Surface Modification of Hemp Fiber to Improve Spinning Properties

By

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ABSTRACT

Finer count hemp yarn (<20-50 tex) for apparel and smart textiles applications with a minimum number of imperfections cannot be manufactured using the cotton spinning system due to the presence of non-cellulosic materials in the fiber structure. An investigation has been carried out to identify the spinning properties of hemp fibers including strength, length and length variation, single fiber entity, fineness, bending modulus and softness by removing non-cellulosic materials from hemp. Different chemical and enzyme treatments were employed to improve the selected spinning properties of hemp fiber. It was found that bleaching at 95°C for 240 min (min) after scouring removed the largest amount of non-cellulosic materials. After the carding process, the softness of treated samples, with the highest weight loss (%) was found to be improved by about 2.6 times more than the virgin hemp fiber.

Table of Contents

ABSTRACT i
List of Tables v
List of Figures vi
ACKNOWLEDGMENT viii
Chapter 1 1
1 Introduction 1
1.1 Bast Fibers 2
1.1.1 Comparison of Properties of Different Bast fibers and Cotton 2
1.1.2 Physical Properties 3
1.1.3 Mechanical Properties 3
1.1.4 Chemical Properties 4
1.1.5 Thermal Properties 5
1.1.6 Composition of Bast Fibers 6
1.2 Hemp Fiber 8
1.2.1 Hemp Fiber Structure 9
1.2.2 Hemp Classification 11
1.2.3 Hemp Application 11
1.2.4 Hemp Production 13
1.3 Why Bast Fibers cannot be used for Smart Textile Applications and Apparels 14
1.4 Spinning Properties of Fibers for Cotton Spinning Systems 15
1.5 Cotton Fiber Spinning System 16
1.5.1 Blow Room 17
1.5.2 Carding 19
1.5.3 Drawing 20
1.5.4 Combing 21
1.5.5 Roving Machine 23
1.5.6 Ring Spinning Machine 24
1.6 Hemp Spinning 26
1.6.1 Long Fiber Spinning 26

1.6.2 Spinning for Short Fibers 27
1.6.3 Wet Spinning 29
1.7 Comparison of Spinning Properties of Hemp and Cotton 30
1.8 The Problem of Hemp 32
1.9 Improvement of Spinning Properties of Hemp fiber for Cotton Spinning Systems 33
1.10 33
Chapter 2 37
Experimental Methods 37
2.1 Materials 37
2.1.1 Hemp Fiber and Chemicals 37
2.2 Methods 39
2.2.1 Conditioning of Fibers 39
2.2.2 Measurement Of diameter 39
2.2.3 Measurement of Tensile Strength and Related Properties 41
2.2.4 Individualization of Fibers 42
2.2.4.1 Sieve Shaker 43
2.2.4.2 Planetary Mono Mill 43
2.2.4.3 Card Machine 44
2.2.5 Weight Loss Percentage 46
2.2.6 Moisture Regain 47
2.2.7 Surface Modification Procedures 47
2.2.7.1 Trial I & II 48
2.2.7.2 Trial III 51
2.2.7.3 Trial IV 51
2.2.7.4 Trial V & VI 52
2.2.7.5 Trial VII 52
2.2.8 Washing Sample after Treatments 54
2.2.9 Enzyme Treatment 54
2.2.10 Softness Measurement (Subjective Method) 56
2.3 Optimization of Bleaching Process 58
2.3.1 Experimental Procedure 58
Chapter 3 60

Results and Discussion 60
3.1 Weight loss (%) 60
3.1.1 Chemical Treated Fibers 60
3.1.2 Enzyme Treated Fibers 62
3.2 Fiber Length Variation 65
3.3 Mechanical Properties 65
3.3.1 Ratio of Weight Loss (%) to Tenacity Loss (%) 69
3.4 Stiffness and Softness 71
3.5 Fiber Individualization 74
3.6 Moisture Regain 75
3.7 Results of Bleaching Optimization Process 80
3.7.1 Weight Loss (%) 82
3.7.2 Effect of Using Recycled Chemicals on Weight Loss (%) 87
3.7.3 Impact of Bleaching on Fiber Softness 88
3.8 Conclusions 90
Chapter 4 93
Conclusions and Future Work 93
Chapter 5 98
References 98

List of Tables

Table 1.1: Physical properties of bast and cotton fibers 3
Table 1.2: Mechanical properties of bast and cotton fibers 4
Table 1.3: Chemical composition of some natural fibers 6
Table 1.4: List of approved cultivars for the 2012 growing season, ... 11
Table 1.5: Comparison of spinning properties of hemp and cotton 31
Table 2.1: Characteristics of decorticated hemp fiber 38
Table 2.2: Chemicals used for Treatment of Hemp fiber 38
Table 2.3: Specification of manual carding machine 46
Table 2.4: Procedure of chemical treatments 50
Table 2.5: Conditions for enzyme treatment 56
Table 2.6: Standard for softness ranking 57
Table 2.7: Three levels of time and temperature for bleaching 58
Table 3.1: Weight loss (%) of fibers treated with chemical 61
Table 3.2: Weight loss (%) of the enzyme treated fibers. 63
Table 3.3: Mechanical properties of chemically treated hemp fibers 66
Table 3.4: Mechanical properties of enzyme treated hemp fibers 66
Table 3.5: Comparison of fiber softness, stickiness and separation 73
Table 3.6: Moisture Regain (%) of chemical and enzyme treated Hemp fibers 76
Table 3.7-A: Comparison of chemical and enzyme treated fiber's properties. 81
Table 3.7: The ANOVA Procedure 83
Table 3.8: Effect of using used chemicals on weight loss (%) of bleached hemp fibers 87
Table 3.9: Softness ranking of bleached and carded hemp fibers. 89

List of Figures

Figure 1.1: Cross section of bast fiber's stem 2
Figure 1.2: A) Lignin shown as red colour in transverse sections of hemp stem, B) Pectin and wax stained in transverse sections of hemp stem 7
Figure 1.3: Cell wall model of bast fibers 8
Figure 1.4: Three important structures of lignin. 4-Hydroxyphenyl (1), guaiacyl (2), syringyl (3) structures. 8
Figure 1.5: A) Model of transverse hemp stem section zoomed to single fiber, secondary cell wall and finally the cell wall lamella B) Model of microfibril orientation throughout the secondary cell wall 10
Figure 1.6: Different uses of hemp 12
Figure 1.7: Hemp production area (acres) in Canada 14
Figure 1.8: Cleaning of Fibers at different cleaners 18
Figure 1.9: Card machine for short staple fiber 19
Figure 1.10: Draft through a roller drafting arrangement 20
Figure 1.11: Operation sequence for combed sliver 22
Figure 1.12: Arrangement of aprons at roving and ring machine 25
Figure 1.13: Bobbin formation at roving machine 25
Figure 1.14: Ring spinning arrangement for yarn production from the roving 25
Figure 1.15: A) Dried hemp stalks being cut to 1 meter to make them ready them for machine to remove the fiber. Hemp stalks pass under a series of rollers to break them down. B) The bast fiber is collected at the top while the hurds are collected at the bottom 28
Figure 1.16: A) Combing machine separating the long hemp fibers from the short hemp fibers. B) Combed and drawn out hemp fibers are referred to as sliver. 28
Figure 1.17: Hemp sliver is spun together to make yarn. 29
Figure 1.18: Set up for wet spinning 30
Figurer 2.1: Decorticated hemp fiber 37
Figure 2.2: Bioquant life science system 40
Figure 2.3: Protocol for measuring fiber diameter 40
Figure 2.4: Instron Tensile Tester 42
Figure 2.5: A) Horizontal Seive Shaker, B) Six Seive 43
Figure 2.6: A) Planetary Mono Mill, B) Marble Balls 44
Figure 2.7: Manual card machine (Biosystems Engineering dep't, UoM) 45
Figure 2.8: Launder-o-meter with machine terminal & canister on top 48
Figure 2.9: Guarded hot plate apparatus 51
Figure 2.10: Washing set up for fibers 54
Figure 3.1: Weight loss (%) by different chemical treatment 62
Figure 3.2: Effect of treatment time on Weight loss (%) (Enzyme concentration: 1.0%) 64
Figure 3.3: Breaking Load (%) and tenacity Loss (%) of different treated fibers. 67
Figure 3.4: Fibrillar structural model for PET (Polyethylene Terephthalate) fibers. 70

Figure 3.5: Principle of the flax fiber structure. Helical arrangement of fibrils in natural cellulose fiber 71
Figure 3.6: Presence of fibers in bundle form after surface modifications 74
Figure 3.7: Effect of treatment time on moisture regain of enzyme treated fibers. 78
Figure 3.8: Effect of treatment media on moisture regain of enzyme treated fibers..... 79
Figure 3.9: Comparison of weight loss (%) and moisture regain (%) by chemical and enzyme treatment 80
Figure 3.10: Effect of treatment temperature (˚C) on weight loss (%) of bleached hemp fibers... 84
Figure 3.11: Effect of treatment time (min) on weight loss (%) of bleached hemp fibers 85
Figure 3.12: Effect of treatment time (min) and temp (˚C) on weight loss (%) of bleached hemp fibers 86

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Chapter 1

1 Introduction

Hemp is a natural bast fiber and some aspects of hemp fiber make it superior than cotton fiber e.g. it requires fewer pesticides as compared to cotton. It is more environment friendly, requires less time period for growth and less water for cultivation. Canada is one of the major producers and has good climatic condition to produce hemp fibers. In Canada, most hemp production is in Manitoba, Saskatchewan and Alberta [1]. For apparel application almost half of the fiber used is cotton (40 m tons/year) followed by polyester (36 m tons) while the use of hemp fiber is only 80,000 tons per year [2]. High quality apparel and smart textiles (woven and knitted fabrics) are produced from finer count yarns (20-50 tex), either from single or blended fibers, which can only be spun on ring spinning systems (RSS). Hemp would make a strong resource for Canadian/Manitoban agriculture if it could be spun into fine yarn, as one of the Canadian hemp growers quoted that "If I can make hemp yarns suitable for the textile market my hemp fiber will be worth 8.39 times more than the current price of hemp fiber" [3]. In order to spin the fiber or fiber blend on ring spinning system, a fiber must have six spinable properties, which are strength, length and length variation $\leq$ 1/8 inch, single fiber entity, fineness, breaking twist angle (BTA) and softness [4]. Existing hemp fiber cannot be spun on RSS because it lacks these spinning properties. If hemp could be altered to exhibit similar properties to cotton and could be spun on existing machinery, it could become a significant fiber.

1.1 Bast Fibers

Cellulosic fibers collected from the stem of the certain plants are known as bast fibers. These are found in phloem of dicotyledon plants. Being located in the phloem, they must be separated from the xylem and woody part. Commonly known bast fibers are hemp, jute, flax and ramie. A cross section of bast fiber is shown in Figure 1.1. It can be observed that bast fibers are covered by the bark of the plant. Bast fibers lie under the layer of epidermis and cortex. They need further processing in order to be separated from the plant stem. The separation can also be achieved through retting. Retting can be performed by means of water, chemicals, enzymes or micro-organisms.

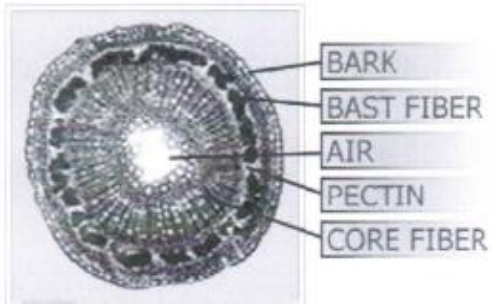


Figure 1.1: Cross section of bast fiber's stem [5]

1.1.1 Comparison of Properties of Different Bast fibers and Cotton

For general textiles and apparel applications, a set of numerous physical, mechanical and chemical properties of fibers are required. For any kind of end use there may be several fibers that could be chosen. However, it depends on the required engineering specification, cost and environmental issues. Some properties of different bast fibers have been compared in Tables 1.1 and 1.2.

Table 1.1: Physical properties of bast and cotton fibers [2, 7, 8]

Fiber	**Length** (mm) textile fiber from stem	**Finished Fiber Length for spinning** (mm)	**Finished Fiber Diameter** (microns, µg/inch)	**Density** (g/cm^3)	**Moisture** (%) (65% RH, 21°C)	**Fiber fineness** (µm)
Flax	300-900	13 - 60	12-30	1.4	7	15.0-17.0
Hemp	1000-3000	5 - 55	16-50	1.48	8	18.0-23.0
Jute	150-360	0.8 - 6	5-25	1.46	12	15.0-20.0
Cotton	15-56	15 - 56	2-6.5	1.52-1.56	8-11	16.0-21.0

1.1.2 Physical Properties

It can be observed from Table 1.1 that the finished length of the hemp fiber is close to the cotton fiber but the length variation within the hemp fiber bundle is very high. In contrast, although the length variation for cotton is relatively high, however, the length variation within a cotton variety is less than hemp. Also, the diameter variation for hemp is higher than for cotton. It is one of the reasons that they cannot be blended with cotton. The density of all bast fibers is close to that of cotton. Jute has the highest moisture regain (*moisture present in a textile material expressed as percentage of oven dry weight, at standard atmospheric conditions of 21°C and 65% relative humidity*) among bast fibers [8], however, hemp has moisture regain comparable to that of cotton, which shows that if other factors allow, cotton could be replaced with hemp for application requiring higher absorption e.g. wound dressing.

1.1.3 Mechanical Properties

Mechanical properties of different bast and cotton fibers are summarized in Table 1.2. It can be observed that tensile strength (*maximum tensile stress sustained by a fiber before*

it fails) of bast fibers is comparable to that of cotton [8]. Jute has higher elongation at break as compared to other bast fibers but less than cotton. Cotton has almost three times higher elongation at break than hemp fibers. All bast fibers have higher tenacity (*breaking load of fiber per unit linear density*) than cotton, and hemp fiber has highest among all these [8]. Because of this reason these bast fibers are more suitable for ropes, nets and sail cloth.

Table 1.2: Mechanical properties of bast and cotton fibers [2, 9]

Properties	Flax	Hemp	Jute	Cotton
Tensile Strength (GPa)	0.90	0.31-0.39	0.22-0.53	0.28-0.84
Tensile Modulus (GPa)	10-80	20-70	10-30	5.5-12.6
Elongation at Break (%)	1.8-3.3	1.7-2.7	2.3-4.6	5.6-7.1
Tenacity (g/den)	2.6-8.0	3.0-7.0	2.0-6.3	1.7-6.3

The tensile modulus, which represents the stiffness of the material, indicates that the flax, hemp and jute are highly stiff as compared to cotton .This higher stiffness also makes these fibers suitable to be used for ropes. Cotton fiber has higher elongation at break as compared to bast fibers which shows that it can withstand more cyclic loads than other fibers.

1.1.4 Chemical Properties

Chemical behavior of hemp fiber is important to study, in order to compare with cotton and other bast fiber. It is because of two reasons; first, they are required to improve the surface characteristics to be processed for yarn manufacturing. Secondly after yarn manufacturing they are converted into fabric and then subsequently dyed and printed. During this conversion of yarn to fabric and dyeing/printing, there is a series of chemical processes commonly known as wet process.

Different bast fibers behave differently when treated with alkali, as they have different components in their constitution along with cellulose. Penetration of dilute alkali affects only the inter-crystalline regions resulting from the swelling, but it fails to reach within the crystalline region, which can be reached by the concentrated alkali like sodium hydroxide. Sodium hydroxide results in not only the swelling but also the changes in crystal structure [10].

Bast fibers constitute of different elements (Table 1.3). Lignin is an important element along with others. On exposure of these fibers to sunlight, their color is changed to yellow because of this lignin oxidation. Lignin is present even in bleached fiber's structure as it is not supposed to be removed completely which will lead to loss in strength of the material. This effect can be countered by applying light-fast bleaching process which removes the lignin only from the surface while the lignin inside the structure is not affected. Although it is important to remove the lignin from the surface, it should be removed on some compromised level as it prevents the fibers from different microorganisms. These microbes reduce the tensile strength of the fibers; create odor and stains on the material [10].

1.1.5 Thermal Properties

For some applications such as fire protective apparels, thermal properties of fibers are important. How quickly a fiber heats up in response to an outside source of heat, is important. One measure of this thermal property is specific heat. Specific heat is the amount of heat required to increase the temperature of the material by one 1 °C. There is not much difference between the heat capacity of different bast fibers. Specific heat of

hemp fiber is 0.323 (cal/g/°C) whereas flax, jute and cotton have specific heat values 0.322, 0.324 and 0.292 (cal/g/°C) respectively [10].

1.1.6 Composition of Bast Fibers

The chemical composition of some bast fibers is given in Table 1.3. There is a difference in the composition of bast fibers caused by variation in the plant growing conditions, cultivation area, specie, weather etc.

Table 1.3: Chemical composition of some natural fibers [2]

Fiber	**Cellulose** (%)	**Hemicellulose** (%)	**Pectin** (%)	**Lignin** (%)	**Others** (%)
Flax	62-71	16-18	1.8-2.0	2.0-2.5	1.5
Hemp	67-75	16-18	0.8	2.9-3.3	0.7
Jute	59-71	12-13	0.2-4.4	11.8-12.9	0.5
Cotton	92-95	5.7	1.2	-----	0.6

The key component of all of these bast and cotton fibers is cellulose. Cotton has the highest cellulose content among all of these natural cellulosic fibers while hemp has the maximum among bast fibers. In order to use bast fibers for any industrial purpose they are required to be processed further so that the cellulose content can be increased. Bast fibers have higher percentage of hemicellulose as compared to cotton fiber and jute has the least among the bast fibers. Hemicellulose consists of polysaccharides which strengthen the cell wall of plants having sugar units in backbone and side chains of the structure. Hemicellulose can be dissolved in alkali but not in water. Figure 1.3 shows the position of lignin, pectin and wax in the cell wall.

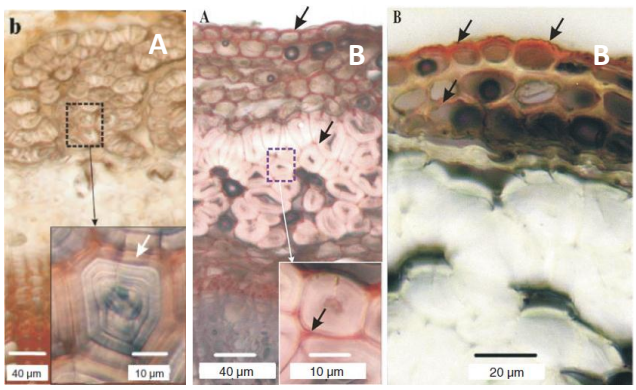


Figure 1.2: A) Lignin shown as red colour in transverse sections of hemp stem, B) Pectin and wax stained in transverse sections of hemp stem [11]

Pectin is also a polysaccharide found in the plant cell wall. It supports the plant in growth, development and defense [12]. These are water soluble substances. Hemp has the least amount of pectin among bast fibers (Table 1.3). Plants retain their structure due to the lignin which is a non-carbohydrate element. It is a phenolic compound contributing almost 30% of the secondary cell wall [13]. It has strong intermolecular bonds and protects the polysaccharides of the cell wall from decomposition. It is insoluble in water. There is no lignin in the cotton because it is a vegetable fiber. Jute fibers among the bast fibers have the highest content of lignin. The presence of lignin, pectin and waxes in the hemp stem can be observed from Figure 1.2. A clear model of the hemp cell wall is presented in Figure 1.3 [14]. It can be observed from figure that pectin and hemicellulose are attached with cellulose content present at the primary cell wall and middle lamella. It shows that cellulose is the major constituting element of the cell wall while hemicellulose and pectin are attached to it. Lignin is a mixed material consisting of representative phenyl propane (Figure 1.4).

