INFLUENCE OF COTTON FIBER PROPERTIES ON THE MICROSTRUCTURAL CHARACTERISTICS OF MERCERIZED FIBERS BY REGRESSION ANALYSIS

Hua Wang

Professor
College of Textiles
Donghua University
2999 North Renmin Road
Shanghai 2011620, China
E-mail: huawang@dhu.edu.cn

Amjad Farooq

Researcher
College of Textiles
and
Key Laboratory of Innovation Center for Textile Science and Technology
Ministry of Education
Donghua University
2999 North Renmin Road
Shanghai 201620, China
E-mails: amjadfarooqbzu@hotmail.com or 417014@mail.dhu.edu.cn

Hafeezullah Memon†

Researcher
Center for Civil Aviation Composites
and
Key Laboratory of Textile Science & Technology, Ministry of Education
College of Textiles
Donghua University
2999 North Renmin Road
Shanghai, 201620, China
E-mails: hafeezullah_m@yahoo.com or hm@mail.dhu.edu.cn

(Received February 2019)

Abstract. This study is conducted on a raw material imported from several countries for the purpose of promoting the development of the world's cotton spinning industry. If the time for selecting the raw material as well as the quality of the cotton spinning is improved, then time and money will be saved and the spinning industry will be improved. For this study, different cotton fibers were selected from different ecological regions. The cotton fibers were processed using the mercerization process, and then they were examined using X-ray diffraction (XRD), Fourier transform IR (FTIR), and scanning electronic microscope (SEM). High-volume instrument study was conducted to evaluate the physical properties of cotton fibers, including short fiber content, tensile strength, elongation at break, Micronaire value, and upper half mean length. In addition, a change in fiber strength, the degree of crystallinity, and degree of orientation of cotton fibers before and after mercerization was also examined. SEM results show that the surface of cotton fibers became smooth and shiny after the treatment. FTIR and XRD revealed that the chemical composition did not change, but the degree of crystallinity decreased and the degree of orientation of alkalized cotton fibers increased after mercerization. In the second phase of this study, a correlation analysis was made between the physical properties of cotton fibers and the microstructural properties of alkalized cotton samples. This analysis revealed that the breaking strength of cotton fibers is strongly negatively correlated with the crystallinity of cotton fibers. The higher the tensile

* Corresponding author
† SWST member

Wood and Fiber Science, 52(1), 2020, pp. 13-27
https://doi.org/10.22382/wfs-2020-003

strength, the lower the crystallinity of cotton fibers, which leads to better mechanical properties of the end product. The degree of the orientation of cotton fibers is highly correlated with a tensile strength of cotton fibers. Xinjiang-Cn, BG-Au, and BG-Tu samples exhibited relatively better physical properties. BG-Au, BG-Tu, and Pakistan samples have higher values of tensile strength and lower values of yellowness; moreover, the raw material from these samples can be preferred according to correlation analysis. Correlation analysis between physical properties reveals that the tensile strength of cotton fibers is positively correlated with the Micronaire value; however, the correlation is not strong because of the lower value of the correlation coefficient as 0.4809.

Keywords: Degree of crystallinity, mercerization, physical properties, microstructural properties.

INTRODUCTION

In spite of the tremendous development of synthetic fiber production in recent years, cotton still holds a 71% fiber production market in the world. It is most versatile for fabric materials and, therefore, extensively used (Li et al 2003). Cotton fiber is the purest form of cellulose and a most noteworthy natural fiber. The financial importance of cotton in the worldwide market is clear by its majority share (more than 50%) among strands for clothing and textile materials. Both the market esteem and excellence of cotton products are straightforwardly identified with the quality of cotton fiber (Gordon and Hsieh 2006).

Mercerization is a major substance treatment process (Yue et al 2013) that fundamentally impacts the behavior of cotton quality attributes eg change in measurement, fine structure, morphology, and mechanical properties. There are different objects of mercerization eg adding luster to cotton fibers/fabric, enhancing dying qualities, and increasing the strength of fibers. Despite the fact that mercerization was initiated in 1850, it is used as a part of the industry because it impacts the mechanical properties of cotton materials. In the cotton market, China is among the biggest cotton producers in the world. However, with the booming of the cotton industry, the local cotton cannot meet the needs of the fast-developing economies (Wakelyn et al 2006). Cotton cultivated in various regions still shows a great contrast because of different biological systems throughout the world (Wang et al 2014). In the early years, examination on cotton strands stayed in a perceptible range with tufts as principle queries. However, with the rapid improvement of instruments eg scanning electron microscope (SEM), X-ray diffraction (XRD), and Fourier transform IR (FTIR) spectroscopy, the examination of single fibers can be thoroughly evaluated and is a common measurement today. It has been accounted that the surface of the single cotton fiber is made out of consistent reversal points, with the length of the reversal zones around a few micrometers, and the recurrence of reversal along the fiber ranges from only 2 per cm to 27 per cm, depending on various varieties (Patel et al 1990; Long et al 2010).

