes can be divided into easily recognizable growth stages. The duration of each growth stage is greatly influenced by light (day length), temperature, nutrition, moisture, and soil variety. A standardized growth stage scale called the BBCH (Biologische Bundesanstalt, Bundessortenamt, and Chemical Industry) decimal system was developed by BASF (Badische Anilin und Soda Fabrik), Bayer, Ciba-Geigy, and Hoechst;

this scale provides an accurate and simplified approach for describing canola growth stages (Canola Encyclopedia, 2020). The BBCH decimal system is illustrated in **Figure 2.4**.

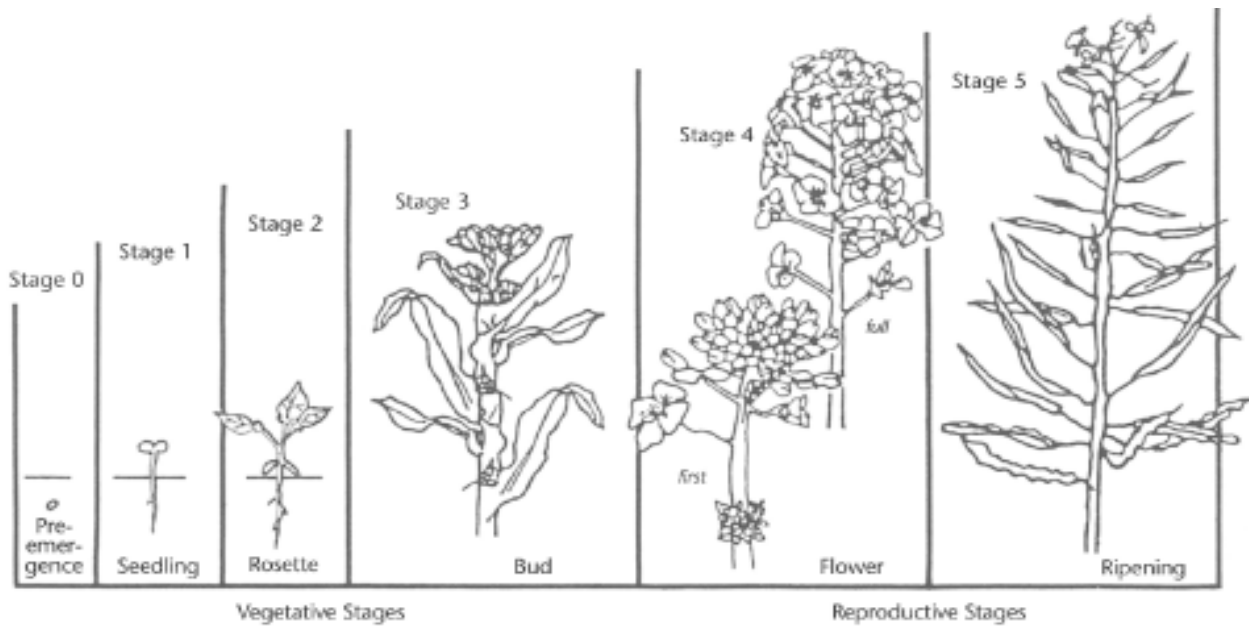


Figure 2.4: Different growth stages of the canola plant (Canola Encyclopedia, 2020)

In addition to leaves, the stems of the canola plant also function as significant photosynthetic structures throughout the growth of the pod and seed. Leaf development is overlapped by stem elongation. Stem elongation occurs simultaneously with leaf development. Just prior to stem elongation, branch initiation and flowering begin. During flowering, the main stem length reaches its maximum length. As the root system deepens, the stem will continue to grow. Depending on the time of seeding and growth conditions, the vegetative stages (from seeding to first flower) can range from 30 to 50 days in *Brassica rapa* and 40 to 60 days in *Brassica napus*. On average, *Brassica napus* plants grow taller (75–175 cm, 30–70") than *Brassica rapa* plants (50–125 cm, 20–50") (Growth stages, 2020).

Plants with thicker stems are low-density crops and are resistant to lodging. In contrast, plants with thinner stems are high-density crops and are prone to lodging, which exacerbates the issues of uneven

pod maturity and creates an ideal environment for the spread of diseases such as alternaria and sclerotinia. The photosynthetic capacity of the stems and pods is significantly decreased by infection, reducing yields (Growth stages, 2020).

2.3.5 Scanning electron microscopy (SEM) imaging of the canola fiber bundle: Figures 2.5 and 2.6 show cross-sectional images of a canola fiber bundle after being isolating from the stem exterior. The hollow structure of the fibers is also visible in a cross-sectional image of the fiber bundle. **Figure 2.6** shows images of fibers with diameters ranging from 7.99 to 23.23 μm (Shuvo et al., 2019). The fibers are most likely attached to each other via the hemicellulose–lignin matrix (Thomas et al., 2011).

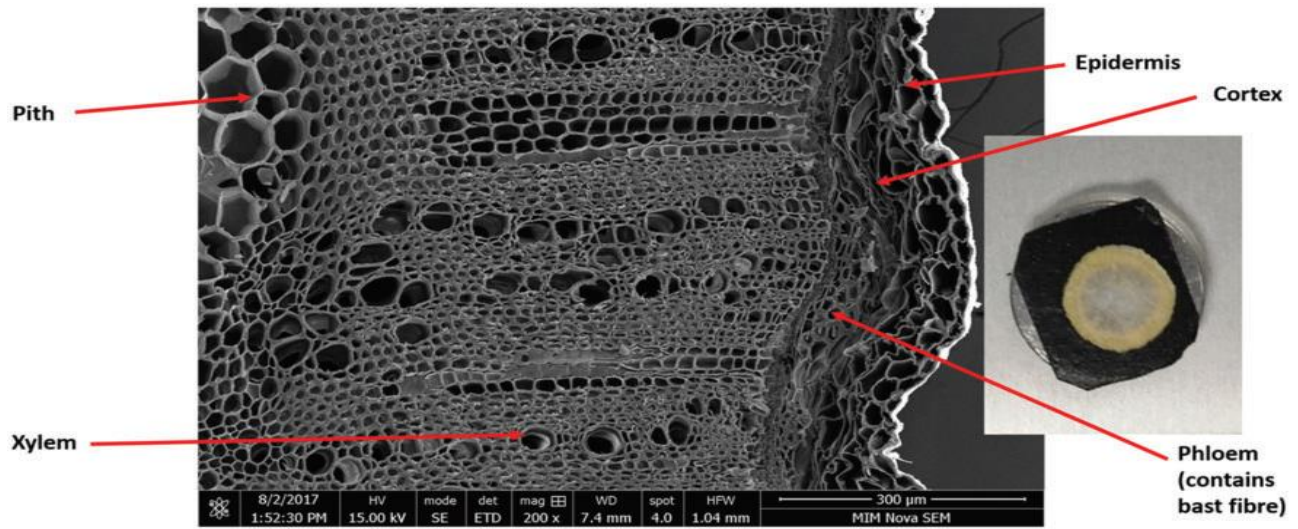


Figure 2.5: Cross-sectional view of a canola fiber bundle after extraction (Shuvo et al. 2019)

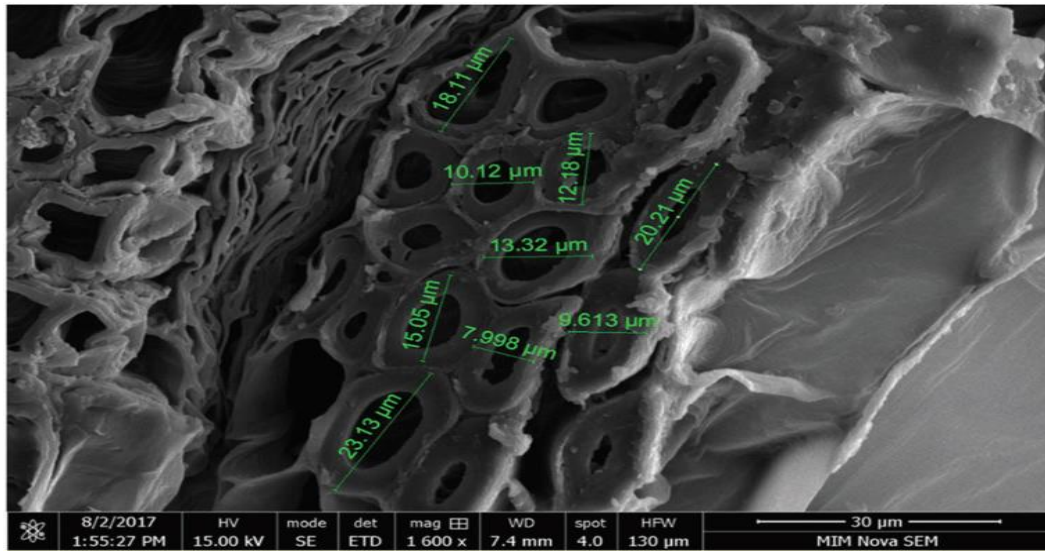


Figure 2.6: Cross-sectional view of a canola fiber bundle showing fibers of different sizes and shapes (Shuvo et al. 2019)

2.3.5.1 SEM images of a single canola fiber: Longitudinal SEM images of the canola fiber surface show a primarily smooth surface with no unusual protrusions or roughness, in contrast to observations of wool cotton twist (**Figure 2.7**). A hollow structure surrounded by thick layers, which most likely consist of the hemicellulose–lignin matrix (**Figure 2.8**) (Thomas et al., 2011), is observed in the canola fiber cross section. The width of the thick outer layer is approximately 15–20 μm (**Figure 2.6 and 2.8**). The shape of the hollow region, also known as the lumen, is polygonal (**Figure 2.5**) and ranges from circular to a thin canal. The the diameter of the hollow region is approximately 5–35 μm and is considered to be responsible for the low density of the canola fiber (Shuvo et al., 2019).

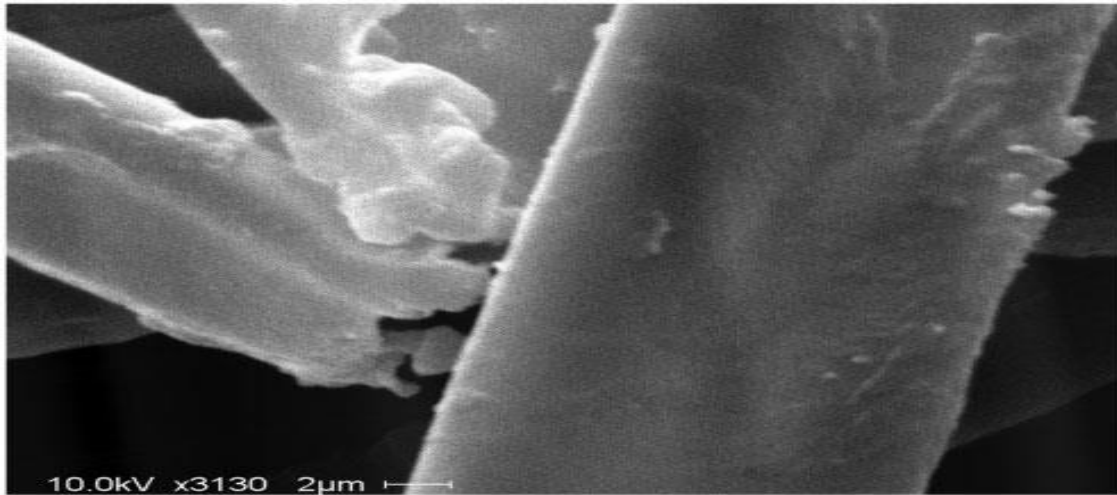


Figure 2.7: Longitudinal view of canola fibers (Shuvo et al., 2019)

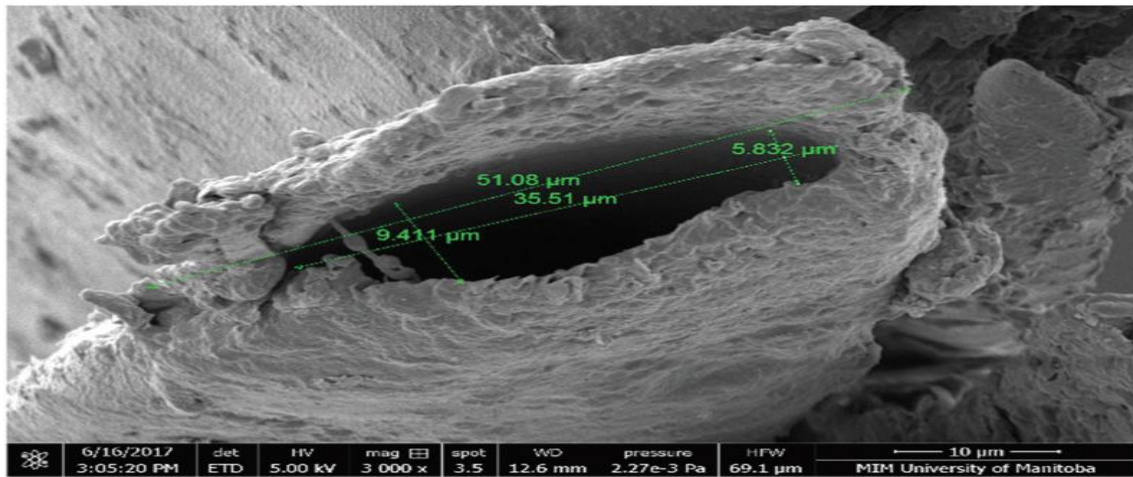


Figure 2.8: Cross-sectional view of a canola fiber (Shuvo et al., 2019)

2.3.5.2 Effect of stem diameter on the mechanical properties of canola fibers: Numerous studies have been performed to investigate the mechanical properties of canola fibers (Khan, 2016; Shuvo et al., 2019; Soriano & Rahman, 2017; Shuvo et al., 2018). However, there are currently no available data regarding the effect of stem diameter on the mechanical properties of canola fibers.

Some studies have been reported for flax and hemp fibers, where fine flax fiber exhibits superior tensile and flexural properties. The optimum fiber diameter corresponds to that of fine fiber (19.3 μm) for developed biocomposites. Fine flax fiber-reinforced biocomposites exhibit the highest hardness,

resulting in better mechanical properties (Rahman et al., 2013). Alcock et al. (2018) studied flax fibers with varying stem diameters and found a statistically significant positive correlation with fiber diameter and a negative correlation with tensile strength. However, no correlations for tensile strength, Young's modulus, or fiber diameter were found in samples with the same stem diameter range for plants grown in different locations or of different varieties. Prasad and Sain (2003) applied tensile testing to hemp fibers with diameters of 4–800 μm . The tensile properties were found to be clearly dependent on the fiber diameter, gradually decreasing with increasing fiber diameter. This trend has also been observed for hemp fibers, as reported by Shahzad (2013).

Based on this research, the current study aims to investigate the effects of stem diameter on the mechanical properties of canola fibers collected from three types of stems based on diameter, including narrow (immature), medium (mature), and wide (over-mature) stems. The maturity of plant stems at different growth stages differs, and hence, the chemical composition (cellulose to lignin ratio) may also differ. In this study, we focus on a single type of canola fiber collected from a single cultivar (HYREAR 3), species (*Brassica napus*), time, and location.

2.4 Surface modification of fibers: Surface modification can reduce moisture absorption and enhance fiber–matrix interfacial bonding, wettability, roughness, and hydrophilic features, which can improve the tensile properties of fiber cementitious composites (Ku et al., 2011). Surface modifications can also be applied to alter the tensile properties for textile applications. Fiber modification methods can be divided into the following four groups.