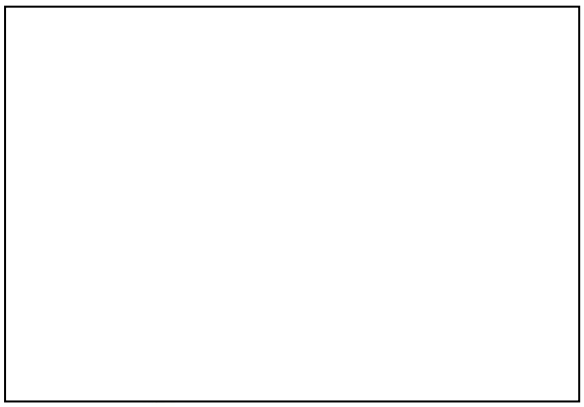
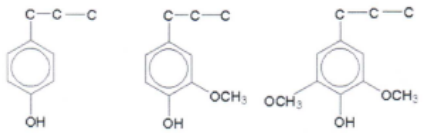


Figure 1.3: Cell wall model of bast fibers [14] (Image removed due to copyright restrictions)



(A) (B) (C)

Figure 1.4: Three important structures of lignin. 4-Hydroxyphenyl (1), guaiacyl (2), syringyl (3) structures. (With kind permission of Springer Science + Business Media [15]

1.2 Hemp Fiber

Regardless of manufacturing sector, researchers have tried to develop environment friendly products and processes. The same concept prevailed in textiles where natural fibers, including cotton, wool, flax and hemp have been used since ancient times. The product range and properties of materials were revolutionized after the development of polymer materials (Polyester, Nylon). One purpose of the development of these polymeric materials was to fulfill the world's massive fiber demand, which was not possible by depending only on natural fibers. These polymeric materials not only provided new opportunities to almost all manufacturing areas including textiles but also led to recycling related problems. Improving the yield and modifying the properties of natural fibers to make them useable is desirable.

Hemp (*Cannabis sativa*) being a natural cellulosic fiber is thought to be one of the alternatives. This is the oldest fiber which belongs to the group of vegetable fibers obtained from the stem of the plant. Originating from central Asia, this fiber has been found in structures developed in 8000 BC [16]. It is being used today for a wide range of technical applications in composite form (automotive industry) and in textiles (apparel ropes, nets etc.).

1.2.1 Hemp Fiber Structure

The chief constituting element of hemp fiber is the cellulose along with pectin, lignin, hemicellulose, natural waxes and fat (Table 1.3). Hemicelluloses are short chained polysaccharides and polyuronides attached with cellulose. Pectins are also short chain polyuronides and have an important function of binding the fiber together and responsible for the hand and luster of the fiber [17]. It is composed of pectic salts and acids [18]. Like other bast fibers, hemp also contains the fiber layer inside the stalk. Figure 1.5 explains the exact position of fibers inside the stem of the plant; moreover it also shows the orientation of microfibril inside the cell wall and presence of the lignin in different layers of secondary cell wall.

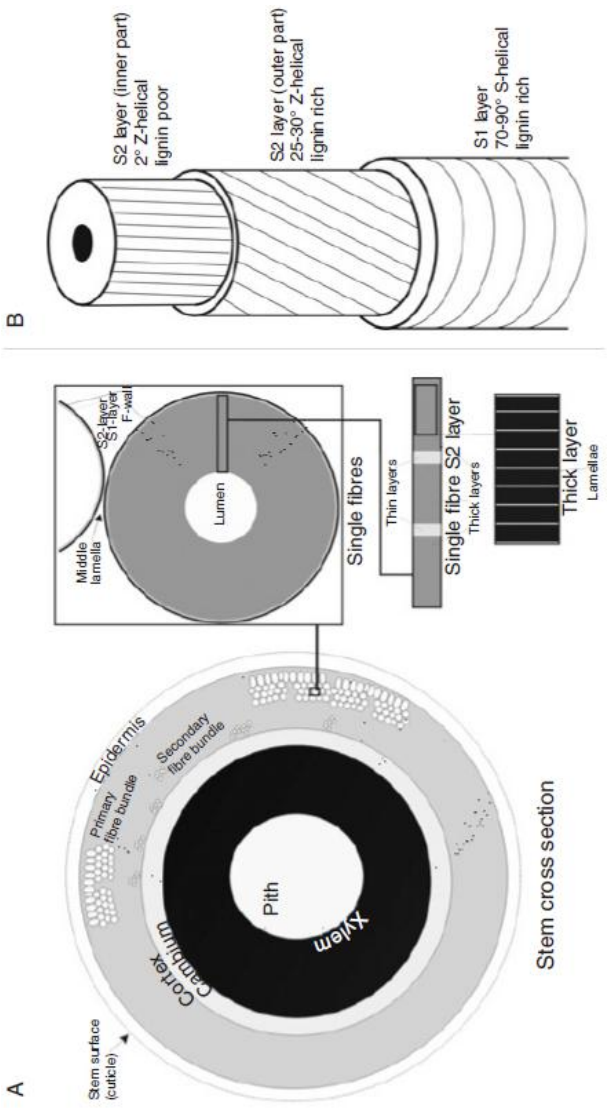


Figure 1.5: A) Model of transverse hemp stem section zoomed to single fiber, secondary cell wall and finally the cell wall lamella B) Model of microfibril orientation throughout the secondary cell wall [11]

Lignin is another important isotropic and non-crystalline element which hinders the strength and mechanical properties of fibers. It is the covering substance responsible for the cell wall's firmness [19]. Lignin provides the rigidity to the cellulose by being incorporated inside the amorphous regions of cellulose [20].

1.2.2 Hemp Classification

In order to have better yield and quality of fibers, more than four hundred (400) varieties of hemp have been developed [21]. In Canada, only the varieties with low content of tetrahydrocannabinol (THC) are allowed to be cultivated [22]. Due to the presence of THC, hemp was banned in North America after 1930, and in 1961 it was banned internationally. In Canada its cultivation was encouraged after removal of the ban in 1998 [22]. Forty one (41) varieties are allowed to be cultivated by the European Union (EU) having low levels of THC. *Cannabis Sativa* is used for extracting fibers because it has a comparatively long stem. Different hemp varieties which are allowed for cultivation in Canada for 2012 season are listed in Table 1.4.

Table 1.4: List of approved cultivars for the 2012 growing season [23]

Alyssa	Sargaszaru	CFX-2	Felina 34
Anka	Lovrin 110	Crag	Ferimon
Zolotonosha 15	Petera	CRS-1	Fibranova
Alyssa	UC-RGM	C S	Fibriko
Anka	USO 14	Delores	Fibrimon 24
CanMa	Uniko B	Deni	Fibrimon 56
Carmagnola	USO 31	ESTA-1	Joey
Carmen	X59(Hemp Nut)	Fasamo	Jutta
CFX-1	Zolotonosha 11	Fedrina 74	Kompolti
Kompolti Hibrid TC	Kompolti		

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1.2.3 Hemp Application

Hemp is used for different domestic and industrial applications including body care, food, construction industry, fuel, and plastics. Figure 1.6 shows the different areas of hemp application. An important use for hemp is in the paper manufacturing sector, in order to reduce the usage of trees. Hemp has been used for paper making for 2000 years [24].

Hemp fiber is also used for construction of buildings for thermal insulation, making concrete and clay [25]. Hemp materials used for construction are waterproof, lightweight and fireproof [26]. Hemp composites were used for floor covering and window frames during the Beijing Olympics in 2008 [27].

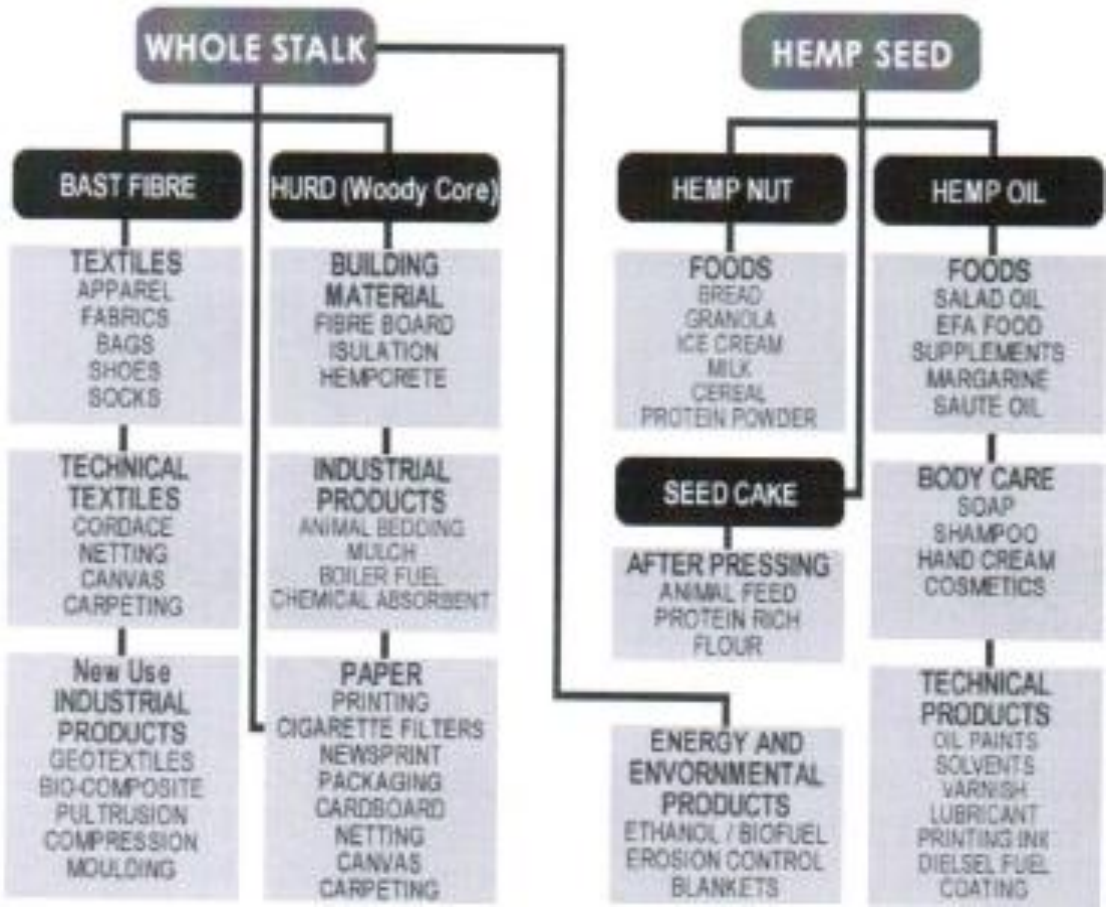


Figure 1.6: Different uses of hemp [28]

Plastics made through fossil fuels can be replaced by composite materials developed through hemp. Wambua *et al*. analyzed the mechanical properties of sisal, hemp, coir, kenaf and jute reinforced polypropylene composites [29]. The authors reported that among all fibers composites tested, hemp reinforced polypropylene composites provided

the highest mechanical properties. Hemp is also an important source of minerals, protein and dietary fibers [30].

Gasoline oil can be replaced by using biofuels, generated through hemp. It will reduce the amount of carbon monoxide produced through regular gasoline oil. Hebbel *et al*. stated that diesel engine oil can be replaced with a blend of hemp oil and diesel (50%, 25%) with and without preheating respectively [31].

Hemp is also used for textiles because of its some superior characteristics e.g. higher tensile strength, low deformation under influence of axial, flexural, tensional forces, low dry and wet elongation, higher absorbency of perspiration, anti-electrostatic and bacteriostatic properties, hygroscopicity, dye absorption and UV protection [32]. Its resistance to decay leads to uses for nets, rugs, ropes and sail cloth. All above textile and non-textile applications of hemp except apparel and smart textiles require no transformation of fiber to yarn and yarn to fabric. Only apparel and smart textile applications where woven and knitted fabrics are used require this two-stage of textile transformation.

1.2.4 Hemp Production

The farming of hemp in Canada is largely confined to the Prairie Provinces. Hemp plant cultivation area is given in Figure 1.7, which indicates the year wise production area. It can be observed that in 1998 each of the Canadian provinces cultivated less than 5000 acres but in the next year (1999), Manitoba reached cultivation of more than 20,000 acres. The maximum Manitoban cultivation was achieved in 2006 at more than 25,000 acres but it reduced to almost 12,000 acres in 2011. Alberta and Saskatchewan reached

their maximum hemp production in 2011. During the year 2003, world hemp fiber production was 77,450 tons [22].

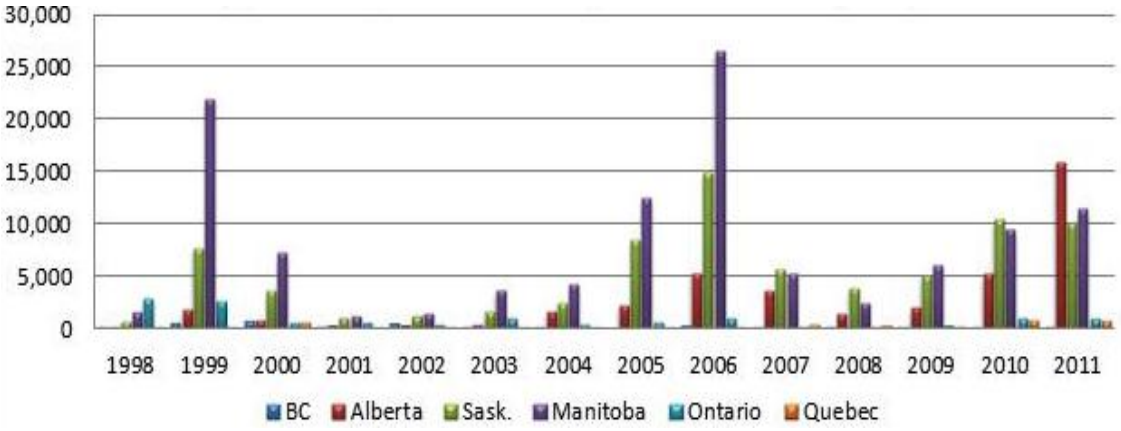


Figure 1.7: Hemp production area (acres) in Canada [22]

1.3 Why Bast Fibers cannot be used for Smart Textile Applications and Apparels

Bast fibers are widely used in different applications both for domestic and industrial uses as mentioned earlier. But if the massive utilization of natural or synthetic fibers throughout the globe for clothing is considered, the share of bast fibers, especially hemp, is very low. It is because of the surface characteristics of this fiber which makes it difficult to process on cotton spinning system. This is one of the major hindrances to hemp's extensive production as it requires higher investments for specialized machinery for hemp to produce quality spun yarns. Also the thick and thin places produced due to incomplete removal of non-cellulosic material (lignin, pectin etc.) makes it unfit to be processed through the cotton spinning system. These are the major reasons of hemp fiber for not being used widely as fabric.

1.4 Spinning Properties of Fibers for Cotton Spinning Systems

In order to have a yarn from fiber, it needs to be spun by using different methods (ring, rotor etc.). Special physical and mechanical characteristics are required for the raw materials in order to be spinable. In the same way, for cotton to be spinable there are different parameters as detailed in this section.

Fineness is the number of fibers in a given cross section and is represented by micronnaire value. In case of cotton yarn, a micronnaire value of 3-4.9 is considered to be medium range. Fiber fineness is one of the factors which limit the spinability of yarn. In a given cross section of yarn different number of fibers can be accommodated depending on fineness of the fibers. More fibers can be occupied in a given cross section if they are fine and vice versa. So, in order to have more strong and delicate yarn, the fibers should be of higher fineness. About 150-200 fibers are combined in a yarn cross section to spin the fibers in the yarn.

Fiber length is another important parameter for fiber spinning. Longer staple length is good for spinning, which along with other advantages can produce a softer yarn in comparison to a yarn of the same quality produced with a shorter staple length. The fiber length should be enough so that the leading end may be caught in roller grips before leaving the trailing roller grip while passing through the spinning system.

The strength of the yarn is equal to the strength of the weakest fiber in the yarn. Fiber cohesive properties contribute to yarn strength. Usually, the Pressley Index (PI) is used to indicate the fiber bundle strength. A fiber bundle with a PI value of 75-80 would be considered medium strength. Fiber elongation is another important property required for

a fiber to accommodate the stresses applied during the spinning and subsequent weaving process. It is usually calculated by the change in length at specified load and is expressed by percentage.

Fiber stiffness is important for twisting. A rigid fiber is more prone to breakage while twisting than a softer fiber. Stiffness is affected by the length and diameter relation of the fiber. With an increase in diameter, flexural rigidity also increases. So in order to reduce stiffness, the diameter should be at a certain defined level.

Fibers are required to be individual to make them spinable. It inhibits the thick and thin places in the ultimate yarn and fabric. Cotton fibers are naturally individual so there is no need for any physical or chemical processing to make it single, as required in case of the bast fibers. During spinning process, fibers are twisted at more than 25,000 rotations per minute (rpm). At this high speed fibers should be soft enough to withstand the twisting. If fibers have lower bending, they will break and will result in lower quality yarn.

1.5 Cotton Fiber Spinning System

Cotton spinning systems are categorized mainly in to two categories

a) Ring spinning (carded or combed) and
b) Open end spinning.

Ring spinning has two further different routes known as carded and combed. Several operations are performed during the spinning of cotton fibers. Cotton obtained from the field is passed through the ginning process which separates the fibers from the seed. Fibers are bundled in the form of a bale which is forwarded to the spinning factory.

Cotton is further mixed with other bales at the spinning facility in order to reduce the variation in raw materials and consequently the final yarn. These bales are conditioned at 21°C and 45% relative humidity to make it further process able [4].

1.5.1 **Blow Room**

The mixed and conditioned fibers are first fed to the blow room processing. At the blow room cleaning, opening, mixing, blending, micro dust removal, and uniform feeding to the carding machine is performed. After reducing the size of fiber clumps, trash (external material) is removed by mechanical agitation/beating. The process of cleaning starts from the bale plucking stage where metallic, wood, stone or other kinds of non-fibrous materials are removed by using magnets or centrifugal air system.

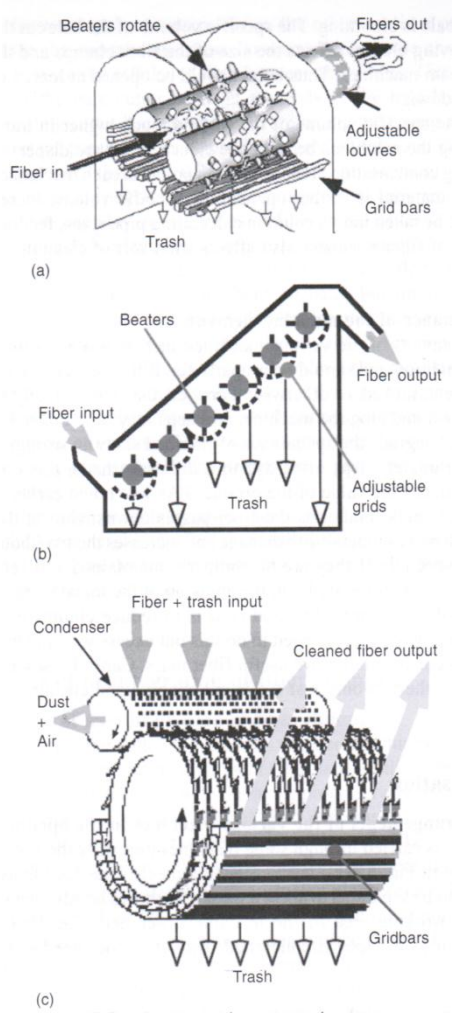


Figure 1.8: Cleaning of Fibers at different cleaners [4]

Different cleaning machines used at different positions in blow room are shown in Figures 1.8 a, 1.8 b and 1.8 c. The blending (e.g. mixing of cotton and polyester) is another important objective of the blow room. Bales of fibers are laid down in a defined order to minimize variation and produce any homogenized blend of fibers at this stage, even if using the same fiber type.