The fiber nature of the crop additionally relies on agronomic practices. Fiber quality is a key element that can decide the fiber cost and nature of cotton production. The cotton fiber is a solitary organic cell. The layers in the cell structure from the outside of the fiber to inside are cuticle, primary wall, secondary wall, and lumen. These layers are completely different structurally and chemically. The phenotype qualities of cotton fibers impact the mechanical and useful properties of the end product. A few researchers suggest that reversals are the strongest point of the cotton fibers (Wakelyn et al 2006). It is accounted that the cotton fibers have polymorphic crystalline cellulose structure and its crystalline morphology is separated into four distinct sorts as cellulose I, II, III, and IV (Ugbolue 1990). Common cotton is described as cellulose I; however, after the industrial mercerization process, cotton's crystallinity is transformed and described as cellulose II (Yue et al 2013). The treatment of cotton with liquid ammonia or essential alkyl amines changes cotton into the category of cellulose III. The presence of the amorphous domain in cotton filaments has been demonstrated by diffuse scattering among XRD (O'sullivan 1997). The tensile strength of cotton fibers is impacted extraordinarily by the degree between the chain molecules' orientation and the fiber axis. Generally, the more the oriented molecules contributed to, the higher the quality of the fiber strength (Wakelyn).

The experimental and statistical approach to determine the relationship between fiber and yarn quality characteristics has been the most popular approach recently. The determination of accurate and fast measurement of fiber properties is possible by high-volume instruments (HVIs). Almost all of the important fiber and yarn properties are measured by this method, and the results are empirically related by the statistical analysis approach. Hence, the interdependence of the different fiber properties can be examined to estimate the relative contribution of each cotton fiber property to the overall yarn properties. Various regression models have already been established using this method (El Mogahzy et al 1990; Ethridge and Zhu 1996; Majumdar and Majumdar 2004).

The quality of final textile products relies on the quality of the raw material fiber. The overall quality of cotton fiber depends on factors such as strength, elongation, fineness, length, short fiber content, etc. Because cotton is a natural fiber, it has a lot of unevenness in its properties. The decision for the nature of raw material selection is very unstructured and crude. Therefore, there is a need to solve such kind of problems through science techniques which might be useful in the spinning industry. Because common decision-making methods cannot handle imprecision and uncertainty of the properties of the raw material fiber, the purpose of this research study was to develop a method that involves a linear correlation regression analysis between the physical properties and microstructural properties. The fundamental goal of this study was to examine and contrast the properties of the different cotton fibers coming as raw material (fiber) into the China cotton market from various countries such as Africa, Australia, Pakistan, India, and some Chinese regions as well. In the present work, the micromorphologies, crystalline structures, and mechanical properties of these cotton assortments, prior and after mercerization were evaluated in detail, and then linear regression analysis was made between physical properties and microstructural properties. The data from this study will give a direction in material determination and forecast about yarn properties and also will provide guidance to the industry in material selection and the prediction of yarn characteristics.

MATERIALS AND METHODS

Materials

Three samples harvested from Xinjiang, China (labeled as Xinjiang-CN), Africa (KATI-AF), and Australia (BG-AU) were obtained and classified as the grade 3 (high-medium length) according to the Chinese cotton classing system (Zhou et al 2008). The samples are identified as mentioned in Table 1.

Physical Properties of Raw Cotton

HVI uses automated testing methods and measures fiber properties from a bundle of strands. This methodology is popular today for both marketing and breeding because it is beneficial for reducing time and cost. Generally, HVI estimations incorporate fiber length, length consistency, bundle tenacity, elongation, Micronaire, shading, and junk content (Negm et al 2015).

Upper Half Mean Length and Length Uniformity

The length uniformity indices (LUIs) provide an indication of short fiber content because cotton fibers of low LUI are likely to contain a high

Table 1. Samples identification.

Regions	Name of the sample
Africa	KATI-Af
	KATI-Ma
	KATI-Ug
	KATI-Sa
Australia	BG-Au
	BG-Tu
Pakistan	Pakistan
China	Xinjiang-Cn
India	India

Table 2. Indexes of upper half mean length and average length uniformity index of cotton samples.

Cotton	Upper half mean length (mm)	Rate of change (CV %)	LUI (%)	Rate of change (CV %)
India	28.33	2.1	81.5	1.1
KATI-Ma	28.57	2.6	82.0	2.5
KATI-Af	27.39	1.8	81.3	1.8
BG-Tu	30.27	1.6	81.4	0.7
Pakistan	28.13	0.9	82.1	0.8
BG-Au	30.37	0.6	82.5	0.7
KATI-Ug	27.72	2.1	80.2	1.3
KATI-Sa	28.39	2.3	81.3	1.6
Xinjiang-Cn	28.47	0.7	81.6	0.9

percentage of short fibers; it can be predicted that yarn quality would be low because of the inefficiency of processing with short cotton fibers.