- (a) Physical treatments to improve fiber properties, such as strength, modulus, and elongation;
- (b) Chemical treatments to alter the interfacial properties of the fiber matrix and the durability of the fiber in textile- and cement-based composites, respectively (Cruz & Figueiro, 2016);

(c) Physicochemical treatments to provide clean and fine photofibrils or fibrils with a very high cellulose content (Akil et al., 2015; Fuqua et al., 2012; John & Anandjiwala, 2008); and

(d) Biological treatments to improve the interfacial adhesion for composite production (Pommet et al., 2008).

(a) **Physical methods:** Physical techniques, such as plasma treatment (**Figure 2.9**), have been successfully utilized to modify the surface of various natural fibers. This treatment significantly improves the mechanical properties of natural fibers (Oliveira et al., 2012). It generally introduces various functional groups on the surface of natural fibers, and these functional groups form strong covalent bonds with the matrix, leading to a strong fiber–matrix interface. Through this technique, the fiber surface is modified to accept either a bond or an ink for printing, resulting in a fiber that is hydrophilic (wetable). Additionally, plasma treatment applied via surface etching may improve the surface roughness through mechanical interlocking, which results in a better interface with the matrix (Shahidi et al., 2015).

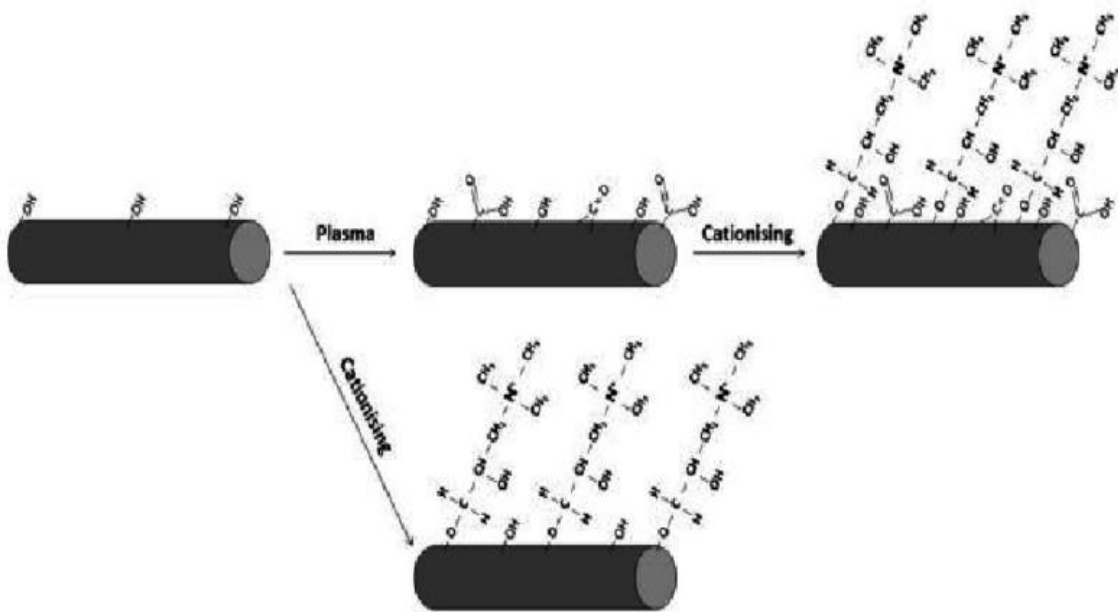


Figure 2.9: Effects of plasma and cationizing processes (Shahidi et al., 2015)

(b) Chemical methods: Natural fibers can be treated with various chemicals, such as alkali, water-repelling agents, softeners, silane, peroxides, and permanganates. The textile roughness can be reduced by some of these chemicals, such as softeners. In addition, the mechanical properties of natural fibers can be significantly improved by other chemicals (such as alkali treatment, as shown in **Figure 2.10**) by modifying their crystalline structure and removing weak components such as hemicellulose and lignin from the fiber structure (X. Li et al., 2007). Some selective chemicals (e.g., water-repelling agents) can reduce moisture absorption and subsequent swelling of natural fibers. Moreover, fiber–matrix interfacial interactions can be induced through the formation of strong chemical bonding by some chemical treatments (for example, with silane coupling agents), substantially improving the mechanical performance of composites (Xie et al., 2010).

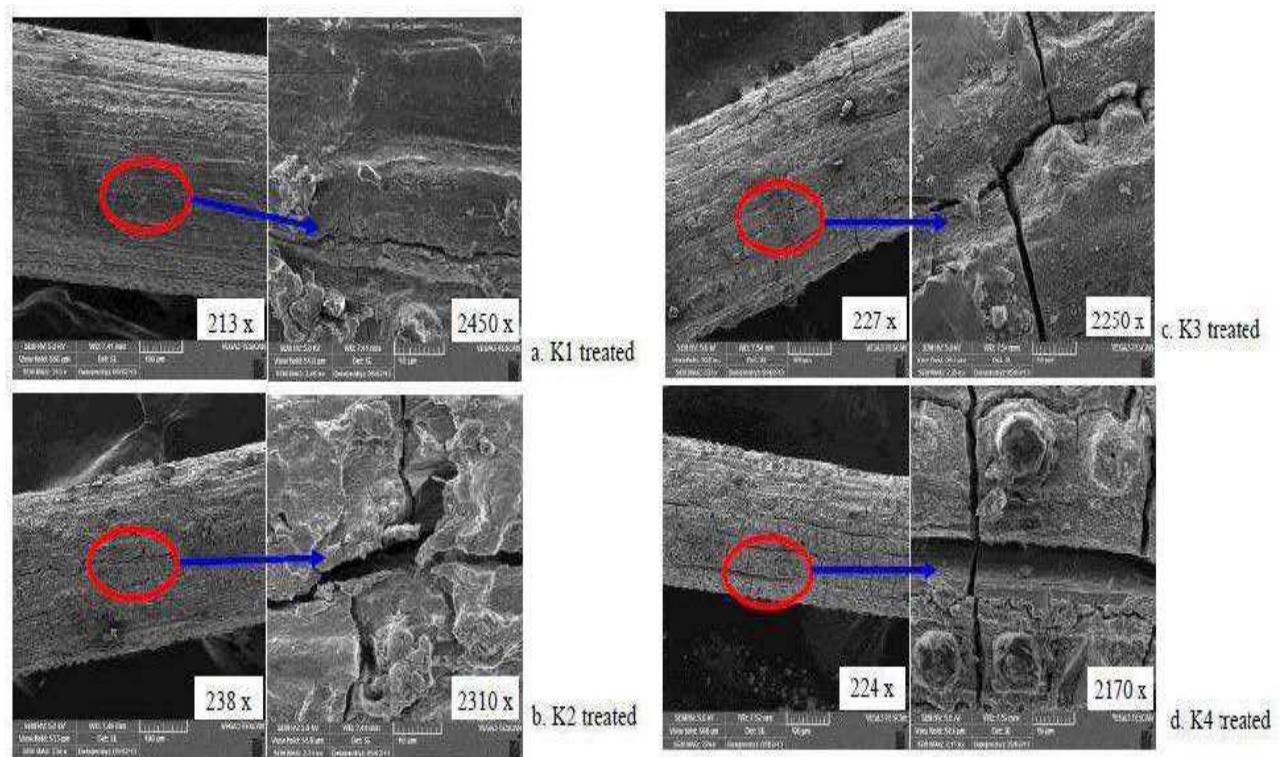


Figure 2.10: Surface morphology of coconut fibers treated with alkali (Arsyad et al., 2015)

(c) Physicochemical methods: The combination of physical and chemical treatments is known as physicochemical treatment, which provides support by improving the separation of fiber bundles and

introducing chemical reactions (Senthamaraikannan et al., 2019). These types of treatments provide fine and clean natural fibers or fibrils with high cellulose content. The mechanical properties of these fine fibers are close to those of pure cellulosic fibers such as cotton, significantly improving the appearance of the plant fiber (Fuqua et al., 2012).

(d) Biological methods: In addition to physical and chemical methods, biological processes can also modify the surface of natural fibers. In a recent study, during the fermentation process of bacterial cellulose, cellulosic nanofibrils were deposited on the surface of sisal and hemp fibers as substrates (**Figure 2.11**). A significant improvement in interfacial adhesion was found for polymeric matrices, such as polylactic acid and cellulose acetate butyrate, when approximately 5%–6% bacterial cellulose was deposited on the natural fiber surface. Therefore, a new generation of natural fiber composites with an improved fiber–matrix interface was developed by this novel surface modification process (Pommet et al., 2008).

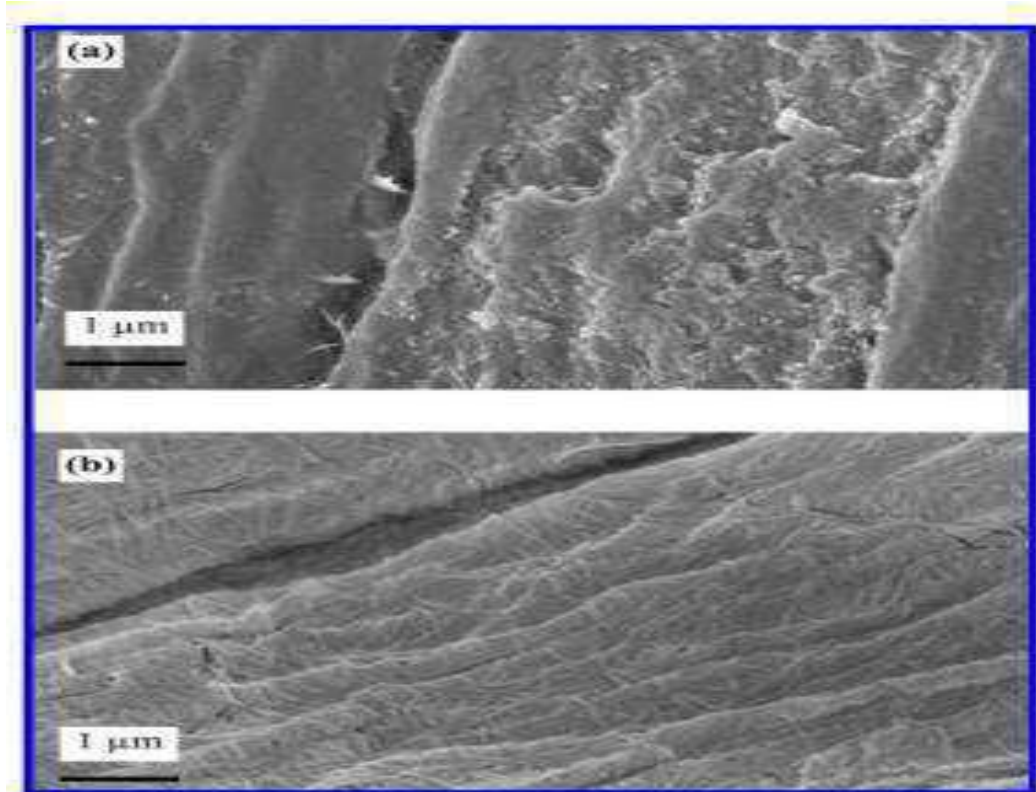


Figure 2.11: (a) Wild hemp fiber and (b) biologically treated hemp fiber (Pommet et al., 2008)

2.4.1 Fabric softener: A fabric is a cloth composed of fibers and yarns knitted together, with a substantial surface area in relation to its thickness and adequate cohesion to yield a desirable mechanical strength. Fabric softener plays an important role in improving the softness of the fabric and influences various fabric properties such as its shade appearance (Needles, 1986). Softener is applied to almost all cloths and home furnishing textiles because most buyers and users consider the hand of a fabric to be one of the most important properties (Schindler & Hauser, 2004; Spencer, 2001).

2.4.2 Purposes of fabric softener: Fabric softener serves two primary purposes.

- (1) Fabric softener makes clothing softer and more comfortable for wearing.
- (2) If used during laundering, fabric softener reduces static cling from synthetic clothing, as reported in the article “Laundry and Cleaning” at ConsumerReports.org (Lebednik, 2020).
- (3) In general, as the number of fibers per centimeter increases in a fabric, the softness of the fabric decreases, thus increasing the fabric stiffness. Softener is used to enhance a fabric's softness while considering the composition and characteristics of its substrate (Needles, 1986).
- (4) Fabric softeners prevent electrostatic charge by adding antistatic properties to the fabrics that build up on the substrates. In this manner, the softener eliminates fabric cling, crackling noises, dust attraction, handling, and wearing.
- (5) Fabric softeners reduce wrinkles in garments, making them easier to iron and saving energy by decreasing the drying time.
- (6) Fabric softener can also add a pleasant fragrance to the fabric (Smulders et al., 2007).

2.4.3 Mechanism of action of fabric softener: Mechanical stress on textiles produced from natural fibers such as cotton and wool is greatly influenced by machine washing. The fabric texture is hardened by squeezing and fraying of the fabric surface; moreover, the fabric becomes tighter after

drying in the air. If fabric softener is added at the final rinse of washing and the fabric is then dried, the surface of the fabric feels softer. Basically, the electrically charged chemical compounds of the fabric softeners coat the surface of the fabric. As a result, the treads from the fabric surface stand up, making a softer and fluffier texture (Smulders et al., 2007).

2.4.4 Types of fabric softeners: The specific softness value of each fiber generally depends on its chemical composition and physical structure. There is a direct relationship between the fineness of a fiber and the softness of a fabric. The fabric twist ration is inversely proportional to its softness. Both hydrophobic- and hydrophilic-containing molecules are present in almost all softeners. Therefore, softening agents are classified according to their chemical properties, including (1) cationic softener, (2) anionic softener, (3) non-ionic softener, (4) amphoteric softener, (5) silicone softener, and (6) reactive softener (Hossain et al., 2019). In addition, fabric softeners also include emulsion stabilizers, acids or bases to maintain an optimal pH for absorption, fragrance enhancers, and coloring agents (Schindler & Hauser, 2004) (**Figure 2.12**).

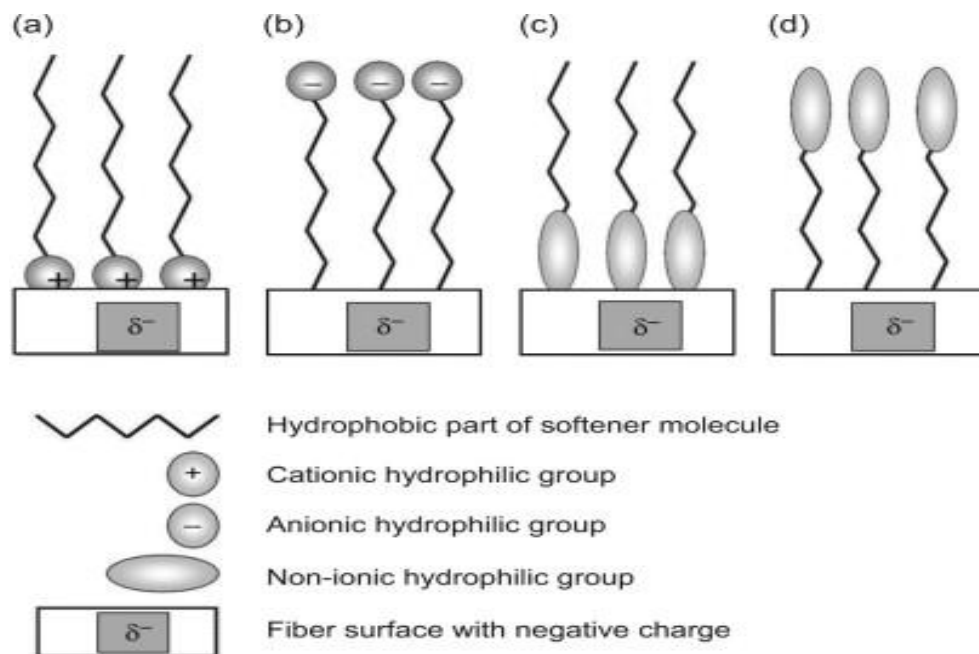


Figure 2.12: Different types of fabric softeners and their mechanism of adherence to the fabric surface (Behar, 2005)

(1) Cationic softeners: Cationic softeners more easily bind to the surface of natural fibers (wool, cotton) than to synthetic fibers. These softeners bind to negatively charged groups on the fiber surface via electrostatic attraction and neutralize their charge. Due to this property, cationic softeners are also considered as molecular finishing agents. The long aliphatic chains of the uncharged groups of the fabric line up toward the outside of the fiber, imparting lubricity. Cationic softeners usually contain three different groups, including quaternary ammonium salts, amino-esters, and amino-amides. These softeners can be applied to any type of fiber. Cationic softeners have a pH of 5–6 and are suitable for exhaustion processes in acidic conditions (pH 4–5) (Schindler & Hauser, 2004).