1.5.2 **Carding**

Carding is the most important process while preparing a good quality yarn. A saying in the industry is that “well carded” is half spun. Different functions performed at this stage include:

- Opening
- Cleaning
- Blending
- Fiber Orientation
- Condensation

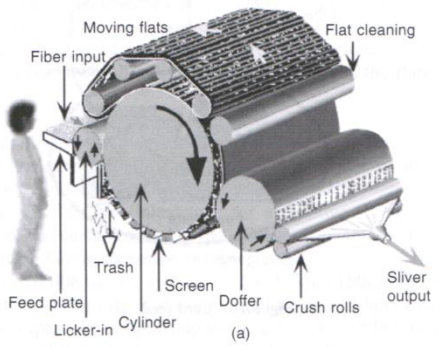


Figure 1.9: Card machine for short staple fiber [4]

Major fiber drafting (increase in length/unit weight) is done when the feed roller provides fiber clump to the licker in (Figure 1.9). Further, flats perform the fiber to fiber opening and orientation as well. Licker in or taker in removes fiber tufts from the feed roller which are further transferred to the flats through the cylinder. These pieces of tufts encounter the tearing by the action of flats. Most of these fibers are transferred to the cylinder again while the unopened tufts either small or big are again processed by the action of flats until these are opened to individual form. During carding, fiber cleaning occurs at three different zones in the card machine including cleaning at:

- Licker-in or taker-in

- Cylinder and Flats
- Fibers recirculation by cylinder (not removed by doffer the first time)

After carding, the fibers are required to be removed from the card machine (Figure 1.9) and a doffer is used for this purpose. It has been observed that the amount of fibers removed from the doffer is directly affected by the speed of the doffer, keeping oscillation frequency one per second [33].

1.5.3 **Drawing**

The carded sliver is further processed to obtain a material with even properties. For this purpose drafting of the sliver is performed (Figure 1.10) at the drawing frame. The objectives of drafting are:

- Fiber orientation
- Blending
- Equalizing
- Dust Removal

The drafting makes the sliver longer, reducing the weight per unit length or increasing the length by causing the fibers to slide on one another.

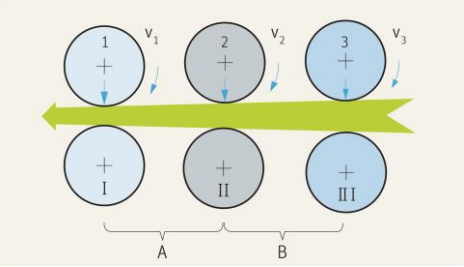


Figure 1.10: Draft through a roller drafting arrangement [34]

Sliver from different cans are passed through a drawing head where doubling or blending of the sliver is performed. This blending improves the evenness in the resultant drawn

sliver which is then passed through the drafting zone where it is drafted to the required extent. There are two types of drafts:

a) Break Draft
b) Main draft

Break draft is performed between the second and third pair of rollers while the main draft at the second and first pair of rollers. Finally the drafted sliver is passed through the condenser roller which condenses the drawn sliver increasing the fiber cohesion and also to accommodate it in the transport can.

1.5.4 **Combing**

In order to manufacture yarn of better quality drawn sliver is passed through an additional process known as combing (Figure 1.11). The objectives of combing are:

- Removal of short fibers
- Improvement in fiber orientation

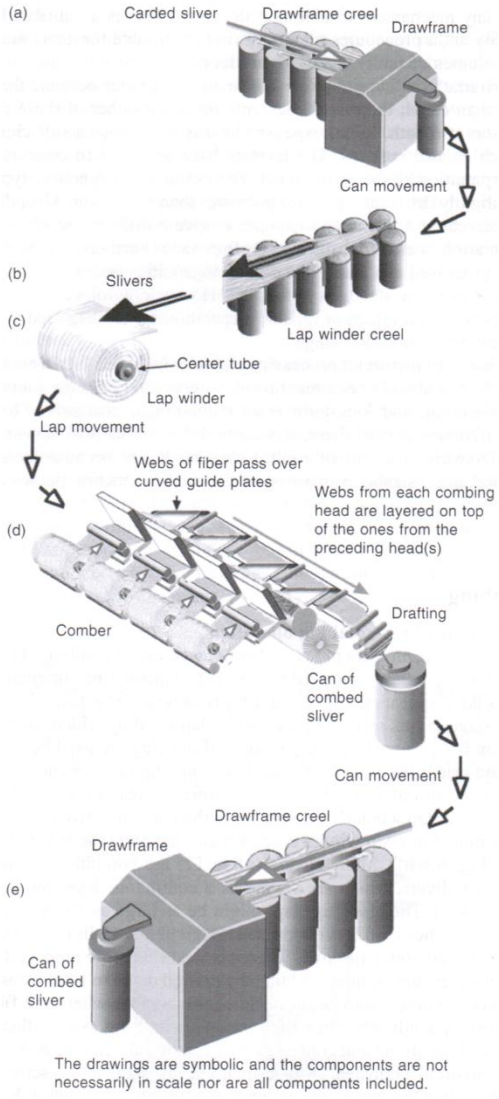


Figure 1.11: Operation sequence for combed sliver [4]

Drawn sliver is converted into lap, which is further combed and again converted into combed sliver. Different numbers of slivers (usually 12) are wound to form lap. The number of slivers combined, defines the diameter and weight of the resultant lap. These laps are combed and are drafted, finally forming a fiber web in sandwich form which is further drafted into combed sliver. At the last stage of this process, combed sliver is again passed through the drawing frame.

1.5.5 **Roving Machine**

There are usually two spinning systems known as open end and ring spinning. In the ring spinning system before going to the ring machine there is an intermittent machine known as the roving frame or simplex which forms roving by reducing the linear density and adds some twist in carded or combed sliver. The roving frame consists of the following parts [4].

- Creel
- Drafting system
- Flyer
- Winders

There are two major portions of the machine, the drafting system and a twisting set up. Klein described the roving machine in more detail [35]. There are two drafting zones in the roving machines imparting break and main draft. Short aprons are used in the main drafting zones to support the movement of the sliver. The fiber length should be enough so that it may be caught in the nip of either front or back pair of rollers otherwise there will be protruding fibers resulting in bad quality yarn.

The flyer inserts the twist into roving. Each revolution of the flyer adds one twist adding roving equal to the diameter of the roll. There is a slot in the flyer to carry the roving inside in order to reduce the effect of centrifugal force on it. In addition to the real twist, the grommet at the start of the flyer also adds the false twist into the roving. As the amount of roving increases on the roller it enhances the package density and roving tension.

1.5.6 **Ring Spinning Machine**

Roving from simplex is transferred to the ring spinning. At the ring spinning machine aprons are used to sport the fiber movement from one pair of rollers to the next (Figure 1.12) and twist is added by the movement of the bobbin (Figure 1.13) but the traveler (Figure 1.14) controls the speed while in case of the roving frame, it is added by means of flyer's movement. Each rotation of the traveler adds one twist in the resultant yarn. False twist is also added due to the pigtail, through which yarn passes just after passing through the drafting zone. Bobbin speed leads while traveler lags behind which causes the winding of yarn on the bobbin. The spindle in a ring frame machine rotates at a speed of 25,000-30,000 rpm in order to insert twist [36]. About 20-30 twist per inch is inserted in a 40 Ne yarn as twist per inch (TPI) is $TPI = 5.0\sqrt{Ne}$, where, twist factor is 5.0 and Ne is the yarn number in the English cotton system [37]. A metallic plate is used to separate the cases of two bobbins in order to save the entanglement of balloons formed by rotating yarns. The traveler revolves around the ring which is carried by a rail. The rail moves up and down which adds the length of yarn across the yarn package (Figure 1.14).

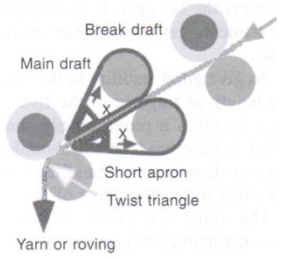


Figure 1.12: Arrangement of aprons at roving and ring machine [4]

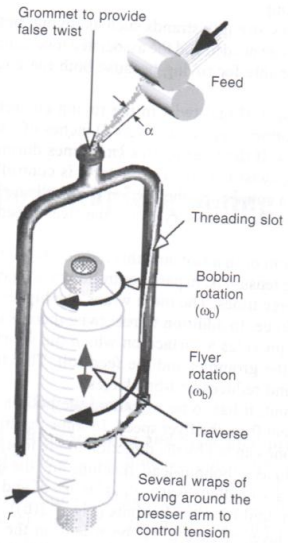


Figure 1.13: Bobbin formation at roving machine [4]

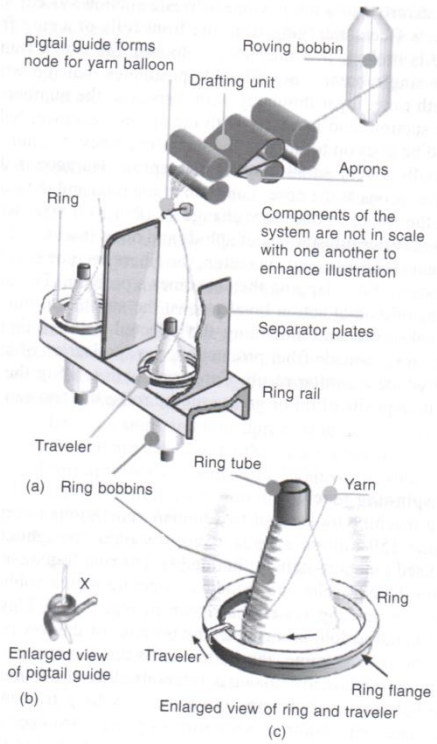


Figure 1.14: Ring spinning arrangement for yarn production from the roving [4]

1.6 **Hemp Spinning**

There are some preparatory processes for hemp before proceeding for spinning. First of all, hemp fibers are separated from stem through a process known as decortication. Decortication is achieved by separating the outer fibers from the inner core material by using mechanical means. Different mechanical processes for decortication are applied by using different machines. Through "Hammer Mill" shearing forces are applied by using rotating hammer [38]. In "Crushing Rollers" machine, compressive forces are applied by using pinned/flat roller in order to separate the fibers from the stalk [39]. The "Cutter Head" machine is a combination of knives and scutching bar which cuts and forces the fibers to be separated. [38]. In "Planetary Ball Mill" shear forces are applied by rotating the balls across the axes of the container. After these mechanical processes, the resulting material consists of a mixture of fiber, fines and core, which is further cleaned to obtain the fibers. Clean hemp fiber is presented to the spinning line after scutching or the tow fibers are passed through mechanical operations in order to have final yarns. Long and short fibers are spun by using two different routes which are detailed here.

1.6.1 **Long Fiber Spinning**

Scutched hemp of the same length and thickness is combined to feed the draw frame. Fiber bundle fineness is increased by treating the hemp with a water oil solution and then passing it through the fiber pressing machine to soften the bundle. Ribbed rollers supported by springs are used to press these bundles. Softened hemp is torn and cut to a defined length so that it may be processed further. These cut fibers are baled in a package and are placed in an environment of higher relative humidity to make them ready

for the next process. Hackling (similar to cotton combing) process removes the short fibers, parallelize and separate the fibers. For this purpose cut fibers are passed over the wooden boards having pins. Hackled fibers are placed on different belts of the drawing frame which is further processed to form sliver.

In order to further parallelize and even the sliver, further drawing and doubling is done. This process ensures improved regularity and more parallel fibers. For this purpose sliver is passed through five consecutive drawing frames. Twist is added in the later three frames in order to form roving in addition to drawing. Finally the roving is taken to the spinning frames where pure or blended hemp yarns are produced. Hemp yarn is wound on a package finally.

1.6.2 **Spinning for Short Fibers**

Hemp short fibers have a relatively different processing route for spinning than the long ones (Figures 1.15-1.17). For this purpose first of all raw material is passed through pre-carding. In this process the hurds from scutched tow and entangled fibers of hackled tow are removed to produce an even sliver to be fed to the carding process. This process is similar to carding only with the difference of rollers and cylinders with greater diameter and stronger wire on these rollers as this process is comparatively rough than the actual carding process.

Objectives of final carding are the same as pre carding i.e. removal of remaining entanglement, parallelization of fibers and increase in fineness by blending, mixing and finally forming carded sliver. Feeder is the first of three important parts of card machine. It unwinds the carded sliver received from pre-carding and feeds it to the card, which is

the second part of machine. Multiple slivers are fed at this point to get blended and even output. Carding action is performed between condensing rollers and the main cylinder. Third part i.e. draw-head receives the carded material and performs the drawing and doubling of the sliver. Instead of five draw-frames as in the case of long fibers, here are three draw frames with relatively shorter drafting zones due to shorter fiber length. The roving is processed through the spinning frame where different types of frames can be used depending on the fineness of the resultant yarn. While winding these yarns, cone winders or cross winders can be used depending upon the linear density of the fibers [40].

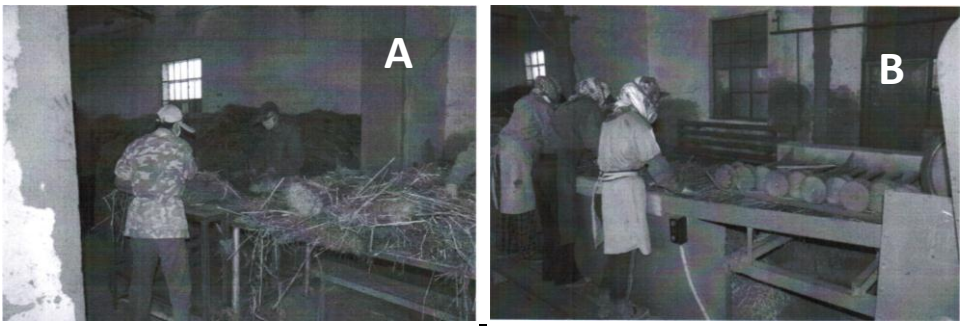


Figure 1.5: A) Dried hemp stalks being cut to 1 meter to make them ready them for machine to remove the fiber. Hemp stalks pass under a series of rollers to break them down. B) The bast fiber is collected at the top while the hurds are collected at the bottom [40]

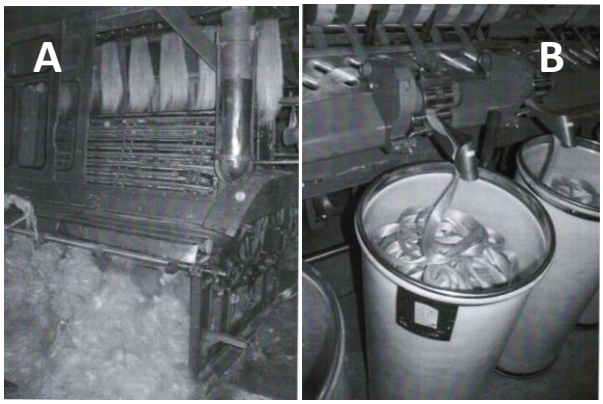


Figure 1.16: A) Combing machine separating the long hemp fibers from the short hemp fibers. B) Combed and drawn out hemp fibers are referred to as sliver. [40]



Figure 1.17: Hemp sliver is spun together to make yarn. [40]

1.6.3 **Wet Spinning**

Wet spinning is another old method of spinning hemp fibers. Before invention of this method fabrics were made with the grey yarns which were not treated with any degumming process. While proceeding for wet spinning, drafting and doubling is done after scutching and hackling. Draw frames with wooden bars are used as these were in the process chain of the dry spinning method. Doubling along with drafting of 8-12 is done here. Finally roving is formed which is wound on a perforated cylinder package eliminating the need for rewinding on a separate package in order to degum and bleach [41].

After formation of the roving, it is degummed and bleached before moving ahead for spinning. Non-cellulosic material including pectin, lignin and hemicellulose is removed by using sodium carbonate or hydroxide at boiling temperatures. After degumming bleaching is done by using some oxidizing agent (sodium hypochlorite or hydrogen peroxide) in order to improve the whiteness of roving.

For wet spinning process, a spinning frame with a container of heated water at 60°C is, mounted before the drafting zone (Figure 1.18). The roving is passed through this trough which loosens the fibers, making it easier to slide them over each other and hence supporting the drafting of roving. After making yarn it is dried and can be used immediately for weaving of fabric. As water is used for manufacturing of yarn through this method, that is why it is called wet spinning.

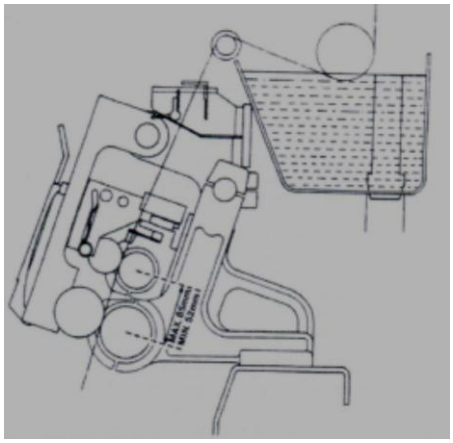


Figure 1.18: Set up for wet spinning [41]

1.7 **Comparison of Spinning Properties of Hemp and Cotton**

Certain sets of physical and mechanical parameters of fibers are required for spinning through the cotton spinning system. These parameters of cotton and hemp fibers are compared to create the definite realization for improvement in properties of hemp fibers. Textile fibers should attain a defined strength level to bear the tension and twist at different processing steps of twisting and drawing. It can be observed from Table 1.5 that the strength of hemp fiber is comparatively more than the cotton fiber which makes it suitable to be used in the traditional cotton (ring) spinning system. The fiber stiffness (*rigidity of a material*) is another big problem which makes it unsuitable for common apparel and other smart applications. Higher stiffness makes it really difficult for hemp to

withstand while passing through simplex/roving frame and ring frame where it needs to be twisted at very much higher (25,000 - 30,000 rpm) speeds, resulting in breakage. It can be observed that hemp has a stiffness of 198 g/dtex (*different values are quoted by different authors*) as compared to 60-70 g/dtex of cotton. This breakage occurs as a result of fiber straining to a critical shearing angle (*angle between fiber axis and strained part*). Brittle materials have higher twist angle e.g. glass 86° while cotton has 55°. Flax (close to hemp) has a twist angle of 68° [43].

Fibers must be individual in order to spin them into yarns. Cotton fibers are individual by nature and do not require any further processing in this regard, but the hemp fibers due to the presence of some non-cellulosic material like pectin, lignin and waxes, are hard to make separate in individual form which makes them unfit for a conventional spinning system. During processing of fiber through drawframe, speed frame and ring (spinning) frame, a drafting zone exists where fibers are drafted according to desired yarn count (Ne, tex or denier). It can create a problem if the fiber length is not enough to be gripped between the nip of the either front or back pair of rollers. If fibers are not caught in either roller nip, they will fall down which are known as 'droppings'. In contrast, the long fibers are gripped by both front and back drafting rollers that lead to fiber breakage.

Table 1.5: Comparison of spinning properties of hemp and cotton [2, 42 - 45]

Property	**Cotton**	**Hemp**
Strength (GPa)	0.28-0.84	0.31-0.39
Stiffness (g/d)	60-70	198
Breaking twist angle (°)	55	*68
Length (mm)	15-56	5-55
Fineness (μm)	16.0-21.0	18.0-23.0
Individual fiber	Single fiber	Bundle of fiber

*Value for flax.