Cotton fiber length is divided into the eight-length level from 25 to 32 mm, the length level is described in Table 1 and the LUI is divided into five categories as shown in Table 2. The length uniformity index is very important for yarn production efficiency as well as yarn strength and evenness. The average length of the upper half of cotton fiber is defined as the length of the cotton fiber. Table 2 compares the upper half of the average length of cotton fiber and LUI of several large cotton-producing areas.

The length of selected areas of cotton samples is between 27.72 and 30.37 mm, India; KATI-Ma, Pakistan; KATI-Sa and Xinjiang-Cn are all 28 mm in length, two of the samples KATI-Ug and KATI-Af are 27.72 mm and 27.39 mm long, respectively. Only two cotton varieties BG-Au and BG-Tu from Australia are 30.37 mm and 30.27 mm longer when compared with other cotton samples. Therefore, according to the previously described data analysis, the two Australian cotton fibers, which are longer will increase the yarn efficiency because of an increase in the fiber friction and cohesion between each other; thus, they will exhibit better properties resulting in increased yarn strength. In Table 2, LUI of several cotton fiber samples can be seen that are ranging from 80.2 to 82.5%, according to the LUI of the evaluation criteria (Table 1). All types of cotton samples are placed in a medium level of LUI, which shows these cotton samples are containing a lesser amount of short fiber. (Negm et al 2015).

Table 3. Micronaire values of cotton fiber and the rate of change.

Cotton sample	Micronaire value	Rate of change (CV %)
KATI-Sa	4.13	3.3
KATI-Af	4.10	3.5
KATI-Ma	4.34	3.2
Xinjiang-Cn	4.35	2.6
KATI-Ug	4.40	3.1
BG-Au	4.13	2.7
BG-Tu	4.32	2.5
India	4.07	4.1
Pakistan	3.78	2.4

Micronaire Value

The fineness and maturity of cotton fiber are calculated in terms of the Micronaire values of the sample, and it is determined by measuring the resistance to airflow of fibers of a specified mass compressed to a fixed volume. For international cotton classers and spinners, Micronaire characteristic is one of the most important factors in cotton raw material selection (Memon et al 2015). The classification for the standard of cotton fiber Micronaire value is presented in Table 3. Table 3 compares the Micronaire values of several cotton fibers.

As seen in Table 3, KATI-Sa, KATI-Af, KATI-Ma, Xinjiang-Cn, BG-Au, Indian, and BG-Tu cotton have average values of Micronaire; similarly, the Pakistani sample has a value of Micronaire as 3.78. These are considered acceptable values.

Table 4. Short fiber content and the rate of change of several cotton fibers.

Cotton samples	Short fiber content (%)	Rate of change (CV %)
India	4.6	17.4
KATI-Ma	6.9	10.5
BG-Au	8.6	10.9
Pakistan	5.2	12.2
KATI-Af	9.3	13.9
KATI-Sa	8.8	10.4
BG-Tu	9.3	8.5
KATI-Ug	10.1	10.2
Xinjiang-Cn	8.9	8.7

Short Fiber Content

There are a number of reasons that influence the resulting amount of short fiber content; among them, harvesting and ginning are the most important factors that influence short fiber contents (Garner et al 1970).

In the drawing process, the short fiber is often not easy to be controlled, resulting in poor yarn evenness and reduction in yarn strength (Lawrence 2003) (Table 4).

HVI test method used for this analysis shows that only the short fiber percentage of cotton fibers of India and Pakistan are 4.6 and 5.2, respectively. The short fiber percentage of the other fibers is significantly higher than that of the Indian and Pakistani cotton fibers, indicating that Indian and Pakistani cotton samples will give relatively better yarn strength and uniformity than other cotton fibers.

Tensile Strength and Elongation at Break

Tensile strength is corresponding to a maximum tensile load of fiber that can bear, expressed in units of cN/tex. Tensile properties that depend on the structure of cotton fibers have a great influence on textile end products. Cotton fibers are not homogeneous in their physical properties and dimensions. Their maturity, diameter, and fineness are different from fiber to fiber (Hosseinali 2012).

Elongation at break refers to the corresponding elongation of the fiber at the maximum breaking load. Fiber elongation at break or the breaking elongation herein referred to as elongation is an important cotton fiber property that directly affects yarn elongation and work-to-break values (Yang and Gordon 2016). Table 5 shows the general phenomena for the behavior of cotton fibers. Comparison of tensile strength and elongation at break is shown for a few major cotton-producing areas (Table 5).