(2) Anionic softeners: Anionic softeners are stable at normal textile processing temperatures and are compatible with other components of dye and bleach baths (Imtiazuddin, 2009); however, these softeners are incompatible with cationic softeners in detergents because they combine with them to form a solid precipitate (Schindler & Hauser, 2004). The anionic groups are oriented outward and are surrounded by a thick hydration layer; thus, these groups can be easily washed off, providing strong antistatic effects with good rewetting properties (Imtiazuddin, 2009).

(3) Non-ionic softeners based on paraffin and polyethylene: Under normal textile processing conditions, non-ionic softeners are stable to extreme pH and heat and are compatible with most textile chemicals. When performed in the presence of alkali, emulsification will provide a higher quality and more stable products with high lubricity and low durability during dry cleaning. Polyethylene can be modified by air oxidation and melts at high pressure, adding a hydrophilic character, such as that of a carboxylic acid group (Imtiazuddin, 2009).

(4) Amphoteric softener: These softeners have fewer ecological problems (readily biodegradable) than similar cationic products while providing softening effects, low permanence to washing, and high antistatic effects (Imtiazuddin, 2009).

(5) Silicone softeners: These softeners exhibit good temperature stability and durability with a high degree of permanence for products that form cross-linked films and display a range of properties from hydrophobic to hydrophilic. Silicone softeners also provide good sewability, high lubricity, crease recovery, elastic resilience, tear strength, and abrasion resistance (Imtiazuddin, 2009).

2.4.5 Economic significance of fabric softeners: When examining a textile, people generally touch the textile in an automatic manner, using their hands to assess the fabric (Schindler & Hauser, 2004). Thus, considering the appeal to consumers, the use of softeners has diversified in recent years, with a focus on fragrance, deodorizing, and/or antibacterial qualities of the fabrics. The market is hence overflowing with a large number of products. Nonetheless, softeners still provide the primary function of softening clothes, a function that has remained unchanged over time (Igarashi & Nakamura, 2018).

2.4.6 Cepreton UN: In this study, we will use a commercially available softener (Cepreton UN) to examine the effect of softener on canola fibers extracted from stems of varying diameter. Cepreton UN is a cationic softener used to soften canola fibers. This softener is a concentrated cold water-soluble cationic softener pastille and is highly popular and widely used in the textile industry (Achroma Life Enhanced, 2020). We assume that this softener will improve the texture of canola fibers.

CHAPTER 3: MATERIALS AND METHODS

3.1 Collection of plant samples: *Brassica napus* plants (type: canola) (cultivar: HYREAR 3) were planted, grown in the field of Carman which was coordinated by the Department of Plant Science, University of Manitoba. After mechanically harvesting the seed, the plants were harvested in the mid of August, 2019. Following harvesting, plant samples were carried out to the textile laboratory located in the Department of Biosystems Engineering at the University of Manitoba. All the harvested samples were stored at approximately 25 ± 2 °C and 33 ± 2 % relative humidity prior to retting and tested following the experimental design of **Figure 3.2**.

3.2 Retting of plant stems: In this study, we did total six extractions and categorized the stems in three types based on 3 different diameter ranges and each range was separated by minimum 0.10 mm. The stem diameter ranges were categorized in 2 groups; in group-1, total 4 extractions were included, such as extraction-1 (E1), extraction-3 (E3), extraction-4 (E4), and extraction-6 (E6) whereas extraction-2 (E2) and extraction-5 (E5) were considered as group-2 (**Table 3.1**). Group-1 extractions have nearly similar diameter range and group-2 extractions also have almost similar diameter range. Each of the plant stem was cut by around 10 cm long and the diameters of the stems were measured by a Digital Slide Caliper (**Model: MASTERCRAFT, 58-6800-4**). We have measured the weight of each set of stems before retting. All those three categories of stems were then kept for retting in a water bath (**Model: HAAKE SWB 20, Germany**) at 40° C temperature for 48 hours. Samples were checked daily in order to find the end point of retting. There is no standards to determine the exact retting time, however, we checked the stems in every 24 hours and when we were able to peel easily epidermis by gently rubbing the surface of the stems, we stopped retting. Generally, it was also observed that 48 hours is the optimum period to collect maximum amount and good quality fibers (Khan, 2016). Over retting and under retting made the fibers mostly rotten or hard to peel, respectively. So, our retting period was approximately 48 ± 2 hours at 40° C. **Table 3.1** shows three

different diameter ranges of stems used in this study and **Figure 3.1** is representing the incubation of plant stems in the water bath.



Figure 3.1: Incubation of plant stems in the water bath for retting at 40°C for 48 hours

Table 3.1: Six extractions with three different types of stems based on different diameters

Extractions	Stems	Diameter ranges	Groups
		mm	
E1	Narrow	2.8-4.5	1
	Medium	4.8-7.8	
	Wide	8.0-10.0	
E2	Narrow	2.5-6.0	2
	Medium	7.0-9.0	
	Wide	9.5-12.5	
E3	Narrow	2.8-4.5	1
	Medium	4.8-7.8	
	Wide	8.0-10.0	
E4	Narrow	2.8-4.5	1
	Medium	4.8-7.8	
	Wide	8.0-10.0	
E5	Narrow	2.5-6.0	2
	Medium	7.0-9.0	
	Wide	9.5-12.5	
E6	Narrow	2.8-4.5	1
	Medium	4.8-7.8	
	Wide	8.0-10.0	

NB: E = Extraction

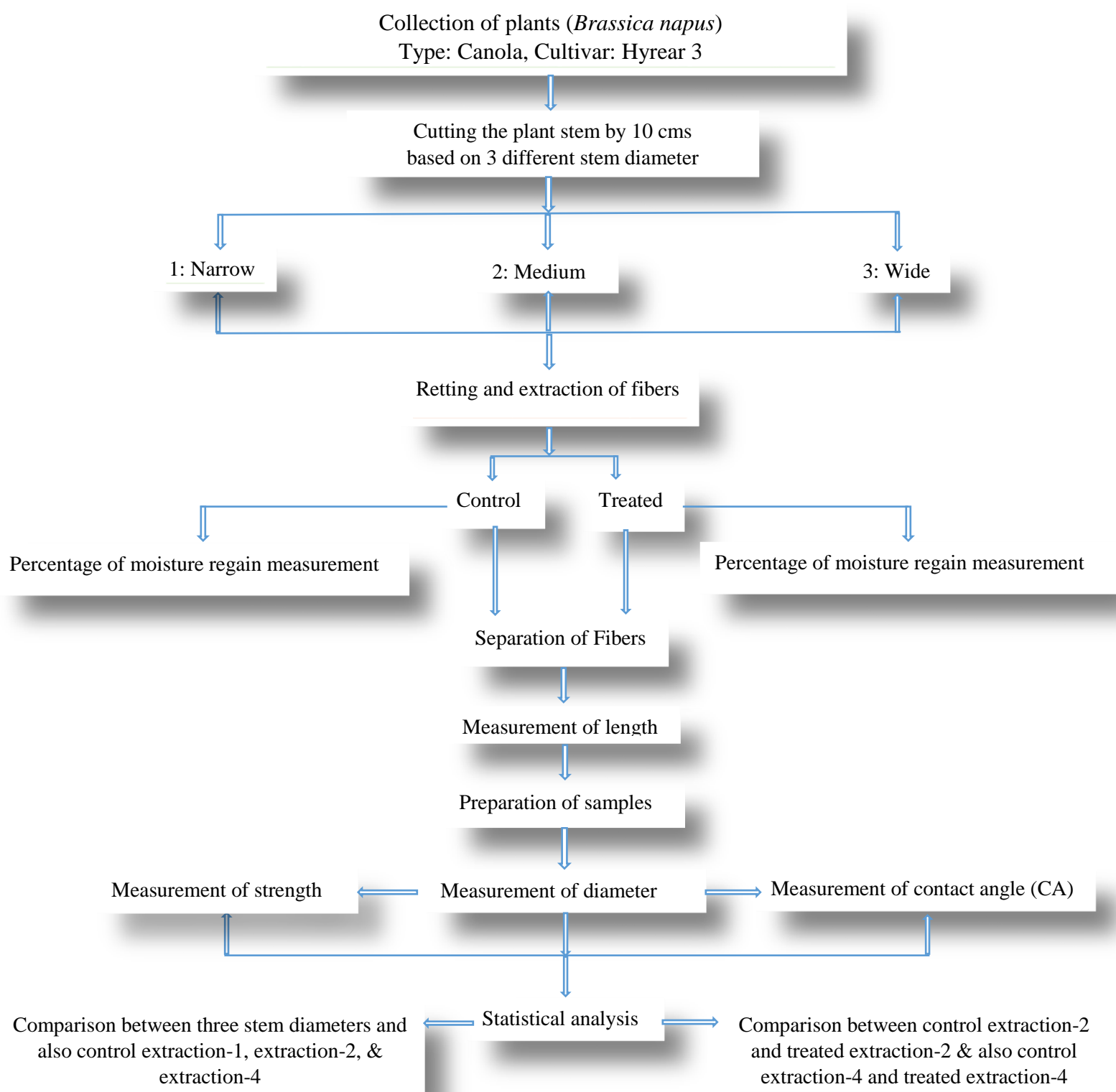


Figure 3.2: Experimental design

3.3 Extraction and separation of fibers:

3.3.1 Estimation of fiber yield (%): The fibers were isolated from the plant stems by rubbing the surface of the plant stems and then peeling the barks by hands after retting. The extracted fibers were then air dried in the lab for 72 hours. The temperature and the relative humidity (RH) of that lab were maintained nearly 25 ± 2 °C and 33 ± 2 %, respectively. The weight of the plant stems and the dried fibers was then measured using the following formula to measure the fiber yield (%) and stored in a small sealed plastic bag in that specific lab for further use (**Figure 3.3**).

Fiber yield (%) of the extracted fibers was calculated using the following formula:

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



Figure 3.3: Extracted and dried fibers storing in a small sealed plastic bag

3.3.2 Separation of fibers: The dried fibers were then soaked in distilled water for 10 minutes and individually separated from each other manually using a sharp needle and again air dried in lab (nearly 25 ± 2 °C temperature and 33 ± 2 % RH) for 72 hours. All the extracted and individually separated dried fibers were packed in a small sealed plastic bag and stored at the lab for further investigation (**Figure 3.4**). In total, six batches of fibers were extracted from 3 types of diameters (wide, medium, narrow) of stems and using same condition for each batch.



(a)



(b)

Figure 3.4: (a) Canola fiber separation from the stem (Shuvo et al. 2019) (b) Manually separated individual dried fibers storing in a small sealed plastic bag

3.3.2.1 Separated individual fibers for diameter and strength and contact angle measurements:

After separation, 50 individually isolated fibers were prepared for diameter and strength measurement and 30 fibers for contact angle measurement of each stem diameter of a single set. Hence, total 150 fibers from each set were taken for fiber diameter measurement and strength testing and 90 fibers were selected for contact angle measurement (E1 and E4). Another 20 fibers were taken from each set (E3 and E6) to compare and contrast between E1, E3, E4, and E6 (**Table 3.2**). The fibers were also compared and contrasted between group-1 (E1+E3+E4+E6) and group-2 (E2) fibers (**Table 3.3**).

Table 3.2: Sample numbers to measure diameter, strength and contact angle (control)

Group-1	E1		E3		E4		E6	
Stems	Fibers for D and S	Fibers for CA	Fibers for D and S	Fibers for CA	Fibers for D and S	Fibers for CA	Fibers for D and S	Fibers for CA
	n	n	n	n	n	n	n	n
Narrow	50	30	20	20	50	30	20	20
Medium	50	30	20	20	50	30	20	20
Wide	50	30	20	20	50	30	20	20
Total	150	90	60	60	150	90	60	60

N.B: E = Extraction, n = number of sample, D = Diameter, S = Strength, CA = Contact Angle

Table 3.3: Sample numbers to compare between group-1 (pooled) and group-2 fibers (E2)

Stems	E1+E3+E4+E6 (Group-1) Range	Fibers for D and S	Fibers for CA	E2 (Group-2) Range	Fibers for D and S	Fibers for CA
	mm	n	n	mm	n	n
Narrow	2.75-4.94	140	100	2.54-5.98	50	30
Medium	4.75-7.83	140	100	7.05-8.60	50	30
Wide	7.85-10.08	140	100	9.45-12.81	50	30
Total		420	300		150	90

N.B: E = Extraction, n = number of sample, D = Diameter, S = Strength, CA = Contact Angle

3.4 Treatment with chemical softener (Cepreton UN) of extracted fibers: Two sets of fibers after extraction (E2 and E4) were used for softener (**Cepreton UN, Achroma life enhanced**) treatment. The exhaust method for Cepreton UN treatment provided by the production company for cellulosic fiber was given in **Table 3.4**. E4 was treated following exhaust method. However, E2 was treated using a modified exhaust method (**Table 3.4**) by modifying time and concentration of Cepreton UN.

3.4.1 Modification of time and concentration of Cepreton UN for modified exhaust method:

Canola fibers are ligno-cellulosic fibers as it has low cellulose and high lignin content (**Table 1.2**) compared to cotton. So, we modified slightly the exhaust method (**Table 3.4**) to get the maximum softness of ligno-cellulosic bast fibers. However, these parameters (**Table 3.4**) were not statistically optimized, so, the modified method cannot be recommended as standards for large scale utilization for canola fiber.

Table 3.4: The parameters and conditions of exhaust method

Parameters	Exhaust method	Modified exhaust method
pH	4.5-5.0	4.5-5.0
Concentration	0.3-2%	10%
Time	20 to 30 minutes	2 hours
Temperature	40° C	40° C
Drying	Dry at room temperature	Dry at room temperature

3.4.2 Treatment of extracted fibers with Cepreton UN using both exhaust and modified exhaust

method: Initially, 2 grams of Cepreton UN were dissolved in 100 ml of water to make a 2% solution, and then 0.5 grams of extracted dried fibers (E4) were soaked and incubated in a water bath (**Model: HAAKE SWB 20, Germany**) for 30 minutes at 40° C using exhaust method (**Table 3.3**). Furthermore, 10 grams of chemical was solubilized in 100 ml of water in a beaker to form a 10% solution, followed by 0.5 grams of dried fiber (E2) soaked in water bath for 2 hours at 40° C using modified exhaust methods (**Table 3.3**). The beaker was covered with a lid to prevent contamination. The pH was measured by using a pH meter and adjusted using acetic acid at 4.5 (**Model: Hanna HI 98127**). Various treatments were performed in the water bath located in the Department of Biosystems Engineering at University of Manitoba at a 40 ± 2 rpm speed. The fibers were incubated for 72 hours without any water washing under a $25 \pm 2^\circ$ C temperature and $33 \pm 2\%$ relative humidity (RH). As a result of treatment, the fiber color remained unchanged when we used exhaust method. However, when we used modified exhaust method, the light greenish fibers became light brownish (**Figure 3.5**). The fibers were then individually separated and stored using the same procedure as in the previous section (**3.3**).