Fiber length of cotton and hemp fiber can be observed from the Tables 1.1 and 1.5. Looking at the fiber length data, it can be seen that hemp fiber length is suitable for ring spinning process. Similarly, the average strength of virgin hemp is comparable to cotton, however, it is not known whether the strength of surface modified hemp will be strong enough to withstand the stresses during various ring spinning processes.

Weight per unit length of fiber is known as its fineness. In case of cotton there is a wide range of yarn fineness that can be achieved. But in case of hemp there are some elements which hinder the finer yarns to be achieved including complete removal of non-cellulosic material and achievement of individual fiber entity. These are the non-cellulosic substances which make it stiff, creating hindrance towards achievement of finer fiber and ultimately yarn.

1.8 **The Problem of Hemp**

For most apparel and smart textile applications woven and knitted fabrics are required. In order to produce fine quality woven and knitted fabrics high quality fine yarn (>20 tex) is essential. This fine count yarn can only be spun on cotton spinning systems. These cotton spinning systems require six important spinning properties: uniform staple length (variation <1/8 inch), fineness, lower stiffness (bending modulus), strength, softness and single entity fiber. Although virgin hemp fiber is strong enough for cotton spinning systems, however the lack of uniform staple length, fineness, high bending modulus (stiffness), softness (no fiber softness data is available in the literature) and single entity fiber (individual fiber) make it difficult for finer hemp yarn to be spun on cotton spinning systems.

1.9 Improvement of Spinning Properties of Hemp fiber for Cotton Spinning Systems

It has been discussed in section 1.8 that due to higher breaking twist angle (stiffness), lack of softness and lack of individual fiber entity status, hemp fiber cannot be processed through conventional spinning systems, which makes its use limited in the apparel and smart textile applications. Its contribution towards daily life can be increased if we can make it possible for hemp to be processed through cotton spinning systems. It requires surface modification of hemp indicating the modification of the following properties until they reach the range of cotton fiber properties:

- Bending modulus (stiffness, breaking twist angle)
- Fiber Length (staple length, which is 0.91 x effective length)
- Fineness
- Individual fiber entity
- Softness
- Strength (tenacity)

In order to improve the hemp fiber, different chemical, mechanical and biological treatments were used in the past which is discussed in section 1.10.

1.10 Literature Review

Although hemp fibers' lack of cotton spinning is well known, however, a very few work could be found on improvement for cotton spinning properties of hemp fiber. Usually, surface modifications have been carried to remove the non-cellulosic materials without considering the spinning properties. Kessler *et al.* investigated chemical method for removal of pectin and lignin [46]. For this purpose acid scouring, alkali boiling and bleaching were performed. The authors claimed that alkaline boiling process totally removed the pectin whereas acid scouring is not helpful for this purpose. Alkaline boiling

followed by bleaching through hydrogen peroxide produced the best results in order to remove the lignin content while acid scouring is helpful to degrade the lignin. However, the authors failed to measure the critical spinning parameters related to bleaching process.

Sedelnik reported an enzyme treatment process followed by a mechanical method (carding), aimed at not only the removal of non-cellulosic material but also changes in the linear density and length of fiber [47]. The author found an increase in cellulose content was 6.4% while hemicelluloses, lignin and pectin are reduced by 28%, 23% and 98% respectively. This treatment reduced the fiber linear density and mean length by 30-40% and 5.5 times respectively. The length and linear density were further reduced after carding process which is 63% and 87% respectively. No reference was made about the spinning properties of modified fiber other than length and fineness.

Dryer *et al.* studied various treatment methods to separate the hemp fibers so that they may be used in textiles for further processing and found that changes in fiber fineness and strength was similar for both enzyme and alkali treatments [48]. However, the enzyme process was environment friendly as the effluents of this process were biodegradable.

Jinqiu and Jianchun investigated hemp fiber morphology, tensile properties, moisture absorption, fiber length, chemical composition, fineness and flexibility after each step of a chemical process [49]. This study reported that 93.0% of impurities were removed; short fiber content is increased and resulted in finer fibers. Fiber strength is reduced but fineness increased the number of fiber in a given fiber count which increased the yarn strength. The moisture absorption of the fibers reduced due to reduction in impurities and

improved crystalline area. The rigidity of the fibers is reduced. No comparison of data was made with cotton fiber.

Wang and Wang analyzed the fineness of different bast fibers including degummed, carded hemp fibers by using Optical Fiber Diameter Analyzer [50]. It was pointed out that the presence of fiber bundles and irregular fiber cross section are the sources of disruption for fineness calculation of bast fibers. The research has not correlated the resulting fiber fineness with ultimate yarn fineness.

Höcker discussed a comparatively new method of bast fiber treatment for textiles, known as plasma treatment [51]. He not only detailed about the process of plasma treatment for shrinkage resistance of wool fibers and induction of dust- dirt repellency in cotton and synthetic fibers but also mentioned the application of plasma treatment for modification of topography and chemical structure of the textile fibers. The same plasma treatment was used for delignification of flax, hemp and jute fibers followed by chemical treatments [52]. Hemp fibers of 2-4 mm were treated for 10-25 min in a reactor. After plasma treatment the fibers were further treated with alkali which increased the delignification percentage. The authors claimed that using only alkali treatment, even with higher concentrations, it takes 2-3 hours at about 100°C to remove lignin not less than 50%, whereas using plasma treatment followed by alkali treatment, almost 64% of lignin can be removed by using lesser concentration of alkali and treatment time. No measurement of spinning properties was conducted.

It can be stated by summarizing the literatures review that although hemp fibers were modified to improve the fineness and linear density, but there are some other spinning

parameters that are also required to make them able to be processed through cotton ring spinning system so that fine quality yarn (30-200Ne) can be manufactured [4]. The current research focused on the spinning parameters such as bending modulus (stiffness/ breaking twist angle), fiber length (staple length, which is 0.91 x effective length), fineness (linear density), individual fiber entity, softness and tenacity in two phases. In phase-I, investigation was carried out to identify the changes in spinning parameters using surface modification. In phase-II, the bleaching process was optimized to remove the maximum amount of non-cellulosic materials and to investigate the impact on softness.

Chapter 2

Experimental Methods

This chapter deals with the details of raw material, chemicals, enzymes and experiments conducted in order to get raw hemp fiber of required quality. The purpose of treatment was to improve the physical and mechanical properties of the hemp fibers so that they can be converted into fine quality yarn.

2.1 Materials

2.1.1 Hemp Fiber and Chemicals

Decorticated hemp fibers (Figure 2.1) were used in this study. The physical and mechanical properties of the hemp fibers were measured. The results are reported in Table 2.1. Different chemicals were used throughout the series of experiments, which are detailed along with their respective suppliers in Table 2.2



Figurer 2.1: Decorticated hemp fiber

Table 2.1: Characteristics of decorticated hemp fiber

Fiber characteristics	Properties	Method	Test Method
Diameter (μm)	96.12 ± 21.94	Bioquant life science image analysis system	
Color	Brown	Visual	
Breaking load (N)	2.258	Instron Tensile Tester	ASTM D1445 / D1445M - 12
Tenacity (N/tex)	0.21	Instron Tensile Tester	ASTM D1445 / D1445M - 12
Moisture Regain (%)	7.87	Conditioning & Thermo Scientific Oven	ASTM D2564

Table 2.2: Chemicals used for Treatment of Hemp fiber

Chemical	Supplier	Purity
1993 AATCC Standard Reference Detergent	AATCC-USA	Not applicable
Sodium Hydroxide (NaOH)	Sigma Aldrich-USA	≥97%
Acetic acid	Kimax-35 USA	5g/l
Sulphuric Acid (H_2SO_4)	Fair Lawn-USA	70%
Sodium Carbonate	Sigma Aldrich-USA	≥99.5%
Sodium Sulphite	Sigma Aldrich-USA	≥98%
Sodium Dodecyle Sulfate	Sigma Aldrich-USA	92.5-100.5%
Hydrogen peroxide	Sigma Aldrich-USA	≥97%
Glycerin		
Enzeco Pectinase DV-2	Enzyme Development Corporation-USA	100%
Pectinase from Aspergillus Aculeatus	Sigma Aldrich-USA	≥3800 units/ml

2.2 Methods

2.2.1 Conditioning of Fibers

To standardize the results, fiber samples were conditioned before any measurement at 65% relative humidity (RH) and 21°C temperature for at least 24 hours, according to the test method ASTM D 1776 [53].

2.2.2 Measurement Of diameter

Fiber diameter was measured before and after chemical treatment .Bioquant Life Science Image Analysis System (BIOQUANT Image Analysis Corporation-USA) software was used for this purpose. Figure 2.2 shows the setup for Bioquant system. It includes two main parts, a microscope camera and a computer. The microscope is used to magnify the sample while the computer system is used to calculate the results. For this purpose the glass slides were prepared with the help of glycerin for fibers which are required to measure the diameter. The magnification was 40X and the measurement was taken from 20 different places along the length of each fiber so that any variation in diameter along the length could be compensated for. The protocol for measuring the width of the fiber by using Bioquant is given in Figure 2.3.

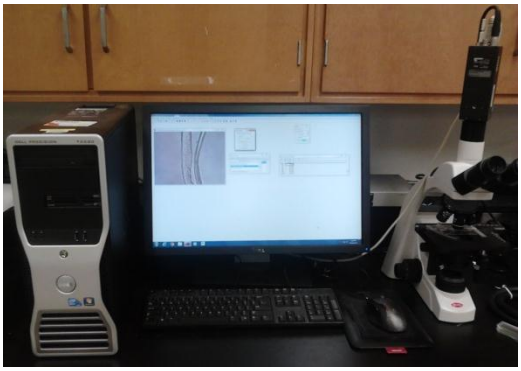


Figure 2.2: Bioquant life science system

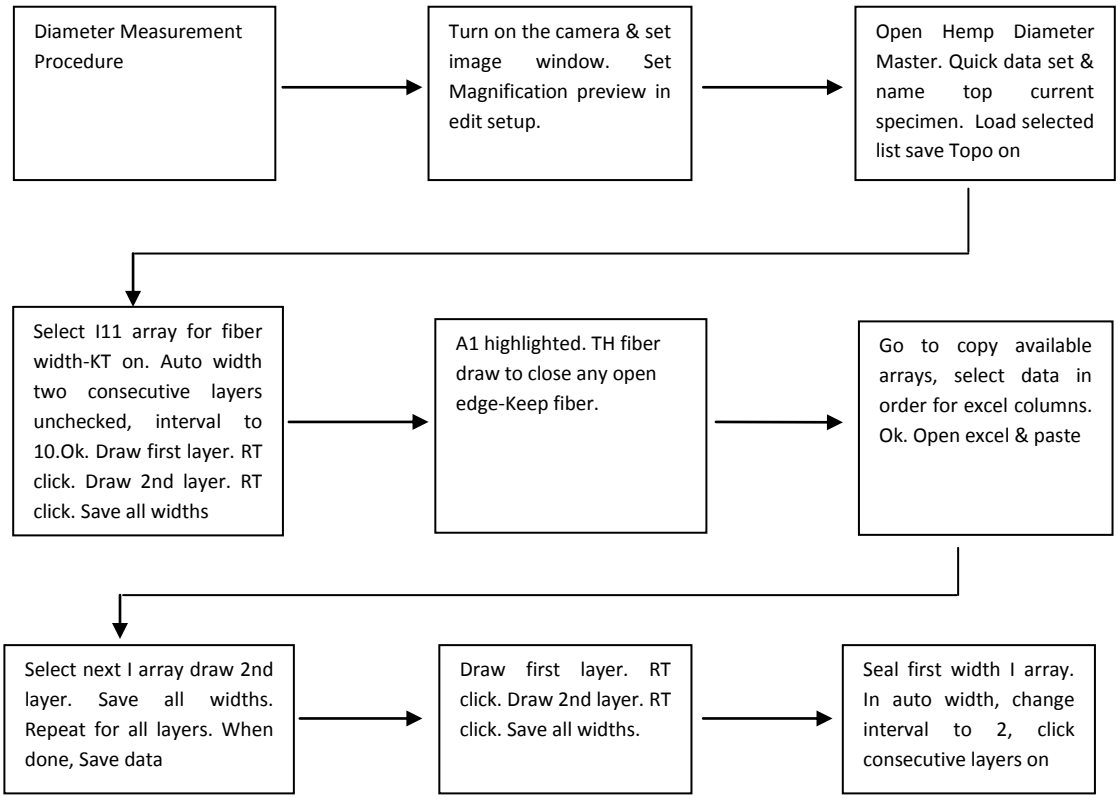


Figure 2.3: Protocol for measuring fiber diameter [54]

2.2.3 **Measurement of Tensile Strength and Related Properties**

The fibers which were used for measurement of the diameter were conditioned at standard atmospheric conditions. Paper cards of 25 X 25 mm dimension were prepared and fibers were fixed in between these cards by using adhesive tape. Breaking load of the fibers was measured both before and after treatment of fibers by using the Instron tensile tester (Figure 2.4) according to the ASTM D1445 / D1445M - 12 [55].

The experiments were planned to be conducted with a load cell of 5N and a jaw distance of 20 mm and a machine speed of 50 mm/min. When the cards were actually used in the Instron tensile tester in order to measure the breaking load, the fibers started slipping through the card and it became hard to take measurements. The experiment was altered by clamping the fibers directly in the machine jaws. The distance between jaws was 15mm while the load cell of 5N was used. Another problem of fiber sample preparation was faced at this stage. It was difficult to obtain single fibers. The experiments were performed manually to obtain the maximum possible separation of the fibers leading to single or multiple fibers which seemed to be single to the naked eye. The trial was successful and there was no more fiber slippage through the jaws. Because of the uneven surface of the fiber bundle (considered as single fiber) breakage was occurring from the point where there were a lesser number of fibers across the length. Therefore the load did not represent the actual breaking load of the fiber. This problem was compensated by measuring the diameter near to the breaking point or the surrounding area of the breaking point of the fiber (or bundle). Glass slides were prepared at the same time, after the testing, to measure the diameter of the respective fibers. By using diameter and breaking

load, the tenacity was determined, which indicated the fiber's strength per unit linear density.

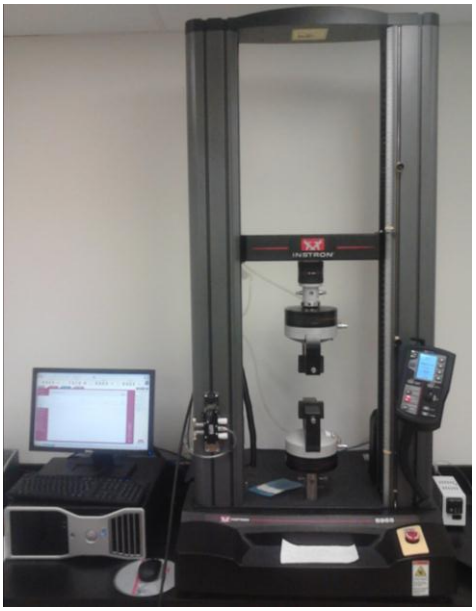


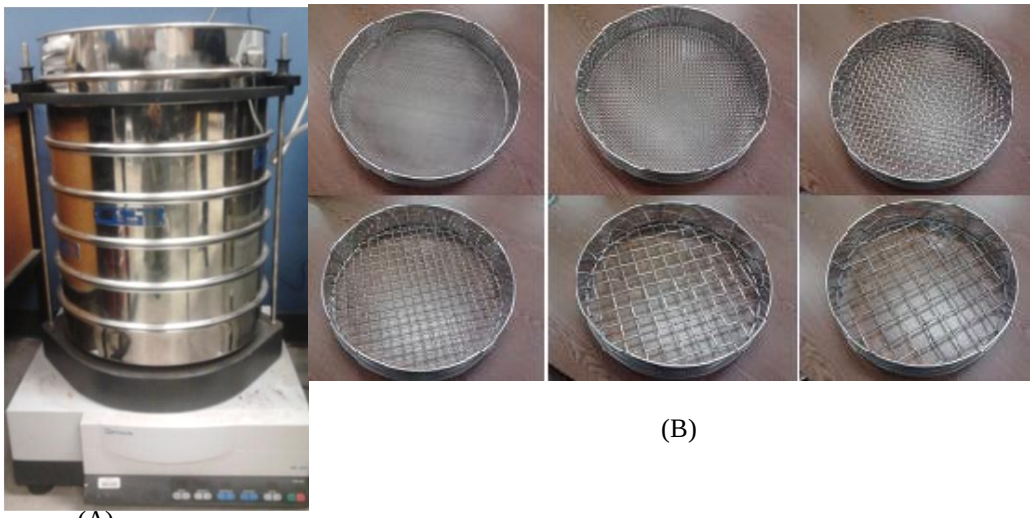
Figure 2.4: Instron Tensile Tester

2.2.4 **Individualization of Fibers**

The fiber bundles were also separated after various chemical treatments by using different mechanical means including a sieve shaker, a planetary mono mill and a manual card machine. The processing of fiber bundles through these machines is detailed in the following sections.

2.2.4.1 **Sieve Shaker**

After conditioning, an attempt was made to separate the entangled fibers by using a "Horizontal Sieve Shaker" manufactured by Retsch, Germany (Figure 2.5A). This machine (Model: AS-400 control) consists of six sieves (Figure 2.5B) and a collecting pan at the bottom which revolves around a horizontal circular axis. There are six different sieves (pans) with different wire opening. Any of these sieves can be used depending on size of the required output material. Maximum grain size which can be used is 63 mm and usually it takes 3-5 min for one operation cycle.



(A) (B)

Figure 2.5: A) Horizontal Sieve Shaker, B) Six Sieve

2.2.4.2 **Planetary Mono Mill**

A Planetary Mono Mill (Figure 2.6A) manufactured by Fritsch-Germany was also used to separate the hemp fiber bundles. This machine uses small balls (Figure 2.6B) which are placed in a metallic bowl, rotating in a counterclockwise direction. When the bowl rotates the fibers, a beating (grinding) action is performed by the marble balls inside. Due to this

beating the bundle is loosened. The maximum feed size is 10 mm while maximum feed quantity is 225 ml.



(A)



(B)

Figure 2.6: A) Planetary Mono Mill, B) Marble Balls

2.2.4.3 **Card Machine**

The Card machine is known as the heart of yarn manufacturing process. One of its functions is fiber to fiber opening. A manual card machine available in the Department of Biosystems Engineering, University of Manitoba, as shown in Figure 2.7, was used. Technical data of card machine is given in Table 2.3. Although there was no doffer on the machine, however it could perform carding adequately. After chemical treatments it was decided to card the fibers/bundles. For this purpose the fibers were passed through the card machine and operated manually. Some of the small fibers fell down the machine and some were dropped on the other side which were collected while most of them were held

in the wires of the cylinder and feed roller which were collected later by using small wooden sticks.



Figure 2.7: Manual card machine (Biosystems Engineering dep't, U of Manitoba)

Table 2.3: Specification of manual carding machine

Parameter	Value
Diameter of Cylinder (cm)	18
Diameter of Taker-in (cm)	4.5
Wire type	Metallic
Cylinder Wires Density /cm²	7
Taker-in Wires Density /cm²	6
Cylinder Speed (rpm)	44
Taker-in Speed (rpm)	6.5
Cylinder Wire length (cm)	1
Cylinder Wire length (cm)	1

2.2.5 **Weight Loss Percentage**

Weight loss represents the amount of non-cellulosic material removed from the fibers. For this purpose every sample was conditioned in standard atmospheric conditions before and after treatment for at least 24 hours and then weighed by using an electronic weighing machine. Weight loss (%) was calculated by using the equation 1.