According to the evaluation criteria of the tensile strength, we can see from Table 5 that the tensile strength of cotton fiber for samples Xinjiang and Africa is in the range of 26-28.9 cN/tex. Australian, Indian, and Pakistani cotton fiber breakage intensity is in the 29.0-30.9 cN/tex range, indicating a strong breaking strength S5. In other words, our selection of raw material from Australia, India, and Pakistan will have better strength properties than Xinjiang and African cotton ties. According to the evaluation criteria, the elongation at break of some cotton is relatively low. The breaking elongation of samples KATI-Ma, KATI-Ug, KATI-Sa, BG-Au, India, and Pakistan cotton fibers is very low, and the breaking elongation of Xinjiang-Cn cotton sample was noted as 7.3%. It predicts that except Xinjiang-Cn, the rest of the cotton fibers have a low resistance to deformation strength, so yarn and fabric can be easily deformed.

Impurities

Impurities include two indicators, namely, the number of impurities and impurity areas. The impurity area refers to the percentage of the area

Table 5. Tensile strength and elongation at break of various cotton samples.

Cotton	Tensile strength (cN/tex)	Coefficient of variation (CV %)	Elongation at break (%)	Coefficient of variation (CV %)
KATI-Ma	27.4	2.5	4.5	6.7
KATI-Af	28.4	4.0	4.9	4.9
KATI-Ug	27.6	3.5	3.7	4.7
KATI-Sa	26.3	2.5	4.0	5.2
BG-Tu	29.3	2.3	5.9	5.1
BG-Au	30.4	2.9	4.9	6.5
India	29.4	1.9	3.2	10
Xinjiang-Cn	27.3	3.2	7.3	5.0
Pakistan	29.7	4.3	4.7	5.3

Table 6. Number of impurities and impurity areas of cotton samples.

Samples	Impurities	Impurity area (%)	Grade
KATI-Sa	11	0.10	1
KATI-Af	24	0.24	2
Xinjiang-Cn	26	0.23	2
KATI-Ma	16	0.12	1
KATI-Ug	17	0.13	1
Pakistan	42	0.42	3
BG-Tu	21	0.25	2
India	31	0.41	3
BG-Au	24	0.21	2

covered by impurity particles in the test area. Trash is measured in terms of the amount of nonlint substances in the cotton eg leaves and barks from the cotton plants. Table 6 shows the comparison of several cotton fiber impurities, impurity area, and impurity level. From Table 6, we can see that African cotton fiber samples KATI-Sa, KATI-Ma, and KATI-Ug have less number of impurities and less impurity area than that of KATI-Af, Xinjiang-Cn, BG-Au, and BG-Tu, so they are classified as grade (Lawrence 2003) 1 and 2, respectively. India and Pakistani samples are classified as grade 3 because of the higher number of impurities and impurity areas.

Color Characteristics

The color of the cotton samples is determined from two parameters: the degree of reflectance (Rd) and yellowness (+b). Rd shows the brightness or dullness (degree of greyness) of the sample, whereas +b is the level of pigmentation in the fibers. A higher value of +b indicates a high degree of yellowness, whereas lower levels of +b indicate a low level of yellowness. There are five known groups of colors: gray, white, tinge, spotted, and yellow-stained (Lawrence 2003). From Table 7, we can see that the reflectivity and yellow degree of cotton fibers, the yellow degree is relatively small.

The reflectance is smaller in India, KATI-Af, and KATI-Ug, which is 76.9, 76.1, and 76.6 and the corresponding yellow degree is 8.3, 9.5, and 8.4, respectively. The higher reflectance of cotton fiber is noted in Australian BG-Au, BG-Tu, and Xinjiang-Cn, which is 81.2, 80.6, and 79.9 and the corresponding yellow degree was 8.6, 6.3, and 8.1, respectively. The reflectivity of the rest of the cotton fiber is in the range of 77-78. These data show that the color and maturity of Australian and Xinjiang cotton samples are better than several other producing areas, so the appearance and fastness of the spinning yarn and end product will be relatively good.

Table 7. Reflectivity, yellowness, and rate change of cotton fiber samples.

Samples	Reflectivity	Coefficient of variation (CV %)	Yellow degree	Coefficient of variation (CV %)
Xinjiang-Cn	79.9	0.7	8.1	1.5
KATI-Ma	78.4	1.3	9.3	1.7
KATI-Ug	76.6	1.4	8.4	2.1
India	75.8	0.8	8.3	2.2
Pakistan	77.1	1.3	8.5	2.1
BG-Au	81.2	0.7	8.6	1.9
BG-Tu	80.6	0.6	6.3	1.5
KATI-Sa	77.7	1.5	9.6	1.7
KATI-Af	76.1	1.4	9.5	2.1

Mercerization

After cleaning and scouring, cotton fibers were mercerized with concentrated alkali cotton to improve cotton fibers performance, enhance their spin ability, appearance, and its yarn and fabric performance. Cotton fibers were treated with the slack condition for each sample. NaOH solution (180 g/L ie 18%), 5% acetic acid, and a large amount of deionized water were used. Temperature of an electrically heated water bath was adjusted to 20°C and on reaching the temperature, mercerization was started. The beaker containing cotton fibers and sodium hydroxide solution with a ratio of 1:50 was placed in the electric heater for 10 min at a constant temperature of 20°C.