(a) Control fibers

(b) 10 % Cepreton UN treated fibers

Figure 3.5: (a) Control Fibers and (b) Cepreton UN treated fibers storing in small plastic bags

3.4.2.1 Treated separated fibers for diameter, strength and contact angle measurement: After treatment, 50 fibers were separated and prepared to measure diameter and strength of each stem diameter of a single set. Therefore, total 150 fibers of each set were undergone for fiber diameter measurement and strength testing. Here, from 10% Cepreton UN treated extraction-2, 150 samples were tested and another 150 samples from 2% Cepreton UN treated extraction-4 were also tested. To compare and test the hypothesis statistically, data collected from control extraction-2 (150 samples) and extraction-4 (150 samples) were also taken. For hydrophobicity testing, 30 individual isolated fibers were prepared to measure the contact angle of each stem diameter of each set. Therefore, a total of 90 fibers of each set were used for fiber hydrophobicity testing. Here, from 10% Cepreton UN treated extraction-2, 90 samples and from 2% Cepreton UN treated extraction-4, another 90 samples were prepared. Control samples collected from extraction-2 (90 samples) and extraction-4 (90 samples) were also taken to compare and test the hypothesis statistically. In the following **Table 3.5**, fiber numbers of each set were presented.

Table 3.5: Sample numbers to measure diameter, strength and contact angle (treated)

Stems	2% Cepreton UN treated E4 (Group-1)	Fibers for D and S	Fibers for CA	10% Cepreton UN treated E2 (Group-2)	Fibers for D and S	Fibers for CA
	mm	n	n	mm	n	n
Narrow	2.75-4.6	50	30	2.54-5.98	50	30
Medium	4.75-7.75	50	30	7.05-8.60	50	30
Wide	7.85-10.0	50	30	9.45-12.81	50	30
Total		150	90		150	90

N.B: E = Extraction, n = number of sample, D = Diameter, S = Strength, CA = Contact Angle

3.5 Sample preparation to measure diameter, strength and contact angle: The length of individual fiber (both control (extraction-1, 2, & 4) and treated (extraction-2 & 4)) was estimated using Digital Crimp Tester (**Model: 520 TAUTEX**) for three times and then averaged the value to get the relative length of the fibers. Then each fiber was attached with a rectangular shaped previously

cut hard paper using glue (**Fabric fusion, Aleenes's Oroginal**). The length of the rectangle was **25.4 mm**. So, this **25.4 mm** length sized fibers (**Figure 3.6**) were further used for the measurement of diameter using Bioquant, strength using Instron, and contact angle (CA) using Tensiometer. The prepared samples were also packed in a small sealed plastic bag and stored at nearly $25 \pm 2^\circ\text{C}$ and $33 \pm 2\%$ RH for future use, where each bag contained 12 samples.

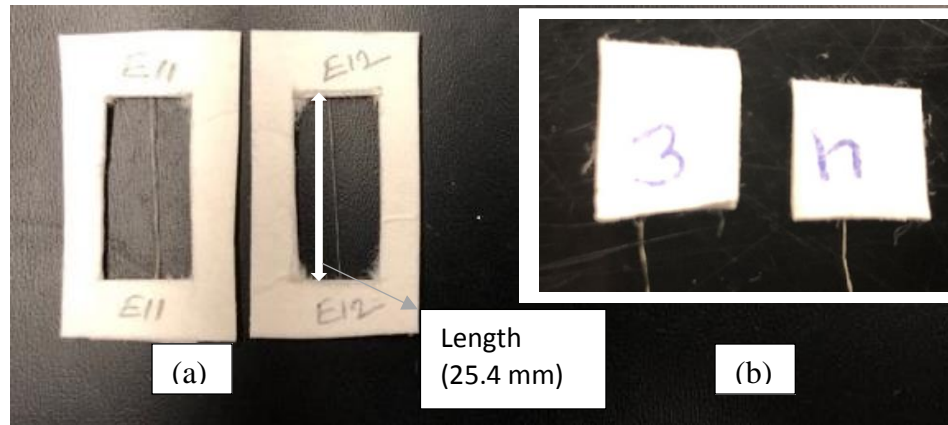


Figure 3.6: Prepared samples for diameter, strength (a), and contact angle (b) measurements

3.6 Determination of diameter of fibers: The diameter of the breaking point of fiber was measured by the **Bi quant Image Analyzer (BIQUANT Image Analysis Corporation, USA)**. The fiber diameter was calculated in micrometers (μm). Two (narrowest and widest) readings of the diameter were taken from each fiber. The mean of these two values indicated the final average diameter of the sample. The narrowest diameter was used in Instron as it is more likely that the narrowest part of fiber would break mostly and strength of this part was the general case for strength measurement. For contact angle measurement, the average diameter calculating in millimeter (mm) was used.

3.7 Determination of strength of fibers: The fiber breaking load was measured using an **Instron Strength Tester (Model: 5965, Massachusetts, USA)**. The machine was mounted with a 1 Kilo Newton (KN) load cell and fiber samples were evaluated with an upper crosshead speed of 2 mm/min following the principle of constant rate of extension (Collier, 1999). The sample length (distance

between the end points of the two clamps in Instron) for measuring the fiber strength was considered as 25.4 mm.

3.8 Determination of contact angle (CA) of fibers: The **Attention Sigma 700 force Tensiometer (Biolin Scientific, Sweden)** determined the dynamic contact angles by dipping (advancing) and withdrawing (receding) a fiber sample in the liquid. In this study, we used average diameter of fibers (taking two diameters from narrowest and widest part of the fibers) and water as liquid to dip the fibers.

3.8.1 General mechanism: When a thin and solid fiber come in contact with the water, the change in its weight is estimated by the balance (**Figure 3.7**). The detected force change is a combination of buoyancy and wetting force on the balance (Yuan & Lee, 2013). The wetting force f is defined as

$$f = \gamma l v \rho \cos \theta \dots\dots\dots(3)$$

where $\gamma l v$ is the liquid surface tension, p is the perimeter of the contact line (i.e., the same as the perimeter of the solid sample's cross-section), θ is the contact angle, and the total detected force change F on the balance is: (4)

$$F = \gamma l v \rho \cos \theta - V \Delta \rho g \dots\dots\dots(4)$$

where V is the volume of the displaced liquid, $\Delta \rho$ is the difference in density between the liquid and air (or a second liquid), and g is the acceleration of gravity (Yuan & Lee, 2013). A smaller contact angle of less than 90° is considered as high wettability. A contact angle greater than 150° is referred to as super hydrophobic showing that there is nearly no contact between the fluid drop and the surface.

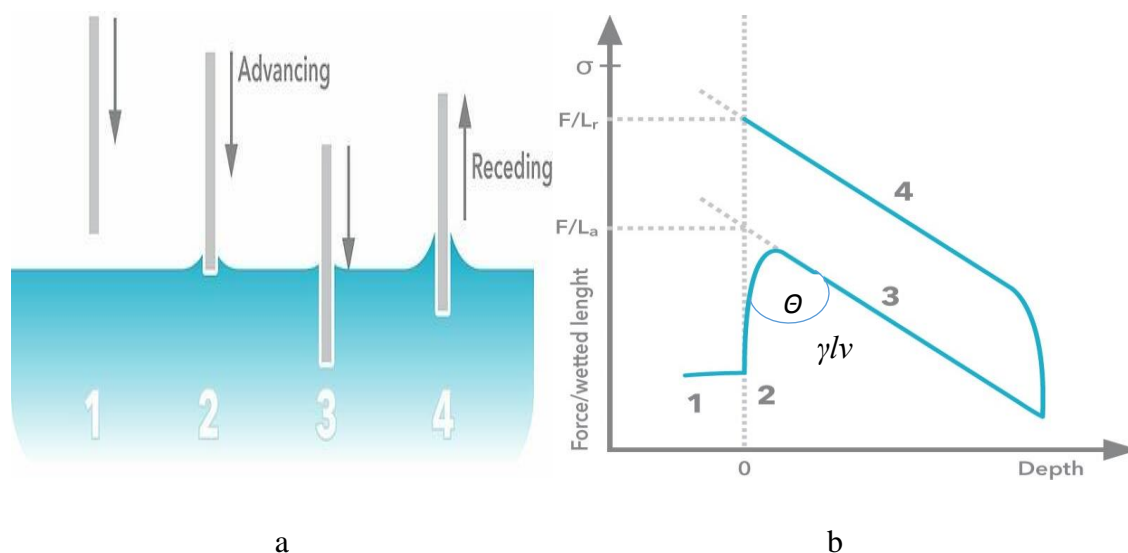


Figure 3.7: (a) A complete cycle of Wilhelmy balance method, (b) Illustration of the Wilhelmy balance method (Yuan & Lee, 2013)

(a, b): (1) The sample moves to the liquid (0.01 to 500 mm/min), then the force/length is zero. (2) The sample is in contact with the liquid surface, the liquid rises and forms a contact angle “ $< 90^\circ$ ”, causing a positive wetting force. (3) The sample approaches, and the increase of buoyancy causes a force, which is measured for the advancing angle. (4) The sample is pulled out of the liquid after having reached the desired depth; the force is measured for the receding angle (Yuan & Lee, 2013).

3.9 Estimation of moisture regain (MR) % of fibers: The moisture uptake of the fibers was measured by incubating the extracted fibers (both control and treated) in different relative humidity % (RH(%)) controlled by using desiccators (ASTM D5229, 2004). At first, all the fiber specimens were conditioned in an oven at $(100 \pm 3)^\circ\text{C}$ for 4 hours, then weighed and then again conditioned in oven at $(100 \pm 3)^\circ\text{C}$ for another 2 hours and measured the weight of the fibers. Similar procedure was repeated for another 1 hour. When the equilibrium weight was found for two consecutive measurement, then the fibers were incubated in desiccator conditioned starting with 11.3% relative humidity (RH) for 24 hours. Then the specimens were removed from the desiccator, weighed to measure the gained moisture weight, measured the moisture regain (%) using the following formula and then put back in desiccator for another 24 hours. When the equilibrium weight was found for two

consecutive measurements, then the fibers were put back in oven again to condition the fibers at $(100 \pm 3)^{\circ}\text{C}$ for 4 hours. The procedure was repeated for another 6 times to measure the moisture regain of fibers for another 6 RH (%) conditions counting total 7 RH (%) conditions. **Table 3.6** presents different parameters used in moisture regain (%) experiment.

Table 3.6: Parameter used to estimate moisture regain (%)

RH (%)	Moisture regain weight at different RH (%)		Oven dry weight at $(100 \pm 3)^{\circ}\text{C}$		
	24 hours	24 hours	4 hours	2 hours	1 hour
11.3%, 23.5%, 55%, 75.5%, 84.3%, 93.6%, 100%	g	g	g	g	g

N.B: % = Percentage, g = gram, RH = Relative Humidity

Moisture regain (%) of the extracted fibers were calculated using the following equation:

$$\text{Moisture regain (\%)} = \frac{\text{Weight of moisture in the fiber specimen}}{\text{Weight of oven dried fiber specimen}} \times 100$$

3.10 Effect of pH on fiber moisture regain (MR) %: We treated all the fibers with Cepreton UN at low pH (4.5). So, it was important to know the effect of individual low pH on fibers. To test this effect, we did a confirmation test by estimating the moisture regain (%). In this experiment, we used only one type of fibers (wide stem fibers of extraction-4) and company provided exhaust method. Here, we used two variables, such as pH 4.5 and 7.0. Here, the pH (7.0) is general water and neutral pH. This experiment was conducted with two replicates. We followed the same protocol explained in section 3.9. **Table 3.7** is showing the constant and variable conditions of the confirmation test.

Table 3.7: Constant and variable conditions of the confirmation test

Constant conditions			
Concentration (Cepreton UN)	Time	Temperature	Fibers (Stem diameter)
2%	30 minutes	40°C	E4 (Wide)
Variable conditions			
1) Fiber incubation in pH = 7.0			
2) Fiber incubation in pH = 4.5			
3) Fiber incubation with 2% Cepreton UN UN in pH = 7.0			
4) Fiber incubation with 2% Cepreton UN UN in pH = 4.5			

NB: E = Extraction

3.11 Statistical analysis: To test the hypothesis, we selected one group (group-1) of fibers among two, because the number of extractions (total 4) for group-1 were higher. To do the statistical analysis, all the properties of the fibers were pooled from extractions 1, 3, 4, and 6 and arranged from narrow to wide stems. In this case, properties of 50 fibers were taken from E1 and E4 whereas properties of 20 fibers were taken from E3 and E6 (**Table 3.8**). The data were analyzed using the software Microsoft Excel, R 3.5.3, and RStudio for Windows 10 (32/64 bits). The mean and the standard deviations were analyzed and linear model was used to find the differences. ANOVA was analyzed using one tail t-test performed by Satterthwaite's method where linear mixed model (fiber properties ~ stem diameter) using lmer in RStudio was conducted considering stem diameter as fixed effect and fiber diameter as random effect to observe the effect of stem diameter on the fiber properties (Bates et al., 2015). All the outliers were removed before the analysis.

Table 3.8: Arrangement of fibers from narrow to wide stems to test the hypothesis

Stems	Extractions (group-1)	Fibers (n)
N	E1	50
N	E3	20
N	E4	50
N	E6	20
M	E1	50
M	E3	20
M	E4	50
M	E6	20
W	E1	50
W	E3	20
W	E4	50
W	E6	20

NB: E = Extraction, N = Narrow, M = Medium, W = Wide

On the other hand, to observe the effect of fiber diameter on fiber properties, the following linear mixed models were used where the random effect was stem diameter and fixed effects were narrowest and average diameter (**Table 3.9**). The models were developed using the simple formulas of different parameters. The average length, tenacity, and aspect ratio were analyzed using narrowest diameter and the contact angle was analyzed using average diameter of fibers. The tensile stress was analyzed using breaking load multiplying inverse area whereas the load at break was analyzed using tensile stress multiplying inverse area. The young modulus was analyzed using tensile stress multiplying inverse tensile strain) and elongation at break was analyzed using tensile strain dividing young's modulus (**Table 3.9**). To get the mean value and standard deviations, transformed (square rooted) data was used but the mean comparisons were done on reverse-transformed (squared) data using Microsoft Excel to reduce overall standard deviation. *Brassica napus* fiber is a natural fiber and the standard deviation of natural fibers are generally high (Alcock et al., 2018; Shuvo et al., 2018).