Equation 1:

$$\text{Weight loss (\%)} = \frac{(A-B)}{A} * 100$$

Where;

A= Weight before treatment (g)

B= Weight after treatment (g)

2.2.6 **Moisture Regain**

Moisture regain is the amount of moisture expressed as the oven dry weight of the material [8]. To calculate it, the samples were first conditioned at standard atmospheric conditions for 24 hours and weighed after treatment. Then the same samples were dried in the oven (Thermo scientific oven) at 105°C. The weight of fibers was measured after one hour and then after every 30 min interval until the resultant weight became constant and weighed the same for at least three consecutive time intervals. The moisture regain was calculated according to equation 2.

Equation 2:

$$\text{Moisture Regain (\%)} = \frac{(C-D)}{C} * 100$$

Where;

C= Wet fiber weight (g)

D= Oven dry fiber weight (g)

2.2.7 **Surface Modification Procedures**

Surface modification of hemp fibers was carried out in two phases. In phase-I three types of treatments were carried out including water, chemical and enzyme. For all of these, Launder-o-meter (SDL-Atlas, Figure 2.8), was used with a machine speed of 40 ± 2 rpm and pre-determined set of time and temperature [42]. In some experiments steel balls

were also added in the canisters in order to create severe conditions. The detail of all chemical treatments is summarized in Table 2.2.



Figure 2.8: Launder-o-meter with machine terminal & canister on top

2.2.7.1 **Trial I & II**

First of all it was deiced to have a trial with water. Decorticated hemp fibers were cut to a length of 30 mm (using a paper cutter machine), conditioned at standard atmospheric conditions (Temperature 20°C, Relative humidity 65%) and weighed through an electronic weighing machine. A fiber bundle (0.760 g) was treated with water using the launder-o-meter. The treatment was carried out according to the recipe as detailed in Table 2.4.

After treatment, the fibers were still joined with each other although they had encountered hot water. After drying, another attempt was made to soften the intermingled fibers by washing with normal tap water and drying at room temperature. Samples were reweighed to calculate the weight loss.

Textile materials are made hydrophilic and waxes are removed by applying a scouring process to them. Scouring was carried out for these fibers by using a launder-o-meter with (AATCC) detergent. After scouring the fiber bundles were treated with alkali (sodium hydroxide, NaOH) solution to improve the absorbency and uptake of the material. To neutralize the effect of alkali in the treated fibers, the bundles were treated with 0.1% solution of acetic acid for 10 min. Afterwards the sample was placed in tap water for 10 min to remove the effect of applied chemicals.

At the end of this process, the fibers were conditioned at standard atmospheric conditions (20°C, 65% relative humidity). After conditioning, an attempt was made to separate the entangled fibers by using a "Horizontal Sieve Shaker" (Figure 2.5A). After passing through this machine, the same fiber bundle was then processed by using a Planetary Mono Mill (Figure 2.6A) manufactured by Fritsch-Germany.

Table 2.4: Procedure of chemical treatments

Trial #	Process/ Treatment	Chemicals and concentration	Time (min)	Temp. (°C)	Sample ID
I	Water	Distilled Water	60	60	E1WT
II	Water	Distilled Water	60	60	E1W1T
	Scouring	AATCC Detergent -1%	90	55	
	Alkali	NaOH-3%	120	57	
	Neutralization with 0.1% acetic acid & washing with tap water				
III	Alkaline → Acid	NaOH – 5% / H_2SO_4-5%	240	87 & 45	E2ALT
IV	Acid → Alkaline	H_2SO_4-5% / NaOH – 5%	240	40 & 92	E2ACT
V	Alcoholic Methanol (CH_3OH)	NaOH – 1%	60		Not used for further analysis
VI	Alcoholic Methanol (CH_3OH)	NaOH – 5% with 50 steel balls	120		E3MT
VII	Detergent wash	AATCC Detergent -5%	30	42	E4ST
	Acid	Sulphuric Acid-10%	240	47	
	Washing with Tap Water				
	Alkaline	[1]Na_2CO_3 – 2.3% / [2]Na_2O_3S – 0.6% / [3]$C_{12}H_{25}NaO_4S$-0.08%	60	90	
	Bleaching (Distilled Water)	[4]H_2O_2-0.88ml/L / NaOH-0.05 gm/L / Glycerin (Wetting Agent)- 0.2 ml/L	120	90	
	Neutralization with 0.1% acetic acid & washing with tap water				
	Washing with Tap Water				

[1]Sodium carbonate , [2] Sodium sulphite, [3] Sodium deodecyle sulphate, [4] Hydrogen peroxide

In order to treat the fibers through trial III & IV, two different bundles of fibers each with the length of 40 mm were cut by using paper cutting machine and conditioned in standard atmospheric conditions. Samples were treated by using same chemicals in the launder-o-

meter but by following two different routes, as detailed below in sections 2.2.7.2 and 2.2.7.3.

2.2.7.2 **Trial III**

An alkali solution was prepared and heated by using guarded hot plate apparatus (Figure 2.9). The fiber bundle was treated in this solution. The treatment recipe and conditions are given in Table 2.4. These fibers were placed into tap water for 10 min to neutralize. They were dried, conditioned in a standard atmospheric environment and reweighed. As the desired results were not achieved after this treatment, this alkali treated sample was then treated with an acidic solution of sulphuric acid, the recipe of which is given in Table 2.4. After chemical treatment, the fiber bundle was conditioned in a standard environment and re-weighed to calculate the weight loss/gain percentage.

2.2.7.3 **Trial IV**

The second conditioned sample was treated with the reverse route as of trial III i.e. first treated by acidic and then by alkaline solution according to the conditions given in Table 2.4. Treated fibers were dried at room temperature, conditioned at standard conditions and re-weighed to calculate the weight loss/gain.



Figure 2.9: Guarded hot plate apparatus

In the second step, the same (acid treated) sample was treated with alkali according to the recipe given in Table 2.4. Fiber samples were conditioned again in standard atmospheric conditions and reweighed to calculate the weight loss/gain.

2.2.7.4 **Trial V & VI**

A hemp fiber bundle with a length of 40mm was treated with 1% solution of sodium hydroxide in methanol (methyl hydroxide, CH_3OH) as a solvent. Methanol is the colorless and simplest form of alcohol, usually used as a solvent. It was a trial to check if it would perform separation of the fibers. First the fibers were treated in the solution (Table 2.4) in two cycles each of 30 min in the launder-o-meter. After the first cycle, the same bundle was again treated with same conditions. In order to achieve the results, the experiment was revised with little change in conditions.

The concentration of alkali was increased from 1 to 5% and the time cycle was increased from 1 to 2 hours. Fifty (50) steel balls were also added in order to increase the intensity of the treatment. Even after such severe conditions there was not much change in bundle separation.

2.2.7.5 **Trial VII**

It was decided to scour the fibers to test if it would help to achieve the required results. The detail of treatment route and recipe is given in Table 2.4. Through this treatment the fibers were scoured with acidic solution. Fibers were first washed by using AATCC

detergent. After the detergent wash, the fibers were washed with tap water in order to remove the detergent completely.

After washing, the fibers were treated with 10% solution of sulphuric acid for four hours in the launder-o-meter. After this treatment, the sample was again washed in tap water to remove the chemicals, dried at room temperature and conditioned in standard atmospheric conditions for 24 hours. The same fiber sample was then treated with alkaline solution at 90°C for one hour. Sodium carbonate was used to soften the water. Sodium sulphate was used to increase the intensity of liquid penetration into the fiber surface. Sodium dodecyle was used as a surfactant to remove oily substances from the fiber surface.

Fibers were bleached so that the luster of the fiber can be improved. For this purpose a bleaching solution was prepared and fibers were treated with the solution at 90°C for two hours. Distilled water was used as a solvent while hydrogen per oxide was used as a bleaching agent. A strong alkali, sodium hydroxide was added. In order to increase the penetration of the alkali and bleaching agent, glycerin was used as a wetting agent. After scouring the fiber sample were neutralized with 0.1% acetic acid, washed with tap water, dried at room temperature and conditioned at standard atmospheric conditions. After chemical treatment fibers were passed through the manual card machine (Figure 2.7) to make them separate.

2.2.8 **Washing Sample after Treatments**

After chemical treatments (procedures are described in section 2.6) the fibers were washed to neutralize them. A ceramic funnel along with a glass beaker was used for this purpose. Samples were dropped into the funnel, which was placed on top of the beaker. All of the solution (acidic, alkaline etc.) was collected in the beaker which was stored for proper disposal. Next the treated fibers needed to be washed. In the funnel the collected fiber sample was placed beneath running tap water for a defined time (10 min in most of the cases). Due to a continuous flow of water, the samples were neutralized. Afterwards, the sample was collected from the ceramic funnel and processed to further treatment.

Since sodium hydroxide is a strong alkali hence used to neutralize the fibers treated with this alkali, they were first neutralized with acetic acid then washed (Figure 2.10) with water according to the procedure mentioned above.



Figure 2.10: Washing set up for fibers

2.2.9 **Enzyme Treatment**

Enzymes are the compounds used as a catalyst for any chemical or biological reactions. In this research enzymes were used for getting rid of pectin and lignin in hemp. Given the

results of the earlier chemical experiments, it was decided to conduct trials with enzymes. A bundle of decorticated hemp fibers was scoured to prepare for further treatment with enzymes. Fibers having length of 30 mm were scoured with 3% solution of sodium carbonate at 60˚C for 90 min in a 300 ml solution. A water solution of pH 5.84 was prepared by using (70%) sulphuric acid.

Two different enzymes Enzeco Pectinase DV-2 (by Enzyme Development Corporation-USA) and pectinase from aspergillus aculeatus (Sigma Aldrich-USA) were used so that their reactivity could be evaluated for future uses. The fibers (E5ET and EPET, Table 2.5) were treated with enzymes by using the Launder-o-meter. After treatment, the fibers were washed with tap water in order to neutralize them, dried at room temperature and finally the weight loss/gain was calculated.

Next, in order to check the effect of different variables on the resultant hemp fibers (sample EPET-1 to 7), the decorticated fibers were treated with different percentages of enzymes. These fibers were scoured with 3% sodium carbonate at 60°C for 90 min by using 400 ml of water, prior to treatment with enzymes. After scouring these fibers were neutralized with tap water for 10 min, dried at room temperature and finally conditioned at standard atmospheric conditions for 24 hours. Now this scoured sample was treated with enzyme (Pectinase from Aspergillus Aculeatus) in acidic conditions for different time intervals according to the recipe given in Table 2.5.

Table 2.5: Conditions for enzyme treatment

Enzyme Conc. (%)	**Enzyme**	**Media**	**Temperature** (°C)	**pH**	**Time** (min)	**Sample ID**
1	Enzeco Pectinase DV-2	Acidic	50 (+50 steel balls)	5.84	480	E5ET
1	Pectinase Aspergillus Aculeatus	Acidic	50 (+50 steel balls)	5.84	480	EPET
1		Acidic	50	5.63	60	E6PET-1
1		Acidic	50	5.63	120	E6PET-2
1		Acidic	50	5.63	240	E6PET-3
1		Neutral	50	7.66	240	E6PET-4
1		Alkaline	50	9.06	240	E6PET-5
2		Acidic	50	5.58	240	E6PET-6
1		Acidic	60	5.67	240	E6PET-7

2.2.10 **Softness Measurement** (Subjective Method)

Subjective method of softness evaluation was used to evaluate the softness of chemical and enzyme treated hemp fibers. Test method AATCC 5-2011 [56] was used as a reference to develop the test method for evaluation. Cotton, wool, flax, olefin and decorticated hemp fibers were considered as standard for ranking as given in Table 2.6. These cotton, wool, flax and olefin fibers were taken from the samples provided with the lab manual written by Katherine, L. Hatch [57]. These fiber samples are used for undergraduate textile students' lab work at the University of Manitoba. A numeric scale was developed for this purpose with the rank ranging between 1 and 5, where 1 indicates

the lowest softness while 5 indicated the highest softness. Observers were made familiarized with the standard ranking of softness. After being familiarized, observers evaluated the softness by touching the fiber bundles. Each observer assigned a numeric value to each sample based on their feeling about softness. For a specific sample an average of all observations was calculated in order to assign the final ranking.

Table 2.6: Standard for softness ranking

Fiber	**Ranking**
Cotton	5
Wool	4
Flax	3
Olefin	2
Decorticated Hemp fiber	1

Softness was evaluated in two phases. In phase-I, softness of two chemically treated fiber samples (ID: E2ACT and E4ST) and one enzyme treated fiber sample (ID: E6PET 6) was evaluated. In phase-II, softness of twelve bleached samples were evaluated.

2.3 Optimization of Bleaching Process

Hemp surface modification was carried out in two phases, as earlier discussed in section 2.2.7. Based on results of preliminary experiments (Phase-I) it was decided to use chemical treatment method rather than enzyme in phase-II. For this purpose bleaching process was selected to focus for further research and bleaching parameters were optimized to achieve results of hemp spinning properties.

Pretreatments and bleaching was carried out according to the recipe given in Table 2.4 (Trial VII, section 2.2.7.5). For the optimum bleaching process three levels of temperature i.e. 60°C, 80°C and 95°C and three levels of time i.e. 120 min, 180 min and 240 min were considered (Table 2.7). A full factorial design with four replicates was developed and thirty six (36) experiments were designed. The experiments were carried out in a random order to increase the validity and reliability of the process.

Table 2.7: Three levels of time and temperature for bleaching

Level	**Time** (min)	**Temperature** (°C)
1	120	60
2	180	80
3	240	95

2.3.1 Experimental Procedure

The details of sample preparation (for example, conditioning and scouring) and bleaching process are discussed in this section. To standardize the results, fiber samples were

conditioned before any measurement and experiments at 65% relative humidity (RH) and 21°C for at least 24 hours, according to the standard procedure developed by ASTM D 1776 [53].

Prior to bleaching, fibers were washed with detergent in order to remove the impurities and then scoured with acid. This process is required for the removal of wax, grease and for degradation of lignin. The detergent washed and scoured samples were then rinsed with tap water to neutralize the acid. The acid scoured samples were then treated with alkali for pectin removal. These preparatory processes are necessary for effective removal of pectin by bleaching. The bleached fibers were neutralized with acetic acid and finally rinsed in tap water.

Chapter 3

Results and Discussion

Preliminary investigation was carried out for understanding impact of different fiber characteristics on six spinning fiber parameters. After treatment, the data analysis was focused on the properties of fibers required to make them easy to be processed through cotton spinning systems to produce a finer count, suitable for apparel applications. For this purpose weight loss (%), length variation, different mechanical properties, stiffness, single fiber entity and moisture regain of the chemical and enzyme treated fibers were analyzed.

3.1 Weight loss (%)

Both of the chemical and enzyme treated fibers were analyzed for their weight loss (%) and results are detailed here.

3.1.1 Chemical Treated Fibers

The weight loss (%) of chemically treated fibers is given in Table 3.1 and presented in Figure 3.1. As stated in Chapter 2, section 2.2.5, weight loss represents the amount of non-cellulosic material removed from the fiber which indicates that more weight loss means more effective is the treatment. Hence the objective of measuring the weight loss (%) was to understand the phenomenon of non-cellulosic materials removal from the hemp fiber.

Table 3.1: Weight loss (%) of fibers treated with chemical

Sample ID	**Weight loss** (%)
Raw Hemp	0.00
EIWT/I (Water)	13.03
E2ALT/III (alkali→acid)	34.72
E2ACT/IV (acid→alkali)	36.49
E3MT/V (methanol)	19.48
E4ST/VII (Bleached)	37.42

After each treatment, the solution color changed to yellow, indicating that the impurities have been removed from the fibers. Hot water treatment (E1WT) of fibers removed 13.03% non-cellulosic material. Alkaline followed by acid treatment (E2ALT) lost 34.72% of the fibers weight whereas the reverse treatment i.e. acid followed by alkaline treatment (E2ACT) removed 36.49% of the weight. Alkaline methanol treatment removed non-cellulosic material in the range of 20%. Bleached fibers (E4ST) lost 37.42% of weight. It can be observed that E4ST, E2ACT and E2ALT treatments lost comparatively more weight. It seems that these treatments removed more pectin and waxes. There is a slight difference of 1.77% in weight loss between E2ALT and E2ACT although in both cases the treatment conditions were the same except the treatment order. Hemp contains 67-75% cellulosic content, in other words 37-25% of non-cellulosic material [16]. It appears that E4ST, E2ALT and E2ACT treatments removed the maximum amount of non-cellulosic material.

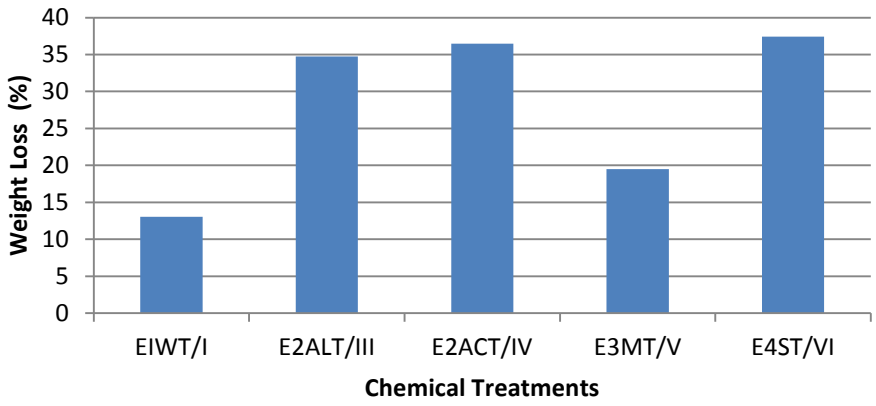


Figure 3.1: Weight loss (%) by different chemical treatment

3.1.2 **Enzyme Treated Fibers**

Weight loss by treating the fibers with enzymes in different media (aqueous, acidic and alkaline) is presented in Table 3.2. In case of treatment with 1% Enzeco Pectinase DV-2 (E5ET) at 50°C for eight hours with addition of fifty (50) steel balls, 8.27% weight was reduced while in the case of fibers treated with Pectinase from Aspergillus Aculeatus (EPET) all fibers dissolved completely in the solution in the same treatment conditions. The series of experiments with different treatment conditions and enzyme concentrations resulted in different amounts of weight loss.

Fibers treated with enzymes in acidic media (enzyme concentration: 1.0%) for 60 min at 50°C (E6PET-1) lost 4.59% of weight while E6PET-2 and E6PET-3 reduced 6.50% and 9.81% of their weight for 120 min and 240 min respectively for the same treatment condition. It appears that the weight loss (%) for Pectinase Aspergillus Aculeatus enzyme is linear with the treatment time with a coefficient of determination (R^2) value of 0.998, as shown in Figure 3.2.

Table 3.2: Weight loss (%) of the enzyme treated fibers

Sample ID	**Media**	**Weight loss** (%)	**Sample ID**	**Media**	**Weight loss** (%)
E5ET	Acidic	8.27	E6PET-4	Neutral	13.69
E6PET-1	Acidic	4.59	E6PET-5	Alkaline	2.61
E6PET-2	Acidic	6.50	E6PET-6	Acidic	18.40
E6PET-3	Acidic	9.81	E6PET-7	Acidic	5.87

The weight loss (%) of fibers treated with 2% Pectinase Aspergillus Aculeatus enzyme for 240 min at 50°C temperature (E6PET-6) is 18.40%, which is almost double the sample (E6PET-3, weight loss: 9.81%) treated in 1% Pectinase Aspergillus Aculeatus enzyme in the same treatment conditions. The sample (E6PET-7) treated at 60°C for 240 min in 1% Pectinase Aspergillus Aculeatus enzyme lost 5.87% of its weight. For the similar treatment conditions at 50°C temperature fiber (E6PET-2) lost 1.7 times more weight. It seems that the optimum treatment temperature in terms of weight loss (%) is about 50°C. It can be stated that at and below this temperature, the rate of weight loss is slower.