Slack mercerization was accomplished by immersing fibers in 18% (wt./wt.) sodium hydroxide solution containing a wetting agent, and then passing the samples through squeeze-rolls to remove excess reagent. These alkali-treated cotton samples were rinsed with plenty of tap water and then with deionized water several times and drained with filter paper to absorb excessive moisture. Three baskets with cotton samples of different countries were baked in the oven at 100°C thermostatic for about 1.5 h until they dried.

RESULTS AND DISCUSSION

Morphological Properties

Figure 1 shows the SEM images of native cotton fibers at the 600× magnification level. The inserted small window in each figure shows an enlarged area of the sample at 4000× magnification level. As shown in Fig 1(a), native cotton fibers showed a twisted ribbon-like shape

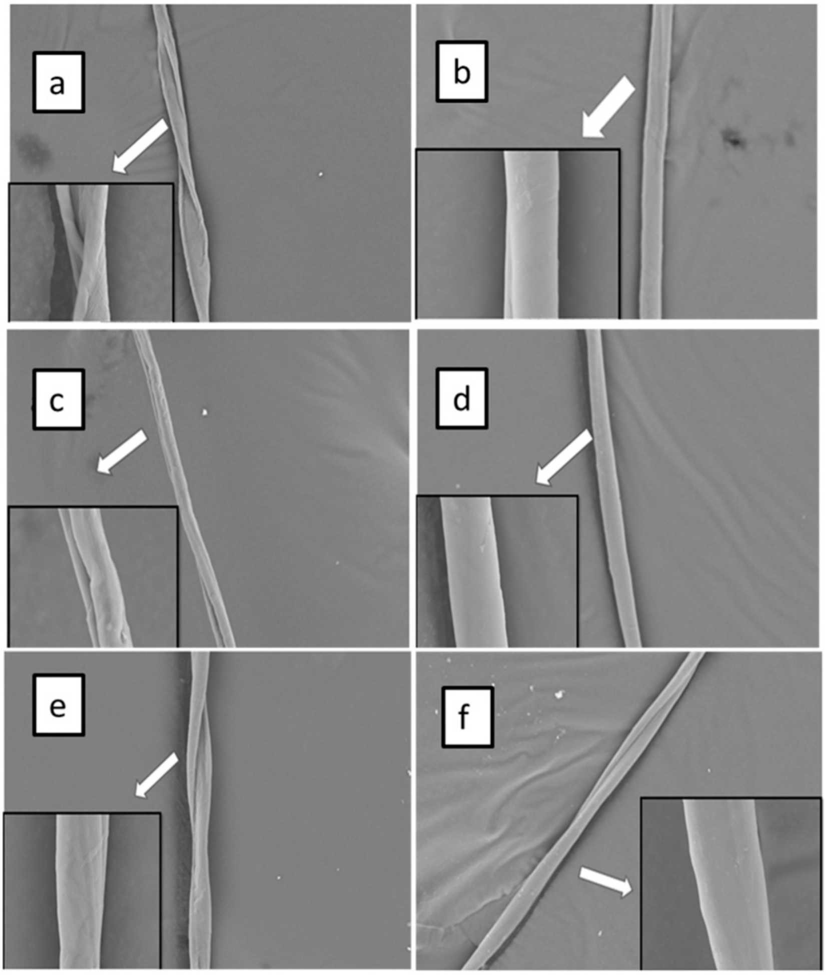

Figure 1. KAT-Af (a), BG-Au (c), and Xinjiang-Cn (e) before treatment and after treatment (b), (d), and (f), respectively.

and had a relatively smooth surface, primarily because of wax coating on the fiber surface.

After mercerization (Fig 1[b]), the entire fibers were converted into a swollen and straightened state, indicating that the assembly and orientation of microfibrils were completely disrupted. The roughened surface of mercerized cotton fibers was a direct result of the removed surface wax layer from the alkali treatment. The roughened fiber surface allows fibers to take dyes easier and may also provide a better bonding surface for fiber-polymer composite systems,

In the case of Xinjiang-Cn before treatment (Fig 1[e]) and after treatment (Fig 1[f]), it was observed that before NaOH treatment, the cotton fibers were in a twisted shape.

At a higher magnification image, it is observed clearly that in the middle of the twist, there are some tiny wrinkles, whereas some wrinkles remained on the surface of the fiber with the edges quite smoother. After NaOH treatment, the reversals disappeared, and the shape changed to an almost-round column.

Comparing with the Xinjiang-CN fibers, the KATI-Af fibers before treatment are wider. After treatment, the fiber turned into a column shape with a larger diameter. The fibers have a loose reversal structure, and remained wrinkled after treatment, as seen on the whole surface of the fiber. However, BG-Au fiber exhibited a relatively good twist length among the three samples with much increase in diameter (Fig 1). In the zoomed image, besides the wrinkles in the middle of the band, small irregular shapes with a diameter of hundreds of nanometer can be seen on the edge of the fiber band before treatment.