Table 3.9: Models used in this study to test the hypothesis

Formulas	Models
	avelength/tenacity/aspectratio/contact angle ~ narrowstdiameter/avediameter
Tensile stress = Force/Area	tensilestress ~ loadatbreak x invarea
Tensile stress = Force/Area	loadatbreak ~ tensilestress x invarea
Young's modulus = Stress/Strain	youngsmodulus ~ tensilestress x invtensilestrain
Young's modulus = Stress/Strain	elongationatbreak ~ tensilestress/youngsmodulus

N.B: invarea = inverse area, invtensilestrain = inverse tensile strain

CHAPTER 4: RESULTS AND DISCUSSION

4.1 Fiber yield (%) of the extracted fibers: The fiber yield (%) was measured using the total weight of the stems and the retted fibers after extraction and drying. Here, there were six extractions and each extraction contained stems of three separate diameters, and so, there were three different yield (%) in each extraction. **Table 4.1 (a)** provided the information of yield (%). Here, it is important to note that the extraction 1, 3, 4, and 6 had the similar stem diameter range categorized as group-1 and extraction-2 and 5 had the nearly same stem diameter range categorized as group-2 (**Table 4.1 (a)**).

The mean values and the standard deviations were provided in **Table 4.1 (b)** for group-1 (E1, E3, E4, and E6) and group-2 (E2 and E5) extractions considering 3 stem diameters. There was no specific pattern was observed between narrow, medium and wide stem fiber yield (%) of group-1 and group-2 extractions and it was found variable. The yield (%) was found between 2.51% and 4.84% and it was variable between different stems (narrow, medium and wide) (**Table 4.1 (a)**). Hence, the stem diameter didn't have any effect on the fiber yield (%). Previously in our lab, the fiber yield (%) of different cultivars of *Brassica napus* was found between 6.23 % and 13.82 % (Khan, 2016; Shuvo et al., 2018). In this study, the yield (%) was found comparatively lower. It might be due to the variations in room humidity condition, water condition, retting time, temperature etc. It is also important to note that canola bast fiber extraction was manual. So, individual expertise in fiber extraction was an important factor to get higher yield (%). Thinner stems are prone to lodging which enhances the problems of uneven pod maturity and disease spread. Photosynthetic capacity of the stems and pods is significantly decreased by the disease infection, reducing yield (%) (Canola Encyclopedia, 2020). So, growth condition of plants is also a significant factor to get increased or decreased yield (%).

Table 4.1 (a): Fiber yield (%) of group-1 and group-2 fibers based on different stem diameters

Extractions	Stem diameter	Yield (%)	Groups
1	Narrow	2.51	1
	Medium	3.17	
	Wide	2.76	
2	Narrow	3.82	2
	Medium	3.36	
	Wide	2.49	
3	Narrow	2.62	1
	Medium	2.60	
	Wide	3.23	
4	Narrow	4.28	1
	Medium	4.07	
	Wide	4.30	
5	Narrow	4.31	2
	Medium	4.32	
	Wide	3.92	
6	Narrow	4.84	1
	Medium	3.81	
	Wide	4.17	

Table 4.1 (b): The mean value of fiber yield (%) of group-1 and group-2 extractions

Group-1		Group-2	
E1, E3, E4, and E6		E2 and E5	
Stem diameter	Yield (%)	Stem diameter	Yield (%)
Narrow	3.56±1.16	Narrow	4.07±0.35
Medium	3.41±0.66	Medium	3.84±0.68
Wide	3.62±0.74	Wide	3.21±1.01

N.B: Mean±standard deviation

4.2 Effects of stem diameter on the physical and mechanical properties of the fibers (*p*-values):

ANOVA showed that significant ($p < 0.05$) differences were not found between pooled narrow, medium and wide stem fibers taken from E1, E3, E4 and E6 for the properties of average length and elongation at break. Therefore, stem diameter didn't have any effect on average length and elongation at break because no significant differences were observed. However, while other properties such as fiber diameter, load at break, tenacity, tensile stress, young's modulus, aspect ratio, and contact angle

were considered, significant differences were observed (**Table 4.2 (a)**). Therefore, ANOVA showed that stem diameter had effects on fiber properties except average length and elongation at break.

Table 4.2 (a): Observation of the effects of stem diameter on the physical and mechanical properties of group-1 fibers (*p*-values)

Properties	E1+E3+E4+E6
	<i>p</i>-values
Narrowest diameter (μm)	7.06e ⁻⁰⁷ ***
Average length (mm)	0.0992
Elongation at break (%)	0.259
Load at break (N)	2.08e ⁻⁰⁵ ***
Tenacity (gf/denier)	3.66e ⁻⁰⁵ ***
Tensile stress (MPa)	0.000278***
Young's modulus (GPa)	0.00549**
Aspect ratio (l/d)	0.000242***
Contact angle (°)	0.000633***

N.B: *p*-value significance: 0 '***' 0.001 '**' 0.01 '*' 0.05 '.' 0.1 ' ' 1, E = Extraction

4.2.1 Effects of fiber diameter on the physical and mechanical properties of the fibers (*p*-values):

After ANOVA analysis, insignificant ($p > 0.05$) differences were observed between different fiber diameters for average length in all extractions. Insignificant ($p > 0.05$) differences between different fiber diameters for tenacity in E4 and for contact angle in E2, E3, E4, and E6 were also found. Maximum insignificant ($p > 0.05$) differences were observed in E3 for elongation at break, load at break, and contact angle including average length. Insignificant ($p > 0.05$) differences were also observed for young's modulus as well as elongation at break in E6. On the other hand, significant ($p < 0.05$) differences for load at break, tenacity, tensile stress, young's modulus, and aspect ratio were observed between different fiber diameters for maximum extractions. This information showed that fiber diameter had strong effects on load at break, tenacity, tensile stress, young's modulus, and aspect ratio. However, elongation at break was moderately and contact angle was poorly influenced by the fiber diameter (**Table 4.2 (b)**). In this respect, group-1 and group-2 fibers showed almost similar properties.

Table 4.2 (b): Observation of the effects of fiber diameter on the physical and mechanical properties of the fibers (*p*-values)

Properties	Fiber diameter				
	Group-1				Group-2
	E1	E3	E4	E6	E2
Average length	0.1517	0.187	0.2751	1	0.4165
Elongation at break	0.000972***	0.427	0.004891**	0.350	4.24e-07***
Load at break	4.407e-09***	0.307	0.005887**	0.000976***	0.008426***
Tenacity	0.000457***	0.0297*	0.5724	0.000299***	0.01517*
Tensile stress	< 2e-16***	0.0297*	< 2e-16***	0.000292***	< 2e-16***
Young's modulus	6.825e-16***	0.000267***	7.262e-14***	0.914	7.262e-14***
Aspect ratio	< 2.2e-16 ***	1.28e-06***	5.32e-11***	0.0245*	< 2.2e-16***
Contact angle	0.02345*	0.127	0.6861	0.358	0.6485

N.B: *p*-value significance: 0 '***' 0.001 '**' 0.01 '*' 0.05 '.' 0.1 ' ' 1, "-- = Blank spot, E = Extraction

4.3 Relationship between the different variables of group-1 and group-2 extractions (corrgram

values): The corrgram values of **Table 4.3** showed that the fiber diameter was not related to average length, elongation at break, and contact angle. Load at break and tenacity were positively and moderately correlated to the fiber diameter and tensile stress, young's modulus, and aspect ratio were negatively and moderately correlated to fiber diameter (group-1 and group-2). The results of the corrgram values were nearly consistent with the *p*-values of our current study (**Table 4.2 (a) and (b)**). Alcock et al., (2018) showed that flax fibers had statistically significant negative correlation with tensile strength differed by stem diameter which is consistent with our current study findings.

Table 4.3: Finding the relationship between the variables of group-1 and group-2 fibers

Properties	Fiber diameter	
	Group-1	Group-2
	E1+E3+E4+E6 Corrgram values	E2 Corrgram values
Average length	0.07	0.07
Elongation at break	-0.11	0.07
Contact angle	-0.01	0.03
Tensile stress	-0.36	-0.57
Young's modulus	-0.46	-0.66
Aspect ratio	-0.63	-0.75
Load at break	0.16	0.34
Tenacity	0.16	0.22

N.B: E = Extraction

4.4 The relationship between fiber properties and stem diameter using mean values: In this study, a difference in physical and mechanical performance was found between narrow, medium and wide stem fibers that were separated by at least 0.10 mm range (**Table 3.1**). This large variations in stem diameters might cause large standard deviations during fiber tensile properties (Alcock et al., 2018; Shuvo et al., 2018). To reduce the large standard deviations, data were transformed to square root and squared again to get the relative value.

4.4.1 Effects of stem diameter on canola fiber diameter: To measure the mechanical properties and some physical properties (average length, aspect ratio) of the fibers, the narrowest diameter of the fibers was used. To measure the contact angle (physical property), the average diameter of the fiber was used. **Table 4.4 (b)** showed the mean values of narrowest and **4.4 (c)** showed the average diameter of the fibers isolated from narrow, medium and wide stems of group-1 and group-2 fibers. The mean values for narrowest diameter ranged from (44.19–59.91) μm and the mean values for average diameter ranged from (0.08-0.10) mm. The highest mean value of narrowest and average diameter of wide stem fibers of cumulative extractions of group-1 and medium stem fibers of group-2 (E2) was found. **Table 4.4 (a)** is showing the physical and the mechanical properties of cotton and jute fibers. The diameter for cotton fibers is 14-21 μm and jute is 12- 18 μm which are significantly lower than the canola fibers (**Table 4.4 (b) and (c)**). Therefore, canola fiber diameter might affect its application as textile fibers.

Table 4.4 (a): Physical and mechanical properties of cotton and jute

Cotton : (Müssig et al., 2010; Nayak et al., 2012; Wiegerink, 1940; Li et al., 2017) and

Jute: (Müssig et al., 2010; Cristaldi et al., 2010, All of textiles, 2021; Kaswell, 1963; Schellbach et al., 2015; Textile study center, 2021; Mia et al., 2017)

Fibers	Diameter (μm)	Length (cm)	Moiture Regain (%)	Contact angle (°)	Elongation at break (%)	Load at break (N)	Tenacity (gf/tex)	Tensile stress (MPa)	Young's modulus (GPa)	Aspect ratio (l/d)
Cotton	14-21	1.5-5.6	8.5	~ 0 (control) 156.3 (treated)	3-12	1.63-1.97	1.7-6.3	287-597	4.8	1400
Jute	12-18	100-400	10-12	36-42	1.69-1.83	31.63	26.5 – 51.2	300-700	20-50	150

Table 4.4 (b): The mean values of narrowest diameters of fibers used in this study

Stems	Narrowest diameter (μm) (used for tensile strength measurement)	
	Group-1	Group-2
	E1+E3+E4+E6	E2
Narrow	44.19 \pm 0.66	49.28 \pm 1.46
Medium	45.84 \pm 1.15	59.91 \pm 1.54
Wide	53.15 \pm 1.15	53.58 \pm 1.61

N.B: Mean \pm standard deviation, E= Extraction, μm = Micrometer

Table 4.4 (c): The mean values of average diameters of fibers used in this study

Stems	Average diameter (mm) (used for contact angle measurement)	
	Group-1	Group-2
	E1+E3+E4+E6	E2
Narrow	0.09 \pm 0.02	0.09 \pm 0.03
Medium	0.08 \pm 0.03	0.10 \pm 0.03
Wide	0.09 \pm 0.02	0.08 \pm 0.02

N.B: Mean \pm standard deviation, E= Extraction, mm = Milimeter

4.4.2 Effects of stem diameter on canola fiber length: The canola fiber average length mean values ranged from 4.78-5.85 cm which was near to cotton fiber length (1.5-5.6 cm) and much lower than the jute fiber length (100-400 cm) (**Table 4.4 (a) and (d)**). There was no correlation observed between narrow, medium, and wide stem fiber length of group-1 and group-2 fibers and the mean values were found highly variable. Therefore, stem diameter didn't have any effect on average length of fibers. Fiber length is one of the most important characteristics in productivity of textile manufacturing, such as, most of the shorter fibers (e.g. < 4–5 mm) usually waste in the manufacturing process. Fibers with 5–15 mm in length give the fullness of the yarn rather than its strength, whereas fibers above 12–15 mm long contribute to yarn strength and survive carding without significant shortening (Das, 2013). Therefore, 4.78-5.85 cm canola fiber length might be able to show those properties while using for textile applications.

Table 4.4 (d): Observation of the effects of stem diameter on average length (mean-values)

Stems	Average length (cm)	
	Group-1	Group-2
	E1+E3+E4+E6	E2
Narrow	5.85±1.54	5.08±0.96
Medium	5.77±1.58	5.02±1.17
Wide	5.46±1.72	4.78±1.12

N.B: Mean±standard deviation, E = Extraction

4.4.3 Effects of stem diameter on canola fiber elongation at break: In this study, the elongation at break for canola was found 1.56-1.83 % which was relatively lower than the cotton elongation at break (3-12 %) and similar to jute elongation at break (1.69-1.83%). Bast and leaf fibers have lower elongation at break (%) than seed, stalk, or industrially man-made fibers (Petroudy, 2017). In this study, elongation at break (%) for wide stem fibers of cumulative extractions of group-1 and medium stem fibers of group-2 (E2) was found highest (**Table 4.4 (e)**) making the fibers relatively less stiff. Because fibers with high elongation at break show lower strength and Young's modulus. Higher elongation at break (%) means lower ability to resist changes, thus, enhancing relative flexibility (Petroudy, 2017). Elasticity is a significant character in textile fibers, because textile products must have the ability to stretch and reform after deformation, for example, in the elbow of a garment. Therefore, the fiber elongation at break (%) should be at least 1–2% which was consistent with our present study. With much higher elongation values (15–30%) in synthetic fibers often have spinning and drafting difficulties (Das, 2013). Elongation at break showed that wide stem fibers of group-1 and medium stem fibers of group-2 canola fibers might be useful for textile applications.

Table 4.4 (e): Observation of the effects of stem diameter on elongation at break (mean-values)

Stems	Elongation at break (%)	
	Group-1	Group-2
	E1+E3+E4+E6	E2
Narrow	1.67±0.09	1.56±0.12
Medium	1.71±0.09	1.80±0.11
Wide	1.83±0.07	1.64±0.10

N.B: Mean±standard deviation, E = Extraction

4.4.4 Effects of stem diameter on canola fiber load at break and tenacity: The load at break and tenacity were also found to be lower in canola fibers 0.38-0.76 N and 0.40-0.77 gf/tex than found in cotton fibers 1.63-1.97 N and 1.7-6.3 gf/tex, respectively and much lower than found in jute fibers 31.63 N and 26.5 – 51.2 gf/tex, respectively (**Table 4.4 (a), (f), and (g)**). In a fabric, as the tearing force/breaking load increases, the specific tightness of the yarns decreases and flexibility of yarn increases (Eltahan, 2018). In the manufacturing of industrial fabrics, the high tenacity yarn is very useful, especially airbag fabrics (Keyavlon Impex, 2020; Tyagi, 2010). Therefore, high load at break and tenacity of fiber are important characteristics for the flexibility or elasticity of fibers to make yarn. Generally, stiffer fibers are used for composite productions (Neagu et al., 2006) and flexible fibers are useful for textile applications (Das, 2013). In this study, load at break, and tenacity for wide stem fibers of cumulative extractions of group-1 and medium stem fibers of group-2 (E2) was found highest (**Table 4.4 (f) and (g)**) making the fibers relatively more flexible and less stiff and useful for textile applications.