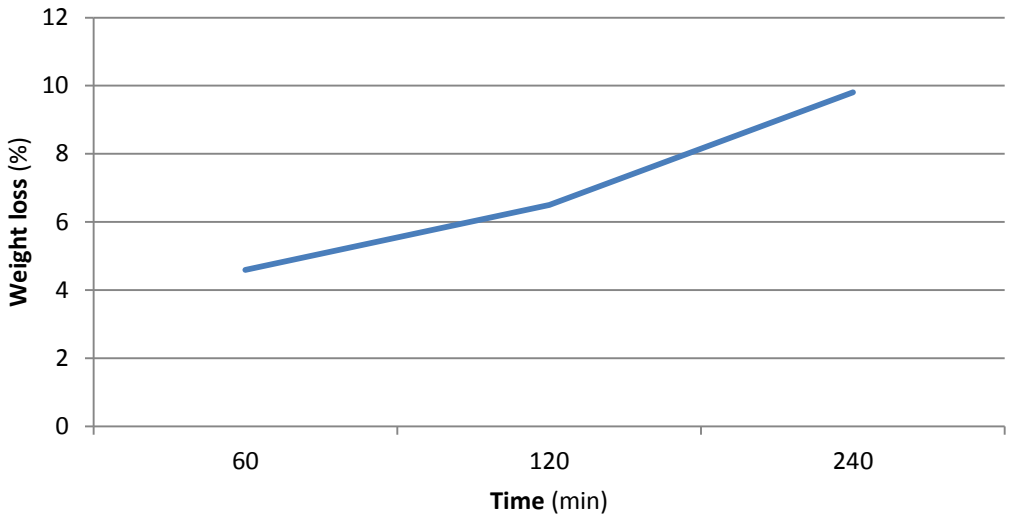


Figure 3.2: Effect of treatment time on Weight loss (%) (Enzyme concentration: 1.0%)

Fibers treated in a neutral environment or aqueous medium (E6PET-4) lost 13.69% weight at 50°C for four hours. For the same treatment conditions (50°C/4hours) in an alkaline media the fiber (E6PET-5) lost 2.61 % weight.

Samples E6PET-4, E6PET-3 and E6PET-5 were treated in similar conditions (50°C/240 min/1% Pectinase Aspergillus Aculeatus) in aqueous, acidic and alkaline media. The weight loss (%) for these samples were 13.69% (E6PET-4). 9.81% (E6PET-3) and 2.61% (E6PET-5). From this weight loss data it can be stated that a neutral environment is the most effective in order to remove non-cellulosic materials from hemp fiber. Three samples E5ET, E5PET and EPET were also treated with the addition of steel balls in the solution. The resultant fibers were broken and entangled and no further analysis was carried out with these samples.

3.2 Fiber Length Variation

After treatment, the length of fibers was measured manually by using a ruler. The change in length is 0-3 mm (<1/8 inch) in case of both chemical and enzyme treatments which indicates that there is no breakage of fibers during treatments. It is a positive indicator as the length variation should not be more than 1/8 inch in order to make hemp fibers spinable through cotton spinning system.

3.3 Mechanical Properties

The mechanical properties of chemical and enzyme treated fibers are presented in Table 3.3 and 3.4 respectively. All of the chemical treatments resulted loss in breaking load strength. Fiber sample (E1W1T) which was treated in a scouring solution (water + detergent + alkali) lost 73.05% of breaking load which is the highest among chemically treated fibers. E2ALT (Alkali → Acid) and E2ACT (Acid → Alkali) lost 55.73% and 53.96% of load respectively while the methanol treated fibers lost 64.94% of breaking load. The bleached samples (E4ST: acid + alkali + bleaching) lost 71.05% of breaking load. Enzyme treatment also affected the breaking load of the fibers. E6PET-3 encountered 67.59% reduction in breaking load and E6PET-4 lost 73.14% while E6PET-5 lost the least amount of breaking load strength (21.18%).

Table 3.3: Mechanical properties of chemically treated hemp fibers

Sample ID	**Breaking load** (N)	**Breaking load loss** (%)	**Diameter** (μm)	**Tenacity** (N/tex)	**Tenacity loss** (%)	**Weight loss** (%)	**Weight loss (%) / Tenacity Loss (%)**
E1W1T	0.47 ± 0.26	73.05	83.19	0.06	70.80	--	--
E2ALT	0.78 ± 0.56	55.73	60.49	0.18	9.14	34.72	3.80
E2ACT	0.81 ± 0.47	53.96	69.70	0.14	29.00	36.49	1.26
E3MT	0.62 ± 0.33	64.94	93.50	0.06	69.71	19.48	0.28
E4ST	0.55 ± 0.27	71.05	127.48	0.03	87.09	37.42	0.43

Table 3.4: Mechanical properties of enzyme treated hemp fibers

Sample ID	**Breaking load** (N)	**Breaking load loss** (%)	**Diameter** (μm)	**Tenacity** (N/tex)	**Tenacity loss** (%)	**Weight loss** (%)	**Weight loss** (%) **/ Tenacity Loss** (%)
E6PET-3	0.57 ± 0.38	67.59	132.08	0.03	86.10	9.81	0.11
E6PET-4	0.47 ± 0.32	73.14	144.73	0.02	90.37	13.69	0.15
E6PET-5	1.38 ± 0.72	21.18	134.36	0.07	67.28	2.61	0.04

From the Table 3.3 and 3.4 it can also be observed that the standard deviations of the breaking load data were very high for treated fibers. Also, the diameter of bleached (E4ST) and enzyme treated samples (E6PET-3, E6PET-4, E6PET-5) were much higher than the virgin samples due to the use of samples from a different batch. Consequently, instead of aspect ratio (length/diameter), tenacity and loss in tenacity were calculated which are shown in Table 3.3 and 3.4.

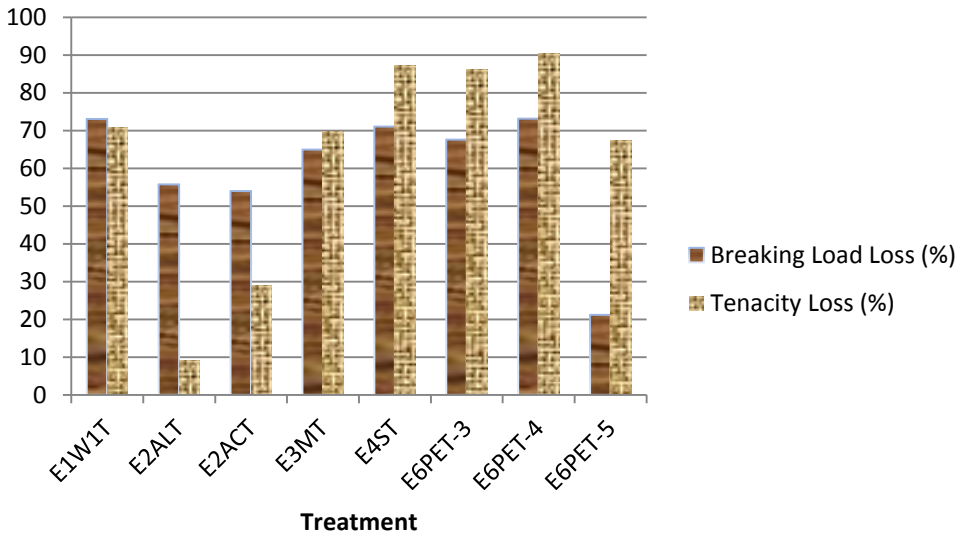


Figure 3.3: Breaking Load (%) and tenacity Loss (%) of different treated fibers.

To calculate tenacity, first the breaking load of the fiber was determined and the diameter of each broken fiber from ten different places was measured using the Bioquant Life Sciences system. Taking the average fiber (average of 100 data points) diameter, the linear density (tex) of fibers was calculated using the equation 3.

Equation 3:

Linear Density (tex) = Weight in grams (mass) for 1000 meters of fiber

Weight in grams (mass) = density X volume (ρ X V)

Where,
Density (ρ) = 1.48 and Volume (V) = ($\pi d^2/4$) x h

Where,

d = diameter of fiber
h = length of the fiber (as fiber is considered like a cylinder)

For raw hemp;

Linear Density (tex) = Weight in grams (mass) for 1000 meters of fiber

Weight in grams = density x Volume (g)

$= \rho \times V \quad (g)$

$= \rho \times (\pi d^2/4) \times h \quad (g)$

As

Diameter (d) = 86.61 µm

Linear Density = 0.20 N/tex (B)

It can be observed from Table 3.3 and 3.4 that most of the treatments resulted in an immense tenacity loss. The scoured samples (E1W1T) lost 70.80% tenacity whereas the alkali treated hemp fibers (E2ALT: weight loss: 34.72) lost only 9.14%. The acid treated (E2ACT: weight loss: 36.49%), methanol treated (E3MT: weight loss: 19.48%), and bleached fibers (E4ST: weight loss: 37.42%) encountered 29.00%, 69.71% and 87.09% reduction in tenacity respectively.

Although two chemical treatments, E2ALT and E2ACT, lost higher amount of weight but they lost least tenacity among all chemically treated fibers. Further, the absolute tenacity values for these two treated fibers were 0.18 N/tex (E2ALT) and 0.14 N/tex (E2ACT). These tenacity values are similar to the tenacity values of virgin acetate (0.12 N/tex) and silk (0.158 N/tex) fibers and much higher than the virgin wool fibers (0.06 N/tex) in standard atmospheric conditions (65% relative humidity and 21°C) [42]. Cotton is comparatively stronger as its tenacity in standard conditions is 0.35 N/tex [2].With this

tenacity range (0.14-0.158 N/tex), hemp fibers can be spun using cotton ring spinning system.

It can be observed from the graph (Figure 3.3) that enzyme treated fibers in alkaline medium (E6PET-5: weight loss: 2.61%) lost least tenacity 67.28% and E6PET-4 (weight loss: 13.69%) the highest 90.37% while E6PET-3 (weight loss: 9.81%) lost 86.10% for the similar treatment condition. Weight loss (%), breaking load and tenacity losses for the enzyme treated fibers are much higher than the chemically treated fibers.

3.3.1 **Ratio of Weight Loss (%) to Tenacity Loss (%)**

The ratio of weight loss (%) to tenacity loss (%) for chemical and enzyme treatments is shown in Table 3.3 and 3.4. The ratio for E2ALT (alkali → acid), E2ACT (acid → alkali), E3MT (methanol) and E4ST (bleached) samples is 3.80, 1.26, 0.28 and 0.43 respectively. For enzyme treated fibers the ratio is 0.11, 0.15 and 0.04 for E6PET-3 (acidic medium), E6PET-4 (neutral medium) and E6PET-5 (alkaline medium).

Usually if the chemicals and enzymes do not penetrate inside the fiber and break the chemical bonds, the weight loss (%) should be proportional to the tenacity loss (%) and the ratio should be 1. In the case of E2ALT and E2ACT samples, the ratio was 3.80 and 1.26 which indicates that these two chemical treatments only removed non-cellulosic materials and did not damage the internal structure. Methanol treatment resulted in the weight loss to tenacity loss ratio of less than 1 (experimental value: 0.28) which indicates that the tenacity loss (%) was more than weight loss (%). Similar results for the alcoholic solution were obtained for other textile fibers [58]. The ratio for enzyme treated fibers

was also 0.11, 0.15 and 0.04 for E6PET-3 (acidic medium), E6PET-4 (neutral medium) and E6PET-5 (alkaline medium) respectively. This ratio suggests that in addition to removing non-cellulosic materials, enzymes damaged the internal structure of the hemp fibers.

Textile fibers consist of both crystalline and amorphous regions as shown in Figure 3.4 and 3.5. The presence of amorphous regions makes the fibers more prone to be affected by any of the chemicals in terms of strength, weight, tenacity etc. During enzyme treatments, the functional group of enzymes penetrates inside the structure and damages the chemical bonds and amorphous regions. Moisture regain data also supports this hypothesis which will be discussed in a later section.

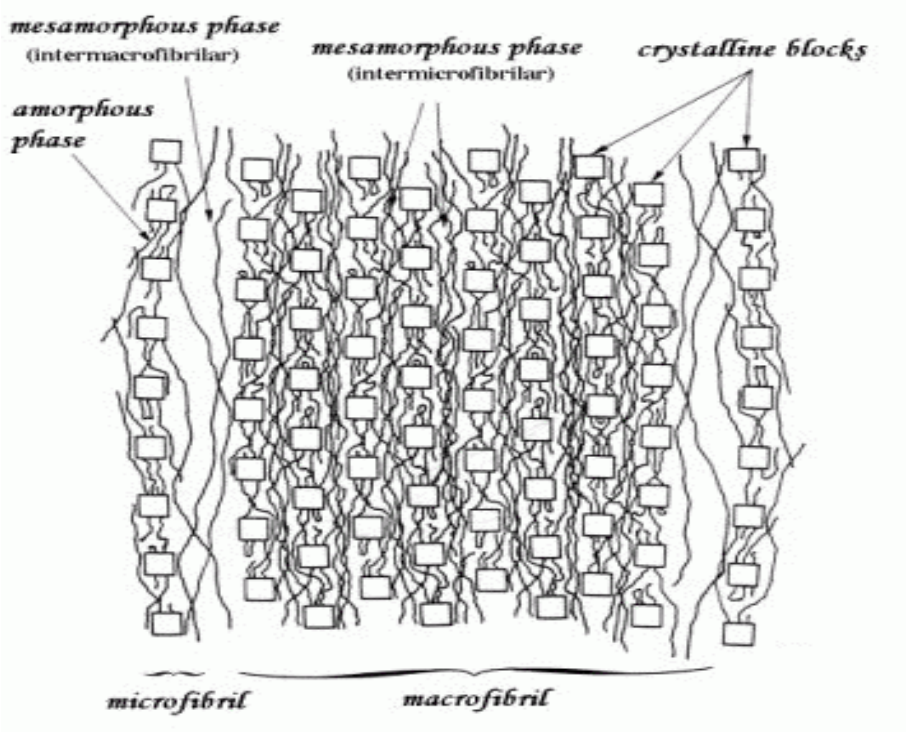


Figure 3.4: Fibrillar structural model for PET (Polyethylene Terephthalate) fibers. Printed with kind permission from Springer science and business media [59]

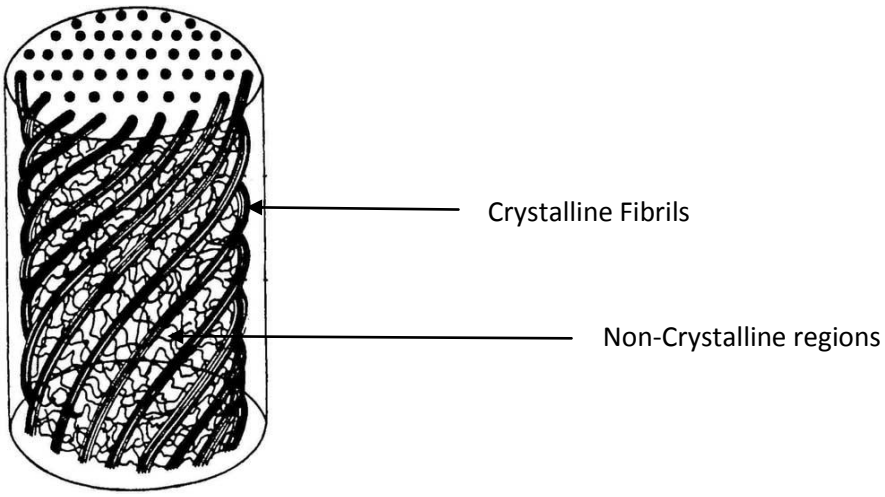


Figure 3.5: Principle of the flax fiber structure. Helical arrangement of fibrils in natural cellulose fiber [60]

3.4 Stiffness and Softness

Cotton spinning systems require the fibers which are easy to bend so that they do not break due to torsional tension while twisting at different stages of the process, particularly in the ring frame machine. The spindle in a ring frame machine rotates at a speed of 25,000-30,000 rpm in order to insert twist [36]. About 20-30 twist per inch is inserted in a 40 Ne yarn as twist per inch (TPI) is TPI = $5.0\sqrt{Ne}$, where, twist factor is 5.0 and Ne is the yarn number in the English cotton system [37]. One of the objectives in treating the fibers with different chemicals and enzymes was to reduce the stiffness of the fibers. Usually, breaking twist angle (BTA) is measured to evaluate the ability of fibers to withstand the twisting. However, the testing equipment to measure BTA was not available in the department; instead a mechanical twister was used to determine the breaking twist (number of twist required to break the fibers). It was found that although both chemical and enzyme treatments improved the breaking twist of hemp, but the treated hemp fibers required fewer twists than cotton fibers to reach the break point

which indicates that the fibers were more brittle and less flexible [61]. Fibers needed to be more flexible to avoid any breakage while twisting through the drafting system of a spinning machine.

A subjective rating was conducted to compare the stiffness of the treated fibers. For this purpose, the guidelines were taken from the test method AATCC 5-2011 [56]. This method deals with fabric evaluation, but it was revised to evaluate the fiber stiffness. Three treated samples, E2ACT (acid → alkali), E6PET-6 (enzyme treated) and E4ST (Bleached) were ranked by three observers (A, B and C). Cotton, flax, and olefin fibers were used for comparison.

Three features of these fibers including softness (stiffness), fiber separation and fiber stickiness were ranked. The scale runs from 5 to 1 where 5 indicate the highest softness while 1 indicates the highest stiffness. In a similar fashion for stickiness, 5 indicate no stickiness while 1 indicates highly sticky. Fiber separation is also ranked on the same pattern as that of stickiness and softness. In this context, 5 indicate higher separation while 1 shows the least separation. The rating results are shown in Table 3.5.

Table 3.5: Comparison of fiber softness, stickiness and separation

Fiber	Ranking for Softness				Ranking for Stickiness				Ranking for separation			
	A	B	C	Average	A	B	C	Average	A	B	C	Average
Cotton	5	5	5	5	4	5	5	4.7	5	5	5	5
Flax	4	4	4	4	4	5	5	4.7	4	1	5	3.3
Olefin	3	3	3	3	5	4	4	4.3	4.5	4	4	4.2
E2ACT	2	2	2	2	2	1	2	1.7	2	1	2	1.7
E6PET-6	1	1	2	1.3	2	1	1	1.3	3	3	3	3.0
E4ST	3	3	3	3	3	2	1	2	3	4	4	3.7

It is clear from Table 3.5 that cotton is highly soft with a ranking of 5, followed by flax and olefin with a ranking of 4 and 3. Among treated fibers E6PET-6 is the least soft (ranking 1.33) while E2ACT is comparatively softer (ranking 2) whereas bleached and carded fibers (E4ST) are the best with highest ranking of 3 among the treated fibers. E4ST are the softest fibers among all chemical and enzyme treated fibers and has ranking similar to olefin fiber.

The phenomenon of stickiness was also observed in the same way as that of softness (Table 3.5). Cotton and flax were found least sticky followed by olefin with ranking of 4.7, 4.7 and 4.3 respectively. Treated fibers E2ACT (acid → alkali) and E6PET-6 (enzyme treated) have a ranking of 1.7 and 1.3 respectively while bleached fibers (E4ST) have an average of 2. It shows that E4ST is comparatively less sticky as compared to other treated fibers.