After treatment, the diameter of the column-shaped fiber is round, with almost no twist detected along with the fiber sample BG-Au. Among all the mercerized samples, the fibers of sample KATI-Af exhibit the smallest detrition in morphology and still some wrinkles remain on the surface and in some areas, even small branches are observed. The mercerized fibers have a loose reversal structure, and the remaining wrinkle after treatment can be clearly observed on the whole surface of the fiber.

However, BG-Au fiber exhibited a relatively good twist length among all samples. Similarly, if we see the impact of sodium hydroxide treatment on other samples, it shows us the same results in other cotton fiber samples as well.

After mercerization, the morphology of cotton fibers undergoes some physical changes, which occur in the form of cotton fiber swelling, increasing the diameter, natural convolution disappears, and natural impurities (pectin, pull-like substance, nitrogenous substances, etc.) disappear from the cotton fiber surface. The most ideal phenomenon happened was that the surface of cotton fibers became smooth and bright. In light of these results, it is suggested that the mercerization treatment greatly improves the micromorphology of cotton fibers. Through mercerization, the reversed fibers become straight and smooth, with the resulting cotton fibers appearing more lustrous with a silky look after mercerization.

Changes in the Crystalline Structure and Orientation

XRD gives an explanation of the degree of crystallinity and orientation of cotton fibers before and after treatment. Instrument model D/Max-2550PC Rigaku Co., Japan was used. Having a maximum output power of 18 kW with the specification, 2θ angle measurement range was 0.5°-145°, 2θ angle measurement accuracy was ≤ 0.01, and test temperature range can be from room temperature to 1500°C. For testing the degree of crystallinity, the fiber samples were prepared in powder form and for examining the orientation of fibers, the clean and tidy well-combed samples were prepared in sliver (tow form).

The crystalline structure of the cotton fiber swells after the treatment with NaOH. When the cotton fiber cellulose structure achieved the most swollen state, it becomes easier for the hydrated hydroxide ions to penetrate into the internal crystals and thoroughly reacted with the fiber, leading to a reduced crystallinity index value. However, the rate of penetration of the hydroxide ions became slower because of the increased viscosity of the NaOH solution at the higher concentration level (18%) (Okano and Sarko 1984, 1985).

After lye mercerizing, the crystallinity of cotton fibers decreases, as the corresponding crystal parameters change ie the angle 2θ with characteristic peaks corresponding to the interplanar spacing *d*/Å characteristic peak at 101,002 is decreasing after treatment, which shows that after lye-mercerized cotton fibers treatment, the degree of crystallinity and form of crystal has changed.

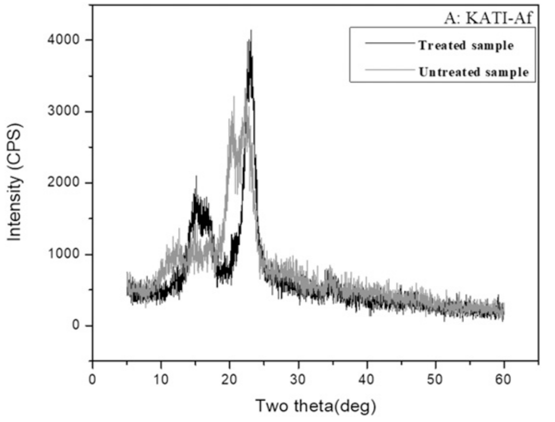

Figure 2. X-ray diffraction patterns for the BG-Au sample.

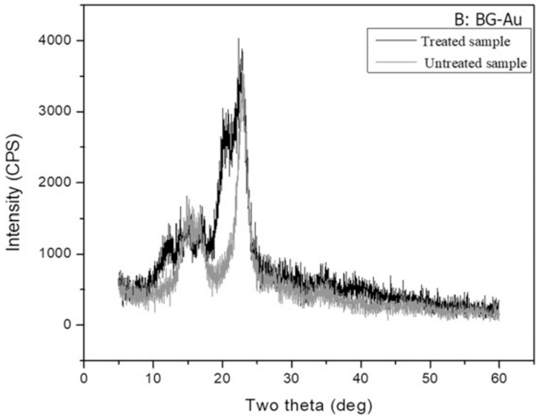

Figure 3. Xinjiang-Cn X-ray diffraction patterns for the Xinjiang-Cn sample.

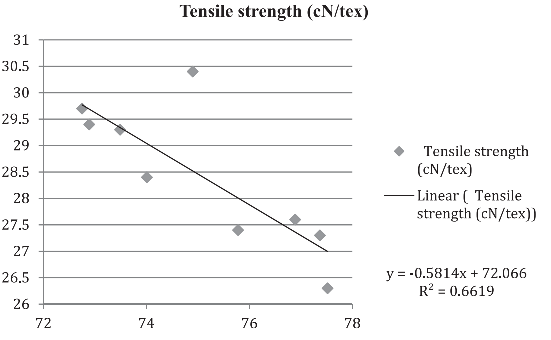

Figure 5. Correlation graph between Micronaire value and the degree of crystallinity.