Table 4.4 (f): Observation of the effects of stem diameter on load at break (mean-values)

Stems	Load at break (N)	
	Group-1	Group-2
	E1+E3+E4+E6	E2
Narrow	0.45±0.05	0.38±0.02
Medium	0.55±0.06	0.49±0.05
Wide	0.76±0.27	0.40±0.04

N.B: Mean±standard deviation, E = Extraction

Table 4.4 (g): Observation of the effects of stem diameter on tenacity (mean-values)

Stems	Tenacity (gf/tex)	
	Group-1	Group-2
	E1+E3+E4+E6	E2
Narrow	0.46±0.05	0.40±0.02
Medium	0.56±0.08	0.50±0.06
Wide	0.77±0.08	0.44±0.04

N.B: Mean±standard deviation, E = Extraction

4.4.5 Effects of stem diameter on canola fiber tensile stress and young's modulus: The average tensile stress for cotton is 287-597 MPa and jute is 300-700 MPa and for canola fiber, it was found 192-358 MPa which was overlapping to cotton and jute but shifted lower for canola. The average young's modulus for cotton is 4.8 GPa and jute is 20-50 GPa and for canola fiber, it was observed 20-37 GPa which was predominantly higher than cotton and overlapping to jute (**Table 4.4 (h) and (i)**). Prasad and Sain (2016) studied on hemp fibers as a raw material of composite productions and observed that the mechanical properties (tensile stress and young's modulus) of natural lignocellulosic hemp fibers were found to be dependent on the fiber diameter reducing with gradual increase in fiber diameter. This is also consistent with the general observation found in synthetic fibers, where the fiber diameter decreases and the amount of internal flaws in the fibers also decreases, thus increasing the tensile stress and young's modulus of fibers. For example, the mean tensile strength and young's modulus of fibers were 4200 MPa and 180 GPa, respectively for hemp fibers with 4 μm diameter. For fibers with 66 μm diameter, these values reduced to 250 MPa and 11 GPa, respectively. For 800 μm diameter fibers, the values decreased to 10 MPa for tensile strength and 2 GPa for tensile modulus. Shahzad (2013) observed that hemp fibers with diameter of 67 μm had 277 MPa tensile stress and 9.5 GPa young's modulus to find their compatibility to be used as reinforcement in composite materials.

Our results were consistent with these findings for wide stem fibers of cumulative extractions of group-1 and medium stem fibers of group-2 (E2). Because the wide stem fibers of group-1 and medium stem fibers of group-2 had highest fiber diameter, but the tensile stress and young's modulus were found lowest (**Table 4.4 (a), (h), and (i)**). Therefore, those fibers had lowest twisting moment force (tensile strength) and lowest ability to withstand changes, hence, the amount of internal flaws were also lowest for those fibers. Moreover, a flexible material has a low Young's modulus and changes its shape considerably (e.g. rubbers) (Property Information, 2020; Omnexus, 2020). This

quality indicated that wide stem fibers of group-1 and medium stem fibers of group-2 were less stiff making relatively suitable for textile applications than the narrow and medium stem fibers of group-1 and narrow and wide stem fibers of group-2. Interestingly, the observations were found by Prasad and Sain in 2003 and Shahzad in 2013 were not consistent with our findings for the fibers of narrow and medium stem fibers of group-1 and narrow and wide stem fibers of group-2, where with the reducing fiber diameter, the tensile stress and young's modulus fluctuated.

Table 4.4 (h): Observation of the effects of stem diameter on tensile stress (mean-values)

Stems	Tensile stress (MPa)	
	Group-1	Group-2
	E1+E3+E4+E6	E2
Narrow	313.92±49.9	228.31±28.41
Medium	358.74±48.96	192.38±22.28
Wide	238.35±40.11	196±30.47

N.B: Mean±standard deviation, E = Extraction

Table 4.4 (i): Observation of the effects of stem diameter on young's modulus (mean-values)

Stems	Young's modulus (GPa)	
	Group-1	Group-2
	E1+E3+E4+E6	E2
Narrow	33.47±4.15	26.73±2.40
Medium	37.51±4.55	20.25±3.13
Wide	30.17±3.15	22.56±2.76

N.B: Mean±standard deviation, E = Extraction

4.4.6 Effects of stem diameter on canola fiber aspect ratio: In short-fiber reinforced rubber, when the fiber aspect ratio (300) is higher, the tensile stress and young's modulus in rubber is also higher on a specific length. Therefore, fiber with high aspect ratio is good for strengthening the fiber reinforced rubber. However, if the fiber aspect ratio exceeds 400, the tensile stress and young's modulus decreases due to uneven dispersion of fibers in rubber on a specific length (Ryu & Lee, 2001). In this study, the aspect ratio for canola fibers found to be significantly higher (8606-13413) (Table 4.4 (j)), because the fiber length of this study was found higher. Cotton fibers also have relatively higher aspect ratio (1400), however, the aspect ratio for jute (150) is much lower which

seems to be good for textile applications (**Table 4.4 (a)**). In general, the aspect ratio for natural fibers seems to be higher and the threshold level of aspect ratio for canola fibers for different applications would be different. In this study, the aspect ratio for wide stem fibers of cumulative extractions of group-1 and medium stem fibers of group-2 (E2) was found lowest (**Table 4.4 (j)**) than the other stem fibers making the fibers relatively less likely to produce composites and more likely to be used in textile applications. It is also important to note that aspect ratio can be controlled by controlling the length of bast fibers, composites and apparels

Table 4.4 (j): Observation of the effects of stem diameter on aspect ratio (mean-values)

Stems	Aspect ratio (l/d)	
	Group-1	Group-2
	E1+E3+E4+E6	E2
Narrow	13413±454	10840±412
Medium	12821±450	8606±256
Wide	10462±482	9289±382

N.B: Mean±standard deviation, E = Extraction

4.4.7 Effects of stem diameter on canola fiber contact angle: Untreated cotton fabric contact angle is near to zero and its super hydrophilic in normal condition, however, by grafting graphene oxide on cotton fabric, the hydrophilic functional groups removed from its surface area, as result the fabric wettability and absorbency reduced considerably (Tissera et al., 2015) and so, its contact angle turned higher (super hydrophobic, contact angle > 150°) and the contact angle for jute fiber is 36-42° which is super hydrophilic (**Table 4.4 (a)**). Therefore, treated cotton fiber relative moisture gain reduced retaining its original quality for long at different atmospheric conditions (Yuan & Lee, 2013; Tissera et al., 2015). In our study, contact angle found for control canola fiber was found 112°-127° which was far higher than control cotton fabric and the highest contact angle was found in the wide stem fibers of cumulative extractions of group-1 and medium stem fibers of group-2 (E2) (**Table 4.4 (k)**) making them more hydrophobic like treated cotton and ability to retain its original quality for long. Therefore, contact angle revealed that wide stem fibers of cumulative extractions of group-1 and

medium stem fibers of group-2 (E2) were relatively more hydrophobic in nature retaining its original quality for long.

Table 4.4 (k): Observation of the effects of stem diameter on contact angle (mean-values)

Stems	Contact angle (°)	
	Group-1	Group-2
	E1+E3+E4+E6	E2
Narrow	112.16±10.80	113.28±7.91
Medium	117.8±16.03	127.78±14.33
Wide	126.11±20.8	118.35±12.85

N.B: Mean±standard deviation, E = Extraction

In summary, mean values showed that stem diameter had effects on fiber properties (except average length) including fiber diameter. The mean values of elongation at break (**Table 4.4 (e)**), load at break (**Table 4.4 (f)**), tenacity (**Table 4.4 (g)**), and contact angle (**Table 4.4 (k)**) were highest for wide stem fibers of cumulative extractions of group-1 and medium stem fibers of group-2 (E2). On the contrary, the mean values of tensile stress (**Table 4.4 (h)**), young's modulus (**Table 4.4 (i)**), and aspect ratio (**Table 4.4 (j)**) were found to be lowest for wide stem fibers of cumulative extractions of group-1 and medium stem fibers of group-2 (E2). For average length, no correlation was observed of canola fiber mean values with stem diameter (**Table 4.4 (d)**). It is interesting to note that though stem diameter had neutral effect on average length of fibers, however, some effect was found in aspect ratio (**Table 4.4 (j)**). Significant ($p < 0.05$) differences were found between properties of group-1 and group-2 fibers when a linear model was used.

All those properties of canola fibers comparing with the values of cotton and jute showed that the mean values of wide stem fibers of cumulative extractions of group-1 and medium stem fibers of group-2 (E2) were close to cotton and jute (**Table 4.4 (a)**) and hence, making the physical and mechanical properties relatively close to cotton and jute than the narrow and medium stem fibers of group-1 and narrow and wide stem fibers of group-2. Here, it is important to note that the diameter

range of wide stem (8-10 mm) fibers of group-1 and the medium stem (7-9 mm) fibers of group-2 was almost similar and fell between 7 mm to 10 mm (**Table 3.1**).

4.4.8 Quality of middle portion stem (7-10 mm) fibers: The flax fiber tensile properties varies with the stem diameter of flax plants and the fibers with best tensile performance occurs in the middle portion of the stem (Charlet et al., 2007) which was consistent with our present study findings (7-10 mm diameter stem) (**Table 4.4 (b-k) and Table 3.1**). Bourmaud et al., (2016) found that the fiber diameter decreases from the bottom to the top of the stem which doesn't go with our present study findings, it might be due to the variation in the architectures of canola and flax plants.

Charlet et al. in 2007 and in 2009 explained that the middle stem fiber cell walls contains the highest contents of both cellulose and non-cellulosic polymers which helps the load transfer from one microfibril to another. When fibers isolated from similar diameter stems from different portions of the stem were compared, the mechanical differences were still found. The differences might be due to the differences in growing conditions where the bottom and top fibers are usually developed in a less desirable or interrupted growing conditions (Charlet et al., 2007, 2009). Moreover, thinner (topper) stems are prone to lodging which enhances the problems of uneven stem and pod maturity and spread of diseases (Canola Encyclopedia, 2020). Interestingly, in our present study, we also found the differences in mechanical properties of fibers taken from similar diameter stems from different sections except the 7-10 mm diameter stems (**Table 4.4 (b-k) and Table 3.1**). Because the chemical composition within 7-10 mm stem fibers might be similar. Alcock et al., (2018) also found that samples with the same stem diameter range had no correlations for tensile strength, young's modulus or fiber diameter that were grown in different locations or were of different varieties, but had correlation grown within same location and same variety which was also consistent with our current findings as well. Because the stems of the present study were collected from same canola field and same variety (HYREAR 3).

A natural fiber (e.g. cotton) consists of a cell wall and lumen and the fiber turns to more mature as the cell wall thickens. A moisture-swollen mature cotton fiber cell wall comprises 50–80% of the fiber cross-section whereas immature cotton fiber comprises 30–45% and dead cotton fiber has less than 25%. Industrial cotton stock does not include too many immature or dead fibers due to lack of adequate strength which can lead to problems such as loss of yarn strength, variable dye uptake and processing difficulties (Das, 2013). In our study, 7-10 mm canola stem fibers were found to be relatively more useful for textile applications rather than other types of stem fibers.

4.5 Effects of stem diameter on fiber moisture regain (%): The increasing trend of moisture regain (%) was observed for 11.3%, 23.5%, 55%, 75.5%, 84.3%, 93.6% RH. On the other hand, a decreased trend in moisture regain (%) was found at 100% RH (**Table 4.5 (a) and (b)**). At saturated condition, fiber moisture regain capacity might be decreased. So, the MR (%) of canola fibers changed with the change in RH (%) which is also true for other natural fibers (Morton, 2008; Moudood et al., 2019). The pattern is similar for all wide, medium, and narrow stem fibers of E1, E4, E2, and E5. It is important to note that the stem diameter range was similar between E1 and E4 and between E2 and E5 (**Table 3.1**). Therefore, the mean value for each type of stem fibers was calculated taking the values from E1 and E4 and from E2 and E5. Lowest mean value of moisture regain (%) was observed for wide stem fibers of E1, E4, E2, and E5 which showed that fibers taken from ≥ 8 mm stem diameter were relatively more hydrophobic in nature than the fibers isolated from < 8 mm stem diameter. The differences between mean values of E1, E4 and E2, E5 fibers were found statistically significant ($p < 0.05$) when a linear model was used.

Contact angle showed that 7-10 mm stem fibers were relatively more hydrophobic which was somewhat consistent with the moisture regain ability. The moisture regain (%) experiment showed less hydrophobicity in the control canola fibers than the relative hydrophobicity found in control fibers by contact angle measurement. Control cotton and jute fibers are hydrophilic whereas treated

cotton fibers are hydrophobic (**Table 4.4 (a)**). Therefore, this study revealed that moisture regain experiment is better than contact angle experiment for canola fiber relative hydrophilicity measurement.

Table 4.5 (a): Moisture regain (%) of fibers in different relative humidity (%) for E1 and E4

RH (%)	Moisture regain (%)								
	E1 (W)	E4 (W)	Mean (W)	E1 (M)	E4 (M)	Mean (M)	E1 (N)	E4 (N)	Mean (N)
11.3	4.358	4.585	4.47±0.16	4.061	5.614	4.84±1.10	5.371	3.729	4.55±1.16
23.5	6.747	6.304	6.53±0.31	7.595	8.156	7.88±0.40	8.462	6.734	7.60±1.22
55	13.768	10.753	12.26±2.13	14.467	14.134	14.30±0.24	13.555	13.043	13.30±0.36
75.5	19.660	16.060	17.86±2.54	20.792	20.557	20.68±0.17	18.878	19.064	18.97±0.13
84.3	21.739	17.345	19.52±3.11	22.532	21.555	22.04±0.69	20.253	20.805	20.53±0.39
93.6	30.637	25.541	28.09±3.60	34.518	38.372	36.45±2.73	26.343	28.472	27.41±1.51
100	27.427	19.958	23.69±5.28	25.313	25.088	25.20±0.16	23.544	24.579	24.06±0.73

N.B: Mean±standard deviation, E = Extraction, W = Wide, M = Medium, N = Narrow, RH = Relative Humidity

Table 4.5 (b): Moisture regain (%) of fibers in different relative humidity (%) for E2 and E5

RH (%)	Moisture regain (%)								
	E2 (W)	E5 (W)	Mean (W)	E2 (M)	E5 (M)	Mean (M)	E2 (N)	E5 (N)	Mean (N)
11.3	4.607	3.473	4.04±0.80	2.116	4.414	3.27±1.62	4.088	4.788	4.44±0.49
23.5	5.482	6.865	6.17±0.98	6.720	7.110	6.92±0.28	5.938	6.958	6.45±0.72
55	10.816	11.481	11.15±0.47	12.807	12.217	12.51±0.42	11.111	11.853	11.48±0.52
75.5	16.920	15.229	16.08±1.20	18.182	18.373	18.28±0.14	18.769	18.165	18.47±0.43
84.3	19.386	17.006	18.20±1.68	19.891	20.544	20.22±0.46	24.179	21.903	20.22±0.46
93.6	29.490	22.936	26.21±4.63	33.607	32.214	32.91±0.98	33.535	33.605	32.91±0.98
100	26.426	20.295	23.36±4.34	30.081	26.395	28.24±2.61	31.826	27.497	28.24±2.61

N.B: Mean±standard deviation, E = Extraction, W = Wide, M = Medium, N = Narrow, RH = Relative Humidity

4.6 Effects of Cepreton UN treatment on canola fiber properties (*p*-values): When E4 and E2 fibers were treated with Cepreton UN using exhaust method (2%) and modified exhaust method (10%), respectively, insignificant ($p > 0.05$) differences between different fiber diameters for average length were found in all treated fibers by ANOVA analysis. Significant ($p < 0.05$) differences were observed for tenacity in all treated fibers and for contact angle in 10% Cepreton UN treated E2 fibers only. Significant ($p < 0.05$) differences for elongation at break, load at break, tensile stress, young's modulus, and aspect ratio were observed as well. This information showed that fiber diameter had

strong effects on elongation at break, load at break, tensile stress, young's modulus, and aspect ratio of treated fibers similar to control samples. However, the influence of fiber diameter on tenacity turned from moderate to strong for treated fibers and the influence was strong for contact angle of 10% Cepreton UN treated fibers (E2) only (**Table 4.6**).