3.5 **Fiber Individualization**

For better quality of fabric the yarn should be even throughout the length. Although they seemed to be separated after treatments, multiple fibers within a fiber could be noticed under a microscope (Figure 3.6).

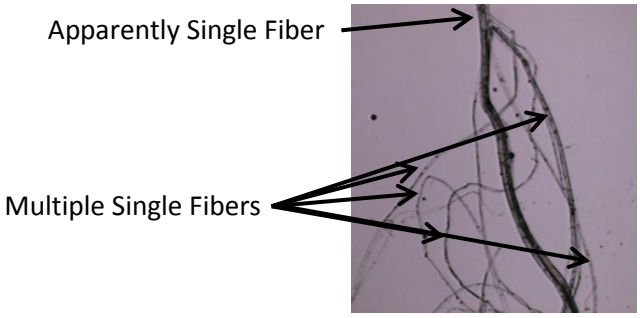


Figure 3.6: Presence of fibers in bundle form after surface modifications (Sample ID: E4ST)

It seemed that the bleached fibers (E4ST) were separate. But when observed through a microscope it was found that they were individual as compared to other treated fibers but degree of individualization was very less. It was attempted to make them separate by using the carding machine. However the fiber bundle was small which created some difficulty while processing through the carding machine.

The phenomenon of fiber separation or individualization was also analyzed through a subjective technique by using test method AATCC 5-2011 as detailed in the section 1.4. In this scale 5 means highly separated fibers while 1 indicates lowest degree of separation of fibers. It can be noticed from Table 3.5 that cotton, flax and olefin fibers have ranking of 5, 3.3 and 4.2 respectively while E2ACT, E6PET-6 and E4ST have ranking of 1.7, 3 and 3.7 respectively.

It can be noticed that bleached fibers (E4ST) are highly individual (ranking 3.7) among treated fibers, while acid treated fibers (E2ACT) are comparatively less individual with ranking of 1.7 and enzyme treated fibers (E6PET-6) are least separate among the treated fibers with ranking of 3.

3.6 Moisture Regain

Moisture regain is important for the fibers when processing for yarn manufacturing (spinning) as they have to pass through different mechanical parts at a very high speed. Due to friction two phenomena can occur, one, heat is generated at the point of contact; second, static charges develop in the material. Both of these issues can cause a hazard and to avoid these problems the processing environment is always controlled for temperature and humidity. Textile materials absorb moisture from the environment which makes it possible for them to avoid overheating and development of static charge which indicates that the fibers should have sufficient ability of moisture regain to absorb moisture.

Table 3.6: Moisture Regain (%) of chemical and enzyme treated Hemp fibers

Sample ID	**Moisture regain** (%)	**Sample ID**	**Moisture regain** (%)
Raw Hemp	7.87	E6PET-1	0.17
EIW1T/I (Scoured)	5.96	E6PET-2	0.14
E2ALT/III (alkali → acid)	5.40	E6PET-3	0.13
E2ACT/IV (acid → alkali)	5.42	E6PET-4	0.14
E3MT/V (methanol)	6.15	E6PET-5	0.16
E4ST/VII (Scoured and Bleached)	3.97	E6PET-6	0.20
E5ET (Enzeco Pectinase Dv-2)	0.13	E6PET-7	0.20

Raw hemp has the highest ability of moisture regain (Table 3.6) which is close to the moisture regain of cotton (7.5%) [62]. All of the subsequent treatments reduced the intensity of this phenomenon. Scoured (E1W1T), alkali (E2ALT) and acid (E2ACT) treated fibers have almost the same moisture regain which is 5.96%, 5.40% and 5.42% respectively. Methanol treated (E3MT) and bleached fibers (E4ST) respectively have 6.15% and 3.97% of moisture regain. E3MT have the highest while E4ST have the lowest moisture regain among the chemical treated fibers. The reasons for reduction of moisture regain in the treated fibers may be due to the decrease in roughness of the fiber surface as well as a reduction in amorphous regions, resulting in less available spaces for water molecules.

Enzyme treatment reduced the moisture regain enormously (Table 3.6). Fibers treated with Enzeco Pectinase DV-2 (E5ET) in an acidic media for 480 min have 0.13% moisture regain. All of the other enzyme treatments were performed by using Pectinase

Aspergillus Aculeatus enzyme. E6PET-1, E6PET-2 and E6PET-3 have a moisture regain of 0.17%, 0.14% and 0.13% respectively while for E6PET-4, E6PET-5, E6PET-6 and E6PET-7 moisture regain is 0.14%, 0.16%, 0.20% and 0.20% respectively.

Higher values of moisture regain belong to E6PET-1 (Temperature: 50°C, time: 480 min, Conc.: 1%, steel balls: 50) E6PET-6 (Temperature: 50°C, time: 240 min, Conc.: 2%) and E6PET-7 (Temperature: 60°C, time: 240 min, Conc.: 1%). All of these treatments were conducted in an acidic environment. However their results cannot be compared as the treatment conditions other than media are different.

E6PET-1, E6PET-2 and E6PET-3 were treated in acidic media, at 50°C for 60 min, 120 min and 240 min respectively. It can be noticed (Figure 3.7) that their moisture regain is decreased with an increase in treatment time although there is also difference in weight loss (%) of these fibers.

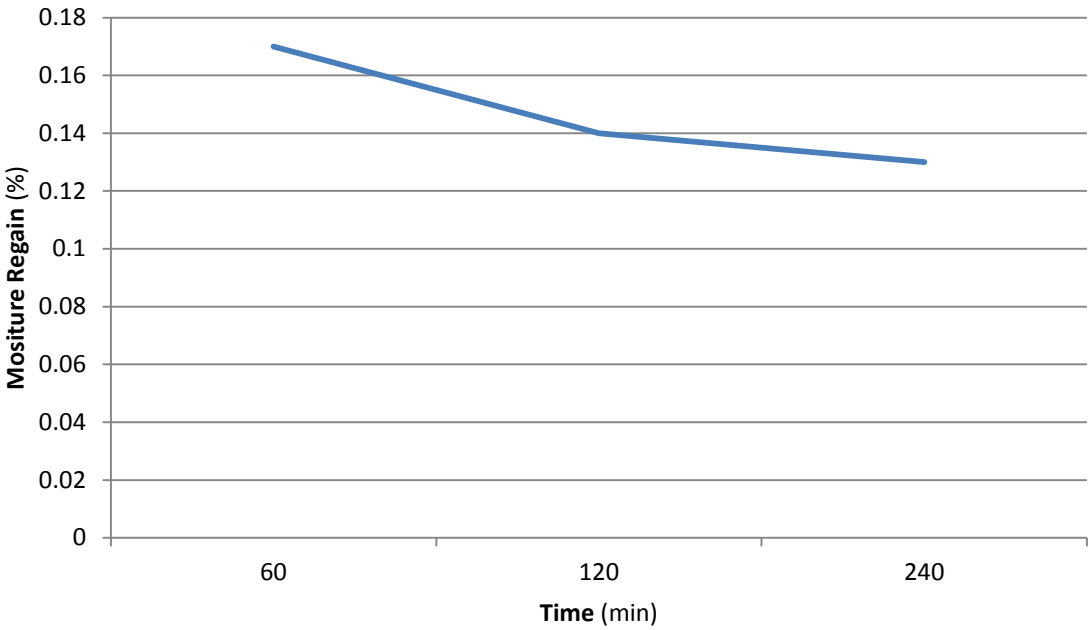


Figure 3.7: Effect of treatment time on moisture regain of enzyme treated fibers.

E6PET-3 (temperature: 50°C, Conc.: 1%) has the least moisture regain which has been treated in the same conditions as that of E6PET-6 (temperature: 50°C, Conc.: 2%) and E6PET-7 (temperature: 60°C, Conc.: 1%) but with a difference in treatment concentration with one (E6PET-6) and temperature with the other (E6PET-7). It can be observed that an increase in temperature and enzyme concentration resulted in higher moisture regain values.

E6PET-3, E6PET-4 and E6PET-5 were treated for 240 min at 50°C with enzyme concentration of 1% but the treatment media for these treatments are acidic, neutral and alkaline respectively. It can be observed (Table 3.6) that the moisture regains for E6PET-3 is 0.13%, 0.14% for E6PET-4, and 0.16% for E6PET-5. It can be concluded that the alkaline medium (E6PET-5) resulted in higher moisture regain while the neutral (E6PET-

4) and acidic (E6PET-3) media reduced the moisture regain of the treated fibers (Figure 3.8).

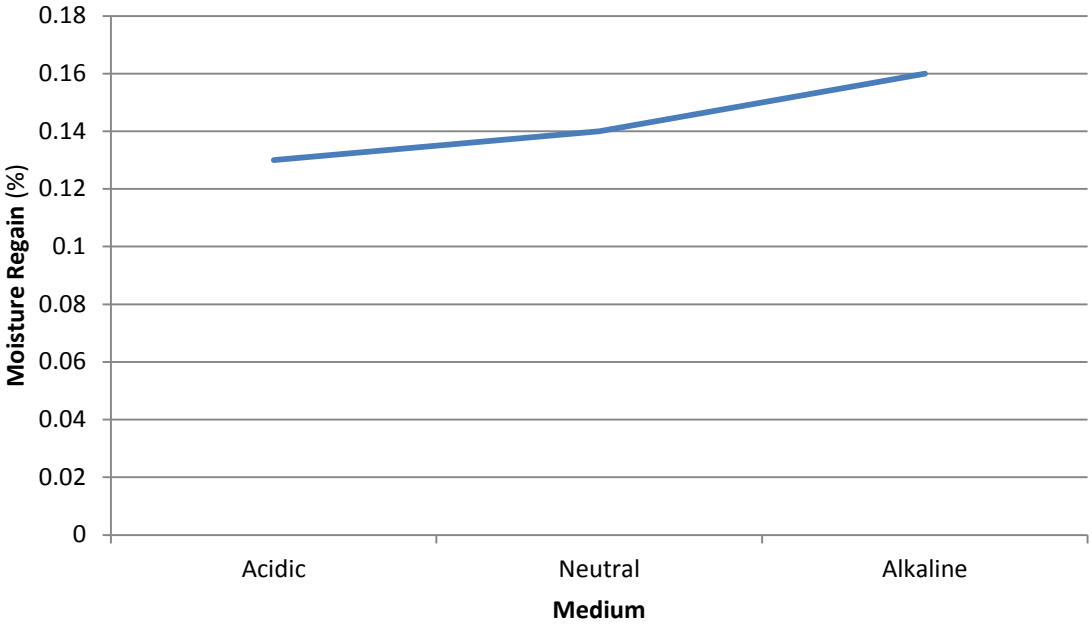


Figure 3.8: Effect of treatment media on moisture regain of enzyme treated fibers.

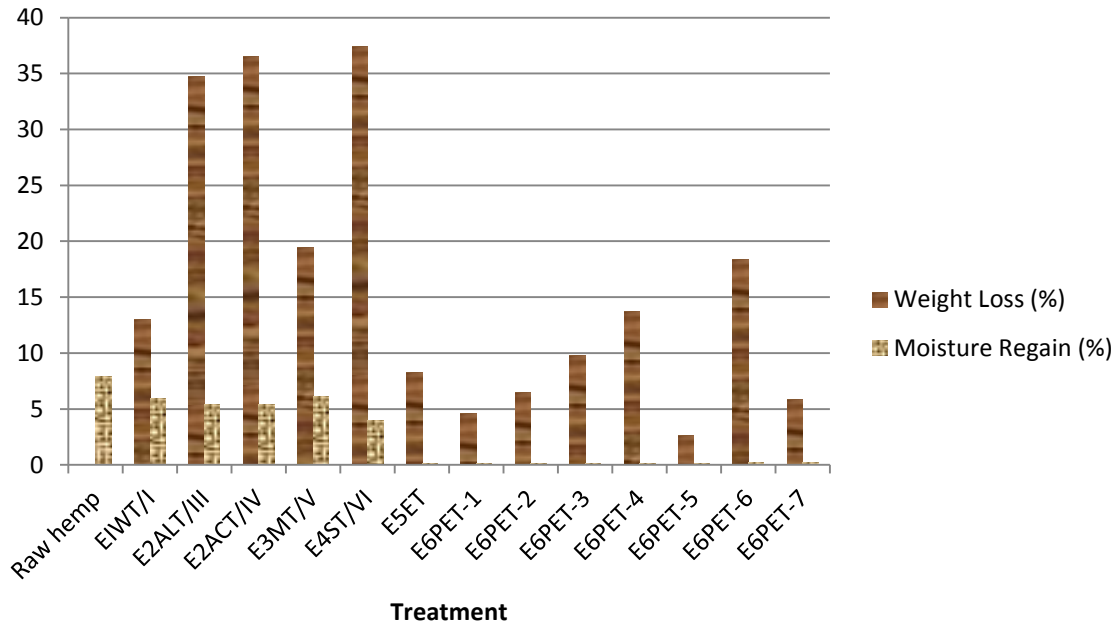


Figure 3.9: Comparison of weight loss (%) and moisture regain (%) by chemical and enzyme treatment

The weight loss (%) and moisture regain (%) of fibers treated by enzymes and other chemicals is presented in graphic form in Figure 3.9. It can be noticed that the chemical treatment affected more than enzyme treatment both in terms of weight loss and moisture regain, resulting in higher weight loss and better moisture regain properties.

3.7 Results of Bleaching Optimization Process

In preliminary studies (phase-I), it was found that chemical treated fibers resulted in higher weight loss, lower tenacity loss and higher moisture regain than the enzyme treatments (Table 3.7-A). Therefore, it was decided to focus on chemical treatment methods rather than enzyme. Further, since among the chemical treatment methods, bleaching resulted in higher weight loss and better fiber softness, it was decided to

optimize the bleaching parameters to achieve maximum removal of non-cellulosic materials and the highest softness.

Table 3.7-A: Comparison of chemical and enzyme treated fiber's properties.

Property	**Treatments and sample codes**			
	Sample ID	**Chemical**	**Sample ID**	**Enzyme**
Weight Loss (%)	E4ST	37.42	E6PET-6	18.40
Breaking Load Loss (%)	E1W1T	73.05	E6PET-4	73.14
Tenacity Loss (%)	E4ST	87.09	E6PET-4	90.37
Softness (Ranking Scale 1-5)	E4ST	3.00	E6PET-6	1.30
Moisture Regain (%)	E3MT	6.15	E6PET-7	0.20

Note: The highest values of different properties resulted by chemical and enzyme treatment are presented in this table.

After conditioning in standard atmospheric conditions, decorticated fibers were scoured and bleached as discussed in the section 2.3. Fibers were rinsed with tap water after detergent wash, scoured with alkali, acid and these pre-treated fibers were then bleached at different time and temperatures. It is worth mentioning here that after bleaching, when the canisters were opened to remove the bleached samples, a loud bang was heard from some canisters, which might be due to the pressure built-up in those canisters during treatment. Fibers were conditioned in standard atmospheric conditions after bleaching and weighed in order to calculate the weight loss percentage.

3.7.1 Weight Loss (%)

After conditioning fibers for 24 hours in standard atmospheric conditions, weight loss (%) was calculated. Weight loss of four bleached hemp fibers at 60°C for 120 min of treatment time varies between 17.93-22.17% and average weight loss is 19.83%. Bleaching of scoured fibers at 60°C for 180 min resulted in the fiber weight loss in the range of 15.63-20.43%, while the average weight loss at these conditions was 18.83%. Treatment at 60°C for the longest period of time i.e. 240 min led the weight loss between the ranges of 20.47-23.73% whereas the average weight loss at these conditions was 21.76%. The second level of treatment temperature was 80°C. Weight loss at this temperature for 120 min is between 20.30-22.43% while the average weight loss at this time and temperature was 21.22%. Keeping the same temperature (80°C) for 180 min of treatment, resulted in fibers with weight loss in the range of 19.77-24.03% whereas the average weight loss was 22.14%. In the same way, the treatment for 240 min at the 80°C temperature resulted in fibers with weight loss in the range of 20.00-28.93% and the average weight loss at this set of time and temperature is 23.09%. Among the three levels of temperature, 95°C was the highest and twelve (12) samples were treated at this temperature. Four fiber samples treated at this temperature for 120 min lost weight in the range of 21.10-23.20% and the average weight loss was 21.83%. In the same way treatment of four fiber samples at 95°C for 180 min resulted in weight loss in the range of 20.23-25.57% whereas the average weight loss was 22.81%. The last set of four treatments at the highest level of treatment time and temperature (at 95°C and for 240 min) resulted in fibers with weight loss in the range of 22.47-31.60% with the average weight loss of 26.97%.

Analysis of variance (ANOVA) was performed on the weight loss data to examine the significance of the effect of experimental factors (temperature and time) and their interactions. Means were compared using Duncan's multiple range tests at a significance level of 0.05. The results of ANOVA are presented in Table 3.7.

Table 3.7: The ANOVA Procedure

Source	**DF**	**Sum of Squares**	**Mean Square**	**F Value**	**Pr > F** (p value)
Temp	2	83.498	41.749	6.5	0.005
Time	2	65.001	32.5	5.06	0.0136
Temp*Time	4	19.682	4.92	0.77	0.556

It can be observed from the Table 3.7 that the p-value for temperature is 0.005 which is less than 0.05 indicating that temperature was a statistically significant variable to effect weight loss (%) of hemp fiber while bleaching. Similarly the p-value for treatment time is 0.0136 which is also less than 0.05 specifying the statistically significant effect of time on weight loss during bleaching of hemp fibers. Interaction of time and temperature (temp*time) had a p-value of 0.556 which is higher than 0.05 which shows that the interaction of treatment time and temperature did not have a statistically significant effect on the weight loss of bleached hemp fibers.

Duncan's multiple range tests was applied in order to assess the significance of different temperature levels on hemp fiber weight loss after bleaching and the results are shown in Figure 3.10. It can be observed from the table that samples treated at 95˚C and 80˚C fall in the same group which shows that their results are not statistically different. Whereas those treated at 80˚C and 60˚C also fall in the same group indicating that bleaching of

fibers at either of these temperatures will provide statistically similar results. Bleaching at 95˚C and 60˚C temperatures have a significant impact on weight loss (%), as both of these temperatures have different grouping. In case of 80˚C the impact is not significantly different from those of 95˚C and 60˚C. It can be concluded that bleaching fiber both at 80˚C and 60˚C, keeping the time constant, will yield the same results. So it will be better to treat the fibers at 60˚C rather than 80˚C to yield the same weight loss due to utilization of less energy.

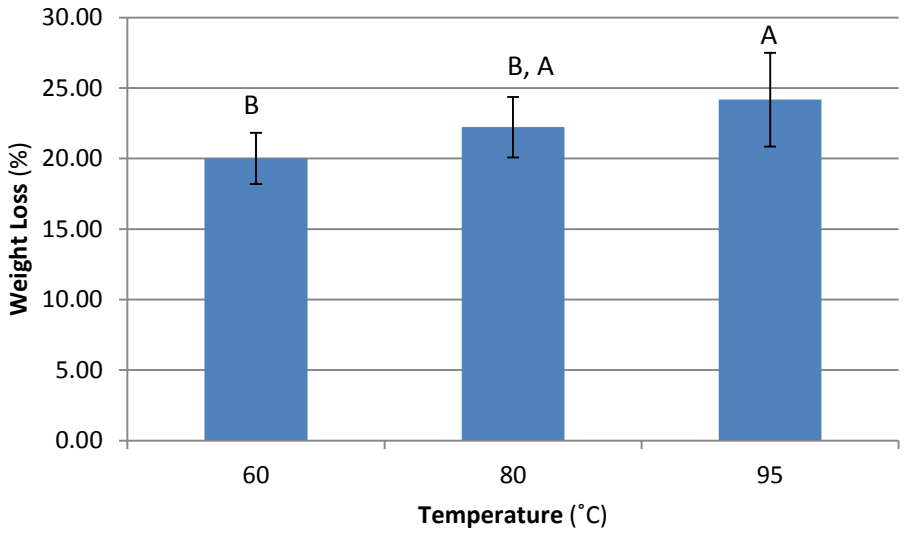


Figure 3.10: Effect of treatment temperature (˚C) on weight loss (%) of bleached hemp fibers

In the same way, Duncan's multiple range tests was applied in order to assess the significance of different treatment times on hemp fiber weight loss after bleaching. The Duncan's analysis results are shown in Figure 3.10. It can be observed from the figure that a treatment time of 240 min lies in one group A, whereas both 180 min and 120 min lie in the same group i.e. B. It indicates that a bleaching treatment at 240 min will result

in fibers with higher weight loss (%) which is statistically different from the other two treatment times. Bleaching treatment times of 180 min and 120 min (being in the same group) will yield the fibers with the weight loss (%) which is statistically not different from each other. It can be stated that statistically same weight loss can be achieved by using a lower treatment time which is 120 min than using the higher treatment time which is 180 min.