Lye-treated cotton fiber XRD crystallinity of the test results can be seen from Figs 2-8. Table 8 shows the crystallization parameters (characteristic peaks corresponding to the 2θ angular position and the corresponding spacing d/Å) (Fig 9).

The XRD patterns of the different cotton sample fibers Xinjiang-Cn, KATI-Af, and BG-Au are shown in Figs 2-4. In the XRD plots, the characteristic peaks of cellulose I can be clearly seen in all the three sample curves before mercerization (22.883, 22.918, and 22.844 of Xinjiang-Cn, KATI-Af, and BG-Au, respectively). However, in the curves of the mercerized samples, the change in 2θ value clearly represents the change in polymorphic crystalline cellulose and new peaks appearance, indicating the decrease in the values of crystallinity (21.976, 21.929, and 21.935 of Xinjiang-Cn, KATI-Af, and BG-Au, respectively). A possible explanation is that parts of cellulose-I are converting to cellulose-II after mercerization treatment. when cellulose is dipped in aqueous alkali solutions, they swell and the ionized H+ in hydroxyl groups are replaced by Na+ to combine into stable compounds, and the lattice of cellulose changes considerably after elimination of the reagent, causing a great change in the crystal structure of treated cellulose (Liu and Wang 2011).

Similarly, different degrees of peak shape, peak intensity, and the diffraction angle of cotton fibers before and after treatment indicate the amorphous regions are increasing and the crystallinity is decreasing in the case of treated cotton fibers. The decrease in crystallinity is noted in all samples. The values obtained for crystallinity changes from XRD are shown in the following

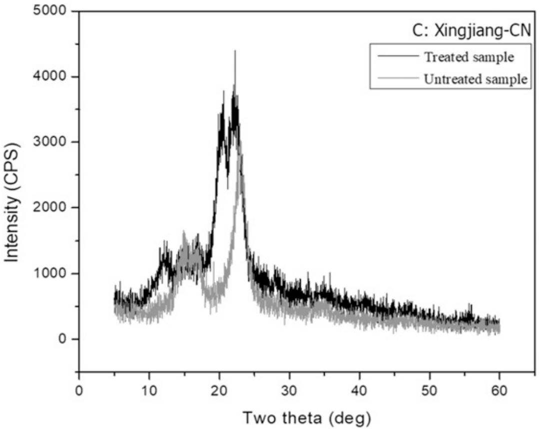

Figure 4. Correlation graph between tensile strength and degree of crystallinity.

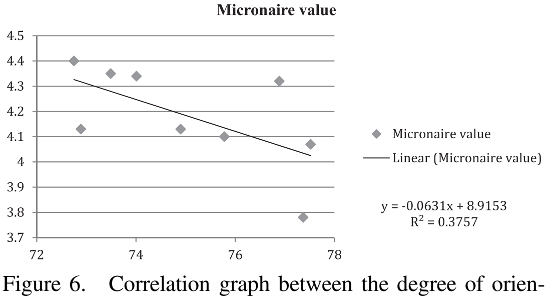

Figure 6. Correlation graph between the degree of orientation and tensile strength.

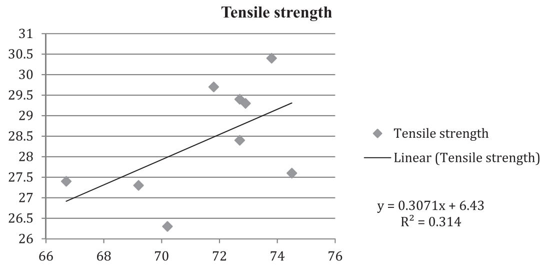

Figure 7. Correlation graph between breaking strength and Micronaire value.

paragraphs. Table 9 shows the crystallinity before and after the processing of cotton fibers for all the samples.

The decrease in crystallinity was observed in all the samples (Table 10). The degree of crystallinity is the percentage share of the crystallization zone; with the reduction in the crystallization zone, the degree of crystallinity in cotton fiber samples decreases (Öztürk et al 2009; Chen 2014).

The degree of crystalline orientation of cotton fiber samples was also studied with XRD, by measuring the percentage of cellulose polymer chains that are parallel to the axis of the fiber. The results are shown in Table 10, from which it can be seen that the mercerization greatly increased the orientation degree of the polymer chains in cotton fibers, which can be attributed to the decrease in crystallinity (Zeronian 1976; Hearle 1979).