Table 4.6: Observation of the effects of fiber diameter on the physical and mechanical properties of the fibers (*p*-values)

Properties	Fiber diameter			
	E2	T.E-2	E4	T.E-4
Average length	0.4165	0.3048	0.2751	0.5817
Elongation at break	4.24e-07***	7.84e-07***	0.004891**	7.898e-07***
Load at break	0.008426***	< 2e-16***	0.005887**	7.581e-07***
Tenacity	0.01517*	0.00015***	0.5734	1.132e-05***
Tensile stress	< 2e-16***	< 2e-16***	< 2e-16***	< 2e-16***
Young's modulus	7.262e-14***	2.621e-11***	7.262e-14***	2.284e-11***
Aspect ratio	< 2.2e-16***	< 2.2e-16 ***	5.32e-11***	4.755e-11***
Contact angle	0.6485	0.003818**	0.6861	0.9309

N.B: *p*-value significance: 0 '***' 0.001 '**' 0.01 '*' 0.05 '.' 0.1 ' ' 1, “-“ = Blank spot, E = Extraction, T. E = Treated Extraction

4.7 Effects of Cepreton UN treatment on canola fiber variables (corrgram values): When E4 and E2 fibers were treated with Cepreton UN using exhaust method (2%) and modified exhaust method (10%), respectively, the corrgram values of **Table 4.7** showed that the fiber diameter was not related to average length, elongation at break, and contact angle. For control and treated E2, load at break and tenacity were positively and moderately correlated to the fiber diameter. For both control and treated samples of E4, the correlation of fiber diameter with load at break and tenacity was not found. On the other hand, tensile stress, young's modulus, and aspect ratio were negatively and moderately correlated to fiber diameter in all treated fibers. The results of the corrgram values were nearly consistent with the *p*-values (**Table 4.6**) of the present study.

Table 4.7: Finding the relationship between the variables of control and treated E2 and E4

Properties		E2 Corrgram values	Treated E2 Corrgram values	E4 Corrgram values	Treated E4 Corrgram values
Fiber diameter	Average length	0.07	-0.08	0.09	0.04
	Elongation at break	0.07	-0.12	-0.17	-0.03
	Contact angle	0.03	-0.29	-0.03	-0.01
	Tensile stress	-0.57	-0.37	-0.38	-0.24
	Young's modulus	-0.66	-0.50	-0.48	-0.43
	Aspect ratio	-0.75	-0.68	-0.50	-0.50
	Load at break	0.34	0.24	0.01	0.02
	Tenacity	0.22	0.29	0.02	0.03

N.B: E = Extraction

4.8 Effects of Cepreton UN treatment on canola fiber properties (mean-values): E4 and E2 fibers were treated with Cepreton UN using exhaust method (2%) and modified exhaust method (10%), respectively. The highest mean value of narrowest diameter of medium stem fibers of control E2 (59.91 μm) and wide stem fibers of control E4 (53.58 μm) and also the highest average diameter of medium stem fibers of control E2 (0.10 mm) and wide stem fibers of control E4 (0.10 mm) was observed. However, the mean values of narrowest diameter were decreased in both medium stem fibers of treated E2 (49.98 μm) and wide stem fibers of treated E4 (42.25 μm). The average diameter of medium stem fiber of T.E2 (0.10 mm) remained unchanged whereas the average diameter of wide stem fiber of T.E4 (0.11 mm) was increased (**Table 4.8 (a)**). It is also important to mention that the wide stem fibers of E4 and medium stem fibers of E2 fell between 7-10 mm stem fibers. The narrowest diameter of treated fibers was decreased in all cases except narrow treated E4 fibers whereas the average diameter was increased in all treated fibers except medium treated E2 fibers. The variations might be due to the effect of variations in the concentrations of Cepreton UN (2% and 10%) and in the incubation times (30 minutes and 2 hours).

Table 4.8 (a): The mean values of narrowest and average diameters of fibers

Stems	Narrowest diameter (μm) (used for tensile strength measurement)				Average diameter (mm) (used for contact angle measurement)			
	E4	T.E4	E2	T.E2	E4	T.E4	E2	T.E2
Narrow	49.28±1.46	43.82±1.12	37.82±0.14	48.58±0.71	0.09±0.03	0.11±0.02	0.09±0.03	0.10±0.03
Medium	59.91±1.54	40.83±0.19	42.64±1.37	49.98±0.92	0.09±0.03	0.11±0.03	0.10±0.03	0.10±0.03
Wide	45.18±1.03	42.25±0.41	53.58±1.61	48.58±0.71	0.10±0.02	0.11±0.12	0.08±0.02	0.09±0.02

N.B: Mean±standard deviation, E = Extraction, T. E = Treated Extraction, μm = Micrometer

Table 4.8 (b): Observation of the effects of stem diameter on the mechanical properties of the E4 & T.E4 fibers (mean-values)

Stems	Average length (mm)		Elongation at break (%)		Load at break (N)		Tenacity (gf/tex)	
	E4	T.E4	E4	T.E4	E4	T.E4	E4	T.E4
Narrow	5.75±1.60	5.26±1.03	1.82±0.07	1.30±0.04	0.56±0.06	0.32±0.03	0.56±0.06	0.34±0.03
Medium	5.80±1.60	5.72±1.68	1.72±0.08	1.49±0.07	0.56±0.06	0.55±0.06	0.56±0.06	0.55±0.06
Wide	5.93±2.01	5.38±1.69	2.16±0.08	2.37±0.12	0.79±0.02	0.96±0.10	0.79±0.02	0.98±0.10

N.B: Mean±standard deviation, E = Extraction, T. E = Treated Extraction

Table 4.8 (c): Observation of the effects of stem diameter on the mechanical properties of the E4 & T.E4 fibers (mean-values)

Stems	Tensile stress (MPa)		Young's modulus (GPa)		Aspect ratio (L/D)		Contact angle (°)	
	E4	T.E4	E4	T.E4	E4	T.E4	E4	T.E4
Narrow	502.664±56.85	249.01±30.03	54.76±3.72	39.94±3.13	14979.31 ±118.56	12405..5 ±345.59	112.1±6.43	126.25±17.53
Medium	446.90±77.62	385.34±33.99	55.50±5.20	54.61±2.89	14056.47 ±114.54	13841.52 ±274.23	122.45±12.38	127.53±15.24
Wide	237.16±50.13	217.86±16.16	40.45±3.65	34.57±1.82	13119.41 ±451.99	12613.54 ±345.22	128.72±16.43	130.39±16.72

N.B: Mean±standard deviation, E = Extraction, T. E = Treated Extraction

Comparing to control fibers, the mean values of elongation at break, load at break, tenacity, and contact angle were increased for wide stem fibers of 2% Cepreton UN treated E4, however, decreased for other stem fibers except contact angle (**Table 4.8 (a), (b), and (c)**) and the mean values were decreased for medium stem fibers of 10% Cepreton UN treated E2, however, increased for other stem fibers except elongation at break for narrow stem fibers where no change was observed (**Table 4.8 (a), (d), and (e)**). Enhanced elongation at break (%) increases the fiber relative flexibility (Petroudy, 2017). In a fabric, with the enhanced breaking load, the tightness of the yarns reduced and the flexibility of the yarn increased (Eltahan, 2018). The high tenacity yarn is used in fabric industries

(Keyavlon Impex, 2020; Tyagi, 2010). Fiber with poor absorbency and wettability help to maintain its long lasting quality at different atmospheric conditions (Yuan & Lee, 2013; Tissera et al., 2015). Therefore, 2% Cepreton UN treated E4 wide stem fibers were less stiff and 10% Cepreton UN treated E2 medium stem fibers were stiffer than their respective control fibers.

Table 4.8 (d): Observation of the effects of stem diameter on the physical and mechanical properties of the E2 & T.E2 fibers (mean-values)

Stems	Average length (mm)		Elongation at break (%)		Load at break (N)		Tenacity (gf/tex)	
	E2	T.E2	E2	T.E2	E2	T.E2	E2	T.E2
Narrow	5.08±0.96	5.92±1.40	1.56±0.12	1.56±0.11	0.38±0.02	0.44±0.05	0.40±0.02	0.44±0.05
Medium	5.02±1.17	5.84±1.46	1.80±0.11	1.64±0.14	0.49±0.05	0.45±0.05	0.50±0.06	0.46±0.05
Wide	4.78±1.12	5.66±1.36	1.64±0.10	1.74±0.05	0.40±0.04	0.61±0.03	0.40±0.04	0.62±0.03

N.B: Mean±standard deviation, E = Extraction, T. E = Treated Extraction

Table 4.8 (e): Observation of the effects of stem diameter on the physical and mechanical properties of the E2 & T.E2 fibers (mean-values)

Stems	Tensile stress (MPa)		Young's modulus (GPa)		Aspect ratio (L/D)		Contact angle (°)	
	E2	T.E2	E2	T.E2	E2	T.E2	E2	T.E2
Narrow	228.31±28.41	238.39±23.81	26.73±2.40	27.88±1.77	10840.97± 412.09	11874.46 ±391.25	113.28±7.91	128.86±16.43
Medium	192.38±22.28	226.80±26.01	20.25±3.13	24.50±3.06	8606.27±2 56.32	12095.6± 389.27	127.78±14.33	119.07±14.85
Wide	196±30.47	340.77±20.61	22.56±2.76	27.35±1.44	9289.10±3 82.59	12210.25 ±423.95	118.35±12.85	121.58±11.76

N.B: Mean±standard deviation, E = Extraction, T. E = Treated Extraction

On the contrary, the mean values of tensile stress, young's modulus, and aspect ratio were found to be decreased for fibers of 2% Cepreton UN treated E4 (**Table 4.8 (a), (b), and (c)**) and increased fibers of 10% Cepreton UN treated E2 (**Table 4.8 (a), (b), and (d)**). The differences between control and treated fibers and also 2% and 10% treated fibers were found statistically significant ($p < 0.05$) when a linear model was used.

In general, the higher fiber aspect ratio makes the fibers relatively less flexible and stiffer increasing the tensile stress and young's modulus (Ryu & Lee, 2001). Moreover, a flexible material can change its shape due to its low young's modulus (Property Information, 2020; Omnexus, 2020). Therefore,

2% Cepreton UN treated E4 fibers were less stiff and 10% Cepreton UN treated E2 fibers were stiffer than the control fibers. Variations were found in elongation at break, load at break, tenacity, and contact angle for narrow and medium stem fibers of 2% Cepreton UN treated E4 fibers and narrow and wide stem fibers of 10% Cepreton UN treated E2 fibers probably due to the effects of variable concentrations of Cepreton UN (2% and 10%) and incubation times (30 minutes and 2 hours). Moreover, with gradual decrease in fiber diameter, the tensile stress and young's modulus of natural fibers increases (Prasad & Sain, 2016). However, in this study, the narrowest diameter of treated fibers was mostly decreased with decreased tensile stress and young's modulus. These unusual properties might be due to the effect of Cepreton UN treatment on the fiber intrinsic properties. Hence, our null hypothesis was accepted for the wide stem 2% Cepreton UN treated E4 fibers and rejected for the medium stem 10% Cepreton UN treated E2 fibers. For average length, the mean values were decreased when the fibers were treated with 2% Cepreton UN (**Table 4.8 (b)**) and increased when the treatment was 10% Cepreton UN (**Table 4.8 (d)**) from their respective control fibers.

Moreover, all those properties of treated canola fibers comparing with the values of cotton and jute showed that the mean values of 2% Cepreton UN treated wide stem fibers of E4 were close to cotton and jute and the mean values of 10% Cepreton UN treated medium stem fibers of E2 were far from cotton and jute (**Table 4.4 (a), 4.8 (b), (c), (d), and (e)**) and hence, the physical and mechanical properties of 2% Cepreton UN treated wide stem fibers of E4 were relatively more close to cotton and jute than the control E4. On the contrary, the physical and mechanical properties of 10% Cepreton UN treated medium stem fibers of E2 were far from cotton and jute than the control E2. Hence, 2% Cepreton UN acts similarly on ligno-cellulosic canola bast fibers and cellulosic seed fibers.

In this study, 2% Cepreton UN was used as company provided exhaust method whereas 10% Cepreton UN was used as modified exhaust method. Considering all the above properties, it was revealed that 2% Cepreton UN or exhaust method is useful to make the fiber flexible for textile applications where

more flexible fibers are used (Das, 2013). On the other hand, 10% Cepreton UN or modified exhaust method is useful for composite productions where less flexible fibers are used (Neagu et al., 2006). Xie et al., (2010) revealed that the fiber-matrix interfacial interactions can be induced through formation of strong chemical bonding by silane coupling agents and therefore, results in considerable improvement in the mechanical performance of composites. In this study, 10% Cepreton UN with low pH functioned in the similar way like silane coupling agents. The variations between the two methods were the concentrations of Cepreton UN and incubation times. Modified exhaust method was not statistically optimized, hence, this method cannot be recommended as standards for large scale utilization for canola fiber.

4.9 Effects of Cepreton UN treatment on canola fiber moisture regain (%): When E4 and E2 fibers were treated with Cepreton UN using exhaust method (2%) and modified exhaust method (10%), an upward trend of moisture regain (%) was observed for 11.3%, 23.5%, 55%, 75.5%, 84.3%, 93.6% RH. A sudden decrease in moisture regain (%) was found at 100% RH for control samples. However, for both 2% and 10% treated samples, at 100% RH, an increasing trend in MR (%) was observed (**Table 4.9 (a) and (b)**). At saturated condition, fiber moisture regain capacity might be increased due to fiber surface modification with Cepreton UN. The pattern was similar for all wide, medium, and narrow stem fibers of control and treated E2 and E4 which revealed that stem diameter didn't have any effect on moisture regain (%) of treated fibers. Here, our null hypothesis was rejected. The MR (%) of canola fibers changes with the change of RH (%) which was also true for other natural fibers (Morton, 2008; Moudood et al., 2019) even after 2% and 10% Cepreton UN treatment. Here, the treated mean value was compared with the control value.

When the fibers (E4) were treated with 2% Cepreton UN, the mean values of moisture regain in almost all RH (%) were found mostly decreased than the control fibers (E4). But when the fibers (E2) were treated with 10% Cepreton UN, the mean values of moisture regain in most of the RH (%) were

found increased or the variation was less than the control fibers (E2) (**Table 4.9 (a) and (b)**). This result was consistent with our previously observed results for E4 (**Table 4.8 (b) and (c)**) and E2 (**Table 4.8 (d) and (e)**). When a linear model was used, the moisture regain differences between control and treated fibers and also 2% and 10% treated fibers were found statistically significant ($p < 0.05$). This result suggests that high concentration 10% Cepreton UN treatment for long time (2 hours) made the fibers to regain more moisture (MR (%)). On the other hand, 2% Cepreton UN made the fibers to regain less moisture. The cotton and jute MR (%) are also very low, only 8.5% and 10-12 % , respectively which showed that quality of the fibers were relatively near to cotton and jute when it was treated with 2% Cepreton UN. Moreover, if the fibers gain and lose less moisture at different RH (%), the relative performance of fibers would remain intact for long time (Condoir, 2020). Hence, using high concentration of softener for long time didn't make the fibers to regain less moisture. All in all, the fibers with exhaust method treatment gained less moisture content than the modified exhaust method treatment. Therefore, 2% Cepreton UN or exhaust method was found to be more useful to make the fiber to maintain its original quality for long time than 10% Cepreton UN or modified exhaust method. Water repelling agents can reduce moisture absorption and subsequent swelling of natural fibers (Xie et al., 2010) where both the methods functioned as water repelling agents.