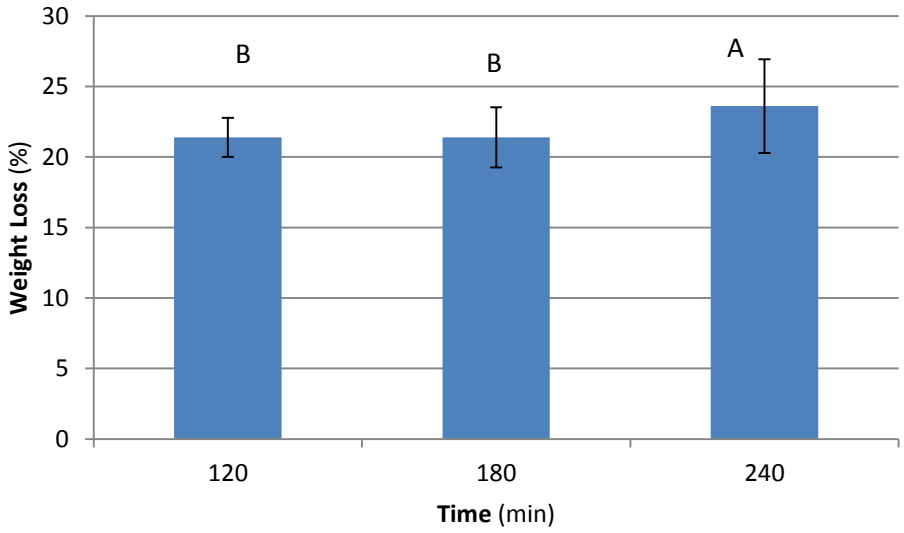


Figure 3.11: Effect of treatment time (min) on weight loss (%) of bleached hemp fibers

The effect of interaction of treatment time (min) and temp (˚C) on weight loss (%) of bleached hemp fibers is presented through a surface plot in Figure 3.12. It can be observed from the graph that the combination of the highest temperature (95°C) and the longest treatment time (240 min) resulted in the highest weight loss (%). This is in agreement with the Duncan's analysis.

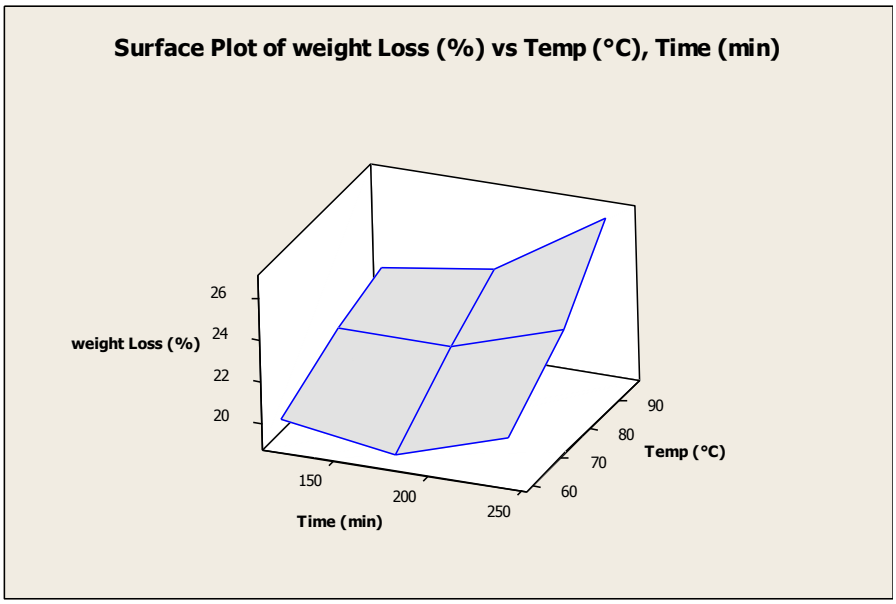


Figure 3.12: Effect of treatment time (min) and temp (˚C) on weight loss (%) of bleached hemp fibers

The weight loss at 80˚C for 120 min and 180 min were similar, which is 21.67%, while treatment for 240 min at the same temperature yielded the bleached fibers with the higher percentage of weight loss which was 23.33%. Bleaching of scoured fibers at 95˚C for 120 min and 180 min resulted in weight loss of 22.50% and 23.33% respectively whereas treatment at 95˚C for 240 min yielded the fibers with the highest percentage of impurities removed which was 26.67%. It is therefore iterated that within the current experimental conditions, in order to achieve larger removal of impurities, higher temperatures and shorter treatment time were more effective than lower temperatures and longer treatment times.

3.7.2 Effect of Using Recycled Chemicals on Weight Loss (%)

In order to check the impact of bleaching process of hemp fibers on the environment, some of the samples were treated with a used chemical solution. For this purpose, scoured fibers were bleached using the recipe shown in Table 3.8 at 95˚C for 120 min. For this part of the experiment, the used bleaching solution was used for the next two sets of experiments at the same conditions of temperature and time as given in Table 4.4.

Table 3.8: Effect of using used chemicals on weight loss (%) of bleached hemp fibers

Run Order	Weight (gm)			
	Before	After	Loss %	Average Loss
With Fresh Chemicals				
1	3.00	2.367	21.10	21.83
2	3.00	2.347	21.77	
3	3.00	2.363	21.23	
4	3.00	2.304	23.20	
With Used Chemicals				
1	3.00	2.533	15.57	15.57
2	3.00	2.533	15.57	

The average percentage of impurities removed by using fresh bleaching chemical solution is 21.83% while the average percentage of impurities removed with the used bleaching chemical solution is 15.57%. The used bleaching solution removed 25.91% less impurities than the fresh bleaching solution. Although the impurities removal efficiency of used bleaching chemicals are less than the fresh chemicals (≈75%), but it seems that bleaching chemicals can be reused as in some cases coarser yarn is required for some application. However, further experiments should be conducted to develop a model for expected level of impurities removal with used chemicals.

3.7.3 Impact of Bleaching on Fiber Softness

One of the objectives of bleaching was to analyze the impact of treatment on hemp fiber softness as it is one of the ring spinning properties. It was found from the preliminary results that well separated (fluffy) fibers give better softness. Three treatments (with four replicates) were further used to analyze the effect of fiber individualization of bleached fibers on softness. For this purpose, a manual carding machine was used to separate the fibers.

Samples with three treatment temperature (60°C, 80°C and 95°C) and 240 min treatment time were selected for softness measurement as statistical analysis showed that these samples contain the least amount of non-cellulosic materials at that specific temperature. Before softness evaluation, these twelve selected samples were passed through the carding machine in order to improve the separation and softness of the fibers. In the textile spinning process, carding is used for fiber to fiber opening, trash removal, fiber parallelizing along an axis and formation of sliver. These carded samples were assigned codes and presented to the observers along with the standard fiber with softness ranking as given in Table 2.6 with a paper having the same codes, as on the samples. Softness results of bleached and carded hemp fibers by four observers are given in Table 3.9.

Table 3.9: Softness ranking of bleached and carded hemp fibers

Observer	**Group 1** (60°C)				**Group 2** (80°C)				**Group 3** (95°C)			
	G1S 1	**G1S 2**	**G1S 3**	**G1S 4**	**G2S 1**	**G2S 2**	**G2S 3**	**G2S 4**	**G3S 1**	**G3S 2**	**G3S 3**	**G3S 4**
A	2	2	2.5	1	2	2	2	2	2	2	2	1
B	1	3	2	4	3	2	4	1	3	2	5	4
C	2	2	2	2	2	2	2	2	2	2	3	2
D	2	1	1	2	2	2.5	2.5	1	3	3	3	3
Average	1.8	2	1.9	2.25	2.3	2.1	2.6	1.5	2.5	2.3	3.3	2.5
	2.0				2.1				2.7			

Group 1 (G1) represents the fibers treated at 60°C for 240 min while group 2 (G2) contains the fibers with treatment at 80°C for 240 min whereas group 3 (G3) represents the fibers treated at 95°C and 240 min. It can be observed from Table 3.9 that hemp fibers of group 1 have a softness rank of 2.0; group 2 has 2.1 and group 3 have a softness ranking of 2.7. Group 3 have the highest level of softness whereas group 1 had the lowest level of softness among bleached and carded hemp fibers. It seems that the bleaching treatment performed at 95°C and 240 min produced relatively softer fibers than the fibers bleached at other two temperatures.

If we compare the highest softness of hemp (2.7, Table 3.9) to the softness of flax (softness: 3, Table 2.6) and olefin (softness 2, Table 2.6), it can be observed that flax softness is comparatively higher than the olefin and bleached hemp fibers. However, bleached hemp is softer than olefin which is used commonly with cotton to produce cotton/olefin blended spun yarn in different proportion by using open end spinning

system [64]. So it can be stated that bleached and carded hemp fiber softness is sufficient for processing through a cotton spinning system.

3.8 Conclusions

As already mentioned, current research is divided in two phases. In phase-I, chemical treatments E2ALT (weight loss: 34.72%) and E2ACT (weight loss: 36.49%) removed most of the impurities. On the other hand E2ALT and E2ACT lost 55.73% and 53.96% of breaking load strength and 9.14% and 29.00% of tenacity. Both breaking load (%) and tenacity loss (%) for these treated fibers are the least among the chemically treated fibers. Weight loss with Enzyme (Aspergillus Aculeatus) treatment showed a linear relation with time and concentration. In this research, temperature of 50°C was found optimum for treatment in terms of weight loss. It is also apparent from the results (Table 3.2) that the neutral media (E6PET-4) was suitable to achieve a higher weight loss (or impurities removal). The results of current research would indicate that 50°C temperature and neutral media may be the best conditions in order to create a suitable environment for the removal of impurities. However, enzyme did not performed significantly for removal of non-cellulosic materials.

Moisture regain results show that among chemically treated fibers E2ALT (5.40%) and E2ACT (5.42%) have higher values of moisture regain (other than moisture regain of E3MT which is 5.42%) which is close to cotton fibers (7.5%). It can be concluded that for both of these treated fibers there will be no problem of moisture absorption while processing through a cotton spinning system. In the case of enzyme treated fibers it seems that better results of moisture regain can be achieved by treating the fibers with

Pectinase Aspergillus Aculeatus in alkaline media for 60 min at optimum temperature and concentration. All treatments, either chemicals or enzyme, reduced the moisture regain. It may be due to these substances (chemicals/enzymes), destroying the amorphous regions, which are responsible for fiber absorbency. This hypothesis is also supported by the observation fact that the strength of the treated fibers was reduced indicating damage of amorphous regions.

Fiber stiffness was another important problem of decorticated hemp fibers that needed to be addressed. Although fiber stiffness was improved by these initial treatments but the stiffness of resultant fibers was higher than required. Future research can be focused on achieving the required level of softness. Steel balls were also added in two treatments (E5ET, E6PET) in order to soften the fibers; however their addition resulted in fiber damage.

Fiber individualization was another important factor to be focused through these series of experiments. However, it was not improved by any of the chemical or enzyme treatments. It is hypothesized that individualization may be enhanced by combining both chemical and mechanical (carding or combing) methods.

In the second phase of optimizing bleaching process (phase-II), decorticated hemp fibers were scoured and bleached at three different levels of time (min) and temperature (°C). The objective was to find out the optimum set of time and temperature for removal of non-cellulosic content from the hemp fiber. The bleached fibers were also analyzed for evaluation of softness using a subjective ranking method.

It can be observed from the results of the weight loss (%) (Which are an indicator of impurities removed) that the treatment temperature has comparatively more effect on the impurities removal than the treatment time. The weight loss results showed that lower temperature and longer treatment time can be used to remove the optimum level of impurities and vice versa.

Fiber softness can be improved by carding the fibers after scouring and bleaching. Results showed that the bleached and carded hemp fibers are better in softness than olefin fibers. These olefin fibers are already being spun with cotton by using an open end cotton spinning system [63]. It can be concluded that hemp fibers can be blended with cotton fibers considering the softness rating.

Chapter 4

Conclusions and Future Work

Use of hemp fiber for woven or knitted apparel and smart textile applications (bandages) has been desired by the growers, traders and researchers of hemp, but it has not yet been achieved. After reviewing the literature for this thesis, it has been observed that numerous attempts have been made to modify the fiber properties to make high quality yarn for apparel and smart textile applications. Different chemicals, enzymes and mechanical treatments have been employed for this surface modification. However, irregularities and impurities in the fiber surface and structure have not been removed completely.

The current research was an attempt to move one step forward for making hemp viable to be used on a mass scale for yarn manufacturing using the cotton ring spinning system. The current research can be divided in to two phases. In phase-I, the critical hemp fiber parameters for cotton spinning system were identified by surface modification using different chemicals and enzymes. In the phase-II, parameters of the bleaching process were optimized in order to remove maximum impurities in terms of weight loss (%) and to make fibers soft.

The current research has identified six parameters critical for spinning of hemp fibers. These parameters are strength, length and length variation (≤1/8 inch), single fiber entity, fineness, breaking twist angle (BTA) or bending modulus and softness. Measurement of weight loss (%) is required to investigate the efficiency of chemical and enzyme treatments in removal of non-cellulosic materials. In phase-I of experiments, different

trials were conducted, using decorticated hemp fibers, to evaluate the impact of these treatments on the six spinning parameters. In phase-II of this research, optimization of impurities removal and improvement in softness was investigated using physiochemical treatment methods.

For preliminary investigation, decorticated fibers were used as raw material and seven sets of trials were performed using different chemicals. Among these treatments, the scouring and bleaching treatment (Sample ID: E4ST) produced fibers with the highest amount of weight loss (%) of up to 37%. It indicates that maximum non-cellulosic materials were removed as hemp contains between 25-37% non-cellulosic materials in its structure. Also, E4ST samples were found to be comparatively softer and more individual than other treated samples. The preliminary investigation also revealed that the chemical treated fibers stick to each other. None of the treatments resulted in the fibers with single entity status (single fiber). Attempts were made for fiber separation using different machines such as card, planetary mono mill and sieve shaker. Although carding improved the fiber separation but none of the mechanical process resulted in completely individual fibers. It was found that the variation in fiber length was less than ±1/8 inch, which is one of the six spinning parameters. The tenacity of the chemically treated fibers was found to be in the range of 0.06-0.18 N/tex, which is sufficient for processing of fibers on the cotton ring spinning system. Compared to virgin decorticated hemp fibers, these chemically treated fibers were relatively fine but are still coarser for cotton spinning systems. Further, the variation in fiber diameter was very high which is due to the existence of multiple fibers in apparently single fiber form.

Two types of enzymes were used for evaluation of the impact of enzyme treatments on six spinning properties. It has been observed that enzymes removed a maximum of 18.4% of impurities from the fiber structure; however, the tenacity loss was 67-90% with the ultimate tenacity range of 0.02-0.07 N/tex. Due to the lower weight loss (%) but high tenacity loss, neither of the enzymes were selected for treatment in phase-II, although the length variation was less than ±1/8 inch.

Another important factor analyzed in phase-I was the effect of chemical and enzyme treatment on moisture regain of the hemp fiber. It was noticed that all treatments lowered the moisture regain of the fibers. The moisture regain of the decorticated raw hemp fibers was 7.87 (%) whereas moisture regains value of chemically treated fibers was 6.15-3.97 (%). Enzyme treatment severely affected the moisture regain of the hemp fibers as the value was only between 0.20-0.13 (%) after treatment. The reduction in moisture regain may be due to the decrease in amorphous region of the fiber or blockage of porous structure. This change in moisture regain (%) can be beneficial for some non-apparel application (automobile), however, for apparel application this may create problem during wet processing treatments (dyeing, printing and finishing). Further research is needed to optimize the moisture regain for a specific application.

For Phase-II, as it was observed in Phase-I that among six spinning properties, strength, length and length variation were acceptable. Focus was not given on fineness because the treatments did not produce single fiber entity. Bending modulus was not measured as it is related to fiber softness. Therefore, in Phase-II, major focus was given on the maximum

removal of non-cellulosic materials and fiber softness using scouring and bleaching process followed by carding.

Non-cellulosic materials in the fiber structure are responsible for lack of spinning properties of hemp fiber. In the current research, bleaching process was included in the series of chemical treatments as this process is essential for preparatory process before textile dyeing and printing and was a contributing factor for removal of non-cellulosic materials. For the optimization of weight loss (%) and softness property, decorticated hemp fibers were scoured and bleached at three different levels of time and temperature. The results showed that the weight loss (%) was increased during bleaching with treatment time and temperature. Maximum percentage of weight loss (26.97%) was observed while bleaching at 95˚C for 240 min. This was confirmed by the Two-Way ANOVA. If temperature is increased beyond 95˚C, it may damage the fiber's crystalline region, resulting in loss of strength of fibers. However depending on end use, fibers may also be bleached at either 60˚C or 80˚C temperature for longer period of time in order to obtain optimum bleaching results.

Statistical (Duncan's Multiple Range) analysis was applied to the results of weight loss (%) in order to evaluate the statistical significance of treatment temperature and time. It has been observed that bleaching processes at either of 95°C and 80°C do not have any statistical significant difference on weight loss (%). Similarly, bleaching treatments on weight loss (%) at either 80°C or 60°C were also not found statistically significant. It is, therefore, recommended to carry out bleaching either at 95°C or 60°C depending on end use of fibers.

The Duncan's Multiple Analysis also showed that weight loss (%) for bleaching time of 240 min has a statistically significant difference than those of bleached at 120 or 180 min. It can be concluded that bleaching should be done either for 240 or 120 min.

After bleaching, twelve selected samples were carded for softness evaluation. These twelve samples contained four samples from each temperature with the highest weight loss (%). Softness of the bleached hemp fibers was found to be similar to olefin fibers with a rating of 2.7 out of 5.0 which is given for cotton. With this softness rating, the hemp fiber can be spun in the ring spinning system if other spinning parameters are acceptable.

Finally, the current research optimized the bleaching treatment which is 95°C and 240 min to remove the largest amount of non-cellulosic materials and to produce fibers with the highest softness. Among other spinning properties, the current research also found that strength and length variation are suitable for cotton spinning properties while future work should carry out to investigate the formation of single fiber entity and lower the bending modulus of hemp fiber.

In order to analyze the impact of previously used bleaching solution on weight loss (%) of scoured fibers, scoured fibers were bleached at 95°C for 120 min and the results were compared with those of bleached with fresh chemicals. The fiber weight loss (%) with used solution was almost 25% less than the fiber weight loss (%) treated with fresh solution. It can be concluded that if other variable allow, used bleaching solution can make this process more economical and environment friendly.

Chapter 5

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