Table 4.9 (a): Moisture regain (%) of fibers in different relative humidity (%) for E4 and 2% Cepreton UN treated E4

RH (%)	Moisture regain (%)											
	Control				2% Treatment				Control			
	E4 (W)	E4 (W1)	E4 (W2)	Mean (W)	E4 (M)	E4 (M1)	E4 (M2)	Mean (M)	E4 (N)	E4 (N1)	E4 (N2)	Mean (N)
11.3	4.585	2.344	1.575	1.96±0.54 ↓	5.614	3.788	4.375	4.08±0.42 ↓	3.729	6.250	5.442	5.85±0.57 ↑
23.5	6.304	5.426	6.400	5.91±0.69 ↓	8.156	8.397	6.918	7.66±1.05 ↓	6.734	8.537	7.383	7.96±0.82 ↑
55	10.753	9.302	8.274	8.79±0.73 ↓	14.134	15.385	13.497	12.26±2.13 ↓	13.043	10.778	9.211	9.10±1.11 ↓
75.5	16.060	15.200	14.844	15.02±0.25 ↓	20.557	14.815	12.422	14.44±1.33 ↓	19.064	18.519	16.232	17.38±1.62 ↓
84.3	17.345	14.844	13.846	14.35±0.71 ↓	21.555	20.611	19.620	20.12±0.70 ↓	20.805	19.876	18.667	19.27±0.85 ↓
93.6	25.212	18.462	19.841	19.15±0.98 ↓	38.372	22.556	21.519	22.04±0.73 ↓	28.472	21.212	20.667	20.94±0.39 ↓
100	19.958	22.094	24.603	23.45±1.77 ↑	25.088	26.515	29.560	28.04±2.15 ↑	24.579	23.810	25.828	24.82±1.43 ↑

N.B: Mean±standard deviation, E = Extraction, W = Wide, M = Medium, N = Narrow, RH = Relative Humidity, 1 = Replicate 1, 2 = Replicate 2

Table 4.9 (b): Moisture regain (%) of fibers in different relative humidity (%) for E2 and 10% Cepreton UN treated E2

RH (%)	Moisture regain (%)											
	Control				10% Treatment				Control			
	E2 (W)	E2 (W1)	E2 (W2)	Mean (W)	E2 (M)	E2 (M1)	E2 (M2)	Mean (M)	E2 (N)	E2 (N1)	E2 (N2)	Mean (N)
11.3	4.607	5.263	5.650	5.46±0.27 ↑	2.116	4.739	4.636	4.69±0.07 ↑	4.088	4.908	3.483	4.20±1.01 ↑
23.5	5.482	6.034	6.180	6.11±0.10 ↑	6.720	7.109	9.211	8.16±1.49 ↑	5.938	7.407	6.533	6.97±0.62 ↑
55	10.816	9.787	12.222	11.01±1.72 ↑	12.807	11.321	9.554	10.44±1.25 ↓	11.111	9.697	9.950	9.82±0.18 ↓
75.5	16.920	15.652	16.292	15.97±0.45 ↓	18.182	15.421	13.548	14.48±1.32 ↓	18.769	15.854	15.000	15.43±0.60 ↓
84.3	19.265	18.777	18.857	18.82±0.06 ↓	19.891	21.226	19.231	20.23±1.41 ↑	24.149	21.875	22.000	21.94±0.09 ↓
93.6	29.490	21.983	20.109	21.05±1.33 ↓	33.607	29.858	24.837	27.35±3.55 ↓	33.453	24.691	26.000	25.45±0.93 ↓
100	26.426	27.632	26.923	27.28±0.50 ↑	30.081	34.762	28.387	31.58±4.51 ↑	31.826	27.273	28.500	27.89±0.87 ↓

N.B: Mean±standard deviation, E = Extraction, W = Wide, M = Medium, N = Narrow, RH = Relative Humidity, 1 = Replicate 1, 2 = Replicate 2

4.10 Effects of pH on fiber moisture regain (%): To ensure the effects of Cepreton UN in the fiber external properties, it was necessary to confirm the non-involvement of the other external factors, such as, pH, where we did a confirmation test using two pH (4.5 and 7.0) following exhaust method.

At pH 4.5, an increasing trend was observed in MR (%) for control fibers in 11.3%, 23.5%, 55%, 75.5%, 84.3%, 93.6% and 100% RH conditions (**Table 4.10 (a)**). An increasing trend was also observed in MR (%) for 2% Cepreton UN treated fibers at neutral pH in 11.3%, 23.5%, 55%, 75.5%,

84.3%, 93.6% and 100% RH conditions (**Table 4.10 (b)**). This observed pattern was almost as similar as 2% Cepreton treatment at low pH (4.5) (exhaust method). At neutral pH 7.0, the increasing trend of moisture regain (%) was observed for 11.3%, 23.5%, 75.5%, 84.3%, and 93.6% RH. On the other hand, a sudden drop in moisture regain (%) was found at 100% RH for control samples at neutral pH (**Table 4.10 (b)**).

The MR (%) was decreased in almost all conditions except control fibers at neutral pH 7.0. This result showed that individually pH 4.5 and individually 2% Cepreton UN and combination of both (2% Cepreton UN and pH 4.5) had effect on MR (%) of canola fibers (**Table 4.10 (a) and (b)**). It was also found that the individual effect of pH 4.5 or 2% Cepreton UN at neutral pH (7.0) is nearly as similar on MR (%) as the combination (pH 4.5 and 2% Cepreton UN). When a linear model was used, the moisture regain differences between treated at pH 4.5 and control at pH 4.5, control at pH at 7.0 and treated at pH 7.0 fibers were found statistically significant ($p < 0.05$). Therefore, only by decreasing the pH of water at 4.5 would make the fibers regain less moisture. On the contrary, Li et al., (2007) revealed that alkali can improve the mechanical properties of natural fibers significantly by removing some weak components like hemicelluloses and lignin from the fiber structure and also by modifying their crystalline structure which is consistent with our current findings. Besides commercial fabric softeners, emulsion stabilizers, acids or bases to maintain an optimal pH for absorption, fragrance enhancers, and coloring agents are also considered as softeners (Schindler & Hauser, 2004). Therefore, all in all, the fibers with low pH (4.5) or 2% Cepreton UN treatment at neutral pH (7.0) would gain less moisture content maintaining its originality for extended period of time at different atmospheric conditions just as the combination (2% Cepreton UN and pH 4.5).

Table 4.10 (a): Moisture regain (%) of control and treated E4 wide stem fibers at pH 4.5

RH (%)	Moisture regain (%)					
	Control			2% Cepreton UN treated (Exhaust method)		
	E4 (W1) (4.5)	E4 (W2) (4.5)	Mean	E4 (W1) (4.5) (2%)	E4 (W2) (4.5) (2%)	Mean
11.3	3.846	5.246	4.55±0.99 [↑]	2.671	2.353	2.51±0.22
23.5	7.018	7.843	7.43±0.58 [↑]	5.900	5.837	5.87±0.04
55	10.140	10.164	10.15±0.02 [↑]	9.412	8.915	9.16±0.35
75.5	14.931	14.052	14.49±0.62 [↓]	15.089	15.234	15.16±0.10
84.3	17.133	17.647	17.39±0.36 [↑]	14.244	13.231	13.74±0.72
93.6	18.182	18.241	18.21±0.04 [↑]	17.529	16.797	17.16±0.52
100	24.211	22.924	23.57±0.91 [↑]	20.058	21.484	20.77±1.01

N.B: Mean±standard deviation, E = Extraction, W = Wide, RH = Relative Humidity, 1 = Replicate 1, 2 = Replicate 2

Table 4.10 (b): Moisture regain (%) of control and treated E4 wide stem fibers at pH 7.0

RH (%)	Moisture regain (%)					
	Control			2% Cepreton UN treated		
	E4 (W1) (7.0)	E4 (W2) (7.0)	Mean	E4 (W1) (7.0) (2%)	E4 (W2) (7.0) (2%)	Mean
11.3	5.212	4.437	4.82±0.55 [↑]	4.459	3.909	4.18±0.39 [↑]
23.5	7.818	6.757	7.29±0.75 [↑]	7.717	7.818	7.77±0.07 [↑]
55	10.323	11.905	11.11±1.12 [↑]	9.873	9.477	9.68±0.28 [↑]
75.5	15.961	15.646	15.80±0.22 [↑]	13.880	13.399	13.64±0.34 [↓]
84.3	17.264	17.347	17.31±0.06 [↑]	17.197	18.667	17.93±1.04 [↑]
93.6	26.384	25.510	25.95±0.62 [↑]	19.683	19.142	19.41±0.38 [↑]
100	23.473	22.259	22.87±0.86 [↑]	23.492	24.351	23.92±0.61 [↑]

N.B: Mean±standard deviation, E = Extraction, W = Wide, RH = Relative Humidity, 1 = Replicate 1, 2 = Replicate 2

CHAPTER 5: CONCLUSION AND FUTURE DIRECTIONS

5.1 Conclusion:

5.1.1 Overall findings of this study for control fibers were stated below:

(a) The stem diameter didn't have any effect on the fiber yield (%).

(b) ANOVA showed that stem diameter had effects on all fiber properties except for average length and elongation at break. ANOVA also showed that fiber diameter had strong effects on elongation at break, load at break, tensile stress, young's modulus, and aspect ratio. However, tenacity was moderately and contact angle was poorly and average length was not influenced by the fiber diameter.

(c) Fiber diameter was moderately and positively correlated to tenacity, load at break, and contact angle (except E4) whereas fiber diameter was moderately and negatively correlated to tensile stress, young's modulus and aspect ratio observed by corrgram.

(d) However, mean values showed that stem diameter had effects on fiber properties except average length. The mean values of elongation at break (%), load at break, tenacity, and contact angle (hydrophobicity) were found to be highest and the mean values of tensile stress, young's modulus, and aspect ratio were found to be lowest for 7-10 mm stem fibers of canola plant. Interestingly, those properties were relatively closed to the properties of cotton and jute fibers as well.

(e) Moisture regain (MR) % was increased in increasing relative humidity (RH) % except 100% RH condition. Lowest moisture regain (%) was found for wide stem fibers of all extractions which showed that fibers collected from ≥ 8 mm stems were relatively more hydrophobic.

(f) Contact angle showed that 7-10 mm stem fibers were relatively more hydrophobic which was somewhat consistent with the moisture regain ability.

Considering all those facts, it was observed that 7-10 mm stem fibers of canola plant were relatively less stiff and relatively more suitable for textile applications than the other stem fibers. Therefore, this study provided an insightful understanding of the quality of the canola fibers of different stem diameters which will ultimately help to choose the best stem to extract different qualities of canola fibers for large scale commercial uses.

5.1.2 Overall findings of this study for treated fibers were stated below:

(a) Both 2% (exhaust method) and 10% Cepreton UN (modified exhaust method) treated fiber diameter had effects on the mechanical and physical properties, such as, elongation and load at break, tenacity, contact angle, tensile stress, young's modulus, and aspect ratio.

(b) On the other hand, fiber diameter had no effect on some physical properties, such as, average length for both 2% and 10% Cepreton UN treated fibers.

(c) Fiber diameter was positively correlated to tenacity and load at break whereas fiber diameter was negatively correlated to tensile stress, young's modulus and aspect ratio.

(d) The mean values of elongation at break (%), load at break, tenacity, and contact angle (hydrophobicity) were found to be relatively higher for 2% Cepreton UN treated wide stem fibers of E4 and the mean values of tensile stress, young's modulus, and aspect ratio were found to be relatively lower for 2% Cepreton UN treated wide stem fibers of E4 than the wide stem control fibers of E4. Those values were found to be closed to that of cotton and jute showing relatively close properties of cotton and jute. All together those properties made 2% Cepreton UN treated wide stem fibers of E4 relatively less stiff than control E4 wide stem fibers.

(e) On the other hand, the mean values of elongation at break (%), load at break, tenacity, and contact angle (hydrophobicity) were found to be relatively lower for 10% Cepreton UN treated medium stem fibers of E2 and the mean values of tensile stress, young's modulus, and aspect ratio were found to

be relatively higher for 10% Cepreton UN treated medium stem fibers of E2 than the medium stem control fibers of E2. Those values were found to be far from that of cotton and jute showing properties which were relatively far from cotton and jute. All together those properties made 10% Cepreton UN treated wide stem fibers of E2 relatively stiffer than control E2 wide stem fibers. Therefore, 2% Cepreton UN treatment (exhaust method) was found better than 10% Cepreton UN treatment (modified exhaust method) in increasing the ligno-cellulosic canola bast fiber relative flexibility and holding fiber originality for long time. So, increasing the concentration of softener and treatment time wouldn't make the fiber softer and better to touch.

(f) An upward trend of Moisture regain (MR) % was observed at different relative humidity (RH) % except at 100% RH of control fibers. However, in both exhaust method and modified exhaust method treated fibers, the MR (%) was found increased at 100% RH. When the fibers (E4) were treated with exhaust method, the mean values of MR (%) at different RH (%) were found mostly decreased than the control fibers (E4) which showed that those fibers could gain less moisture than their respective control fibers.

(g) On the contrary, when the fibers (E2) were treated with modified exhaust method, the mean values of MR (%) at different RH (%) were found to be mostly increased or the variation was found less than the control fibers (E2) which showed that those fibers could gain more moisture than their respective control fibers. Therefore, exhaust method was more useful to make fiber to maintain its original quality for long at different RH (%) than the modified exhaust method.

(h) Individually low pH (4.5) and 2% Cepreton treatment at neutral pH (7.0) had nearly similar effect as 2% Cepreton at low pH (4.5) on MR (%) of canola fibers. Hence, to make fibers retaining fiber original quality, low pH treatment would be a great alternative of any commercial softener.

(i) As 2% Cepreton UN at neutral pH (7.0) also had almost similar effect as low pH (4.5) on MR (%), therefore, using 2% Cepreton UN at neutral pH (7.0) alone was also enough to make canola fibers better to touch and maintaining fiber original quality for long at different atmospheres.

5.2 Future directions: The subsequent work plans were stated below:

(a) It is planned to measure the cellulose and lignin content ratio of canola fibers based on different stems (narrow (immature), medium (mature), and wide (over mature)) to find out the exact reason of the fiber flexibility of 7mm-10 mm stems. In general, the ratio between the cellulose and lignin content in different growth stages of canola plant might be different and this ratio might affect the fiber flexibility or softness.

(b) In this study, only one type of cultivar of *Brassica napus* (HYREAR 3) was used. It is also important to compare and contrast the physical and mechanical properties of fibers of HYREAR 3 with the fibers of other cultivars of *Brassica napus* considering different diameter of stems.

(c) The difference between the exhaust method and the modified exhaust method was the concentration of Cepreton UN (2% and 10%) and also the treatment time (30 minutes and 2 hours). It would be interesting to investigate the physical and mechanical properties of fibers in 10% Cepreton UN for 30 minutes treatment as well. The other treatments could be using low pH (4.5) or 2% Cepreton UN at neutral pH (7.0).

CHAPTER 6: REFERENCES

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