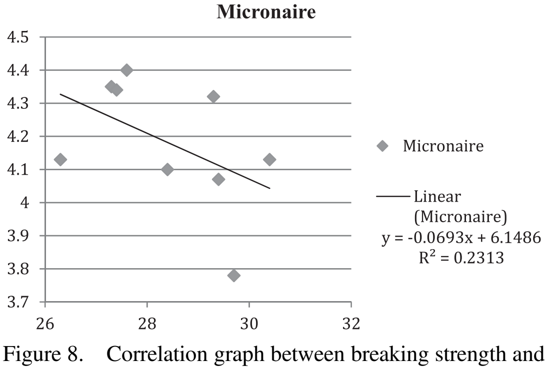

Figure 8. Correlation graph between breaking strength and upper half mean length.

From Table 10, we can see that the decrease in the degree of crystallinity increases the degree of orientation that occurs for each sample of cotton fibers. It is a well-known reality that cotton fibers can be significantly modified after mercerization in terms of crystallinity, the orientation of crystallites, and the orientation of macromolecular chains, all leading to an increase in the mechanical properties. After mercerization, fibers become more amorphous and less crystalline but with the improved orientation of cotton fiber's micro- and macro-units (Hussain et al 1982).

Correlation Analysis of Physical Properties and Crystal Structure of Cotton Fiber

For the relationship between breaking strength, elongation at break, and crystallinity of cotton fiber, a correlation graph was drawn between the breaking strength and degree of crystallinity of several cotton fibers according to Tables 9 and 10 and shown in Table 11. The relationship between the tensile strength and crystallinity of cotton fibers (the regression equation obtained from Fig 4) is expressed as follows.

$$y = -0.5814x + 72.066, \quad R^2 = 0.6619.$$

Therefore, the breaking strength of cotton fibers in different cotton areas was negatively correlated with the crystallinity and the correlation coefficient was found to be 0.8135 as shown in Fig 4. In short, we can see that the smaller the crystallinity of the cotton fiber, the greater will be the breaking strength.

Correlation between Micronaire and Crystallinity

The linear regression equation obtained from Fig 5 for the correlation between the Micronaire value and crystallinity is given in the following equation:

$$y = -0.0631x + 8.9153, \quad R^2 = 0.3757.$$

The Micronaire value and crystallinity are shown in Table 12.

Table 8. Crystalline parameter changes in treated and untreated cotton fibers.

Samples	Characteristic peak 2θ position			Angular spacing *d*/Å		
	101	$\overline{101}$	002	101	$\overline{101}$	002
KATI-Sa (B)	13.896	13.753	22.834	5.9451	5.2950	3.8949
KATI-Sa (T)	11.194	19.201	22.013	7.2676	4.3908	4.0346
Xinjiang-Cn (B)	14.890	16.767	22.893	5.9436	5.2927	3.8921
Xinjiang-Cn (T)	11.365	19.284	21.943	7.1463	4.3736	4.0463
KATI-Ma (B)	14.745	16.564	22.098	5.7940	5.3562	3.8097
KATI-Ma (T)	12.368	20.176	21.906	7.1480	4.3949	4.0456
BG-Tu (B)	14.345	16.450	22.385	5.8978	5.2783	3.8672
BG-Tu (T)	12.295	20.190	21.922	7.2785	4.3782	4.0498
KATI-Af (B)	14.674	16.840	22.943	5.9342	5.2675	3.8453
KATI-Af (T)	12.538	20.272	21.809	7.2789	4.3455	4.5699
Pakistan (B)	15.097	17.557	23.178	5.9676	5.2782	3.8873
Pakistan (T)	12.589	20.348	22.134	7.0863	4.3424	4.0167
India (B)	14.134	15.846	23.022	5.8453	5.4532	3.8335
India (T)	12.332	20.231	22.156	7.1346	4.3567	4.4573
KATI-Ug (B)	13.613	15.643	22.685	5.9433	5.3457	3.4579
KATI-Ug (T)	11.455	19.453	19.456	7.4523	4.4574	4.0659
BG-Au (B)	14.345	16.455	22.344	5.9348	5.3678	3.8567
BG-Au (T)	12.145	20.187	21.955	7.2543	4.3945	4.0787

Micronaire value and crystallinity of cotton fiber were in a negative correlation with $R = 0.6129$. The correlation coefficient between the Micronaire value and the crystallinity is 0.6129. Correlation of degree of orientation and physical properties are presented in Table 13.

Correlation between the Tensile Strength of Different Areas of Cotton Fiber and the Degree of Orientation

The regression equation and correlation coefficient obtained from Fig 6 is given as follows:

$$y = 0.3071x + 6.43, \quad R^2 = 0.314.$$

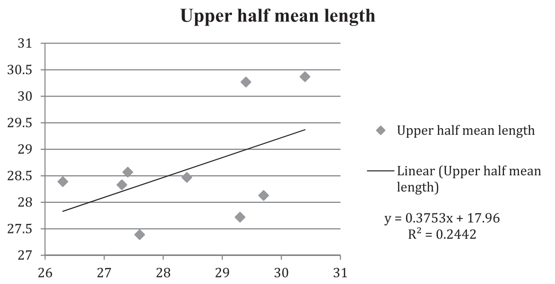

Figure 9. X-ray diffraction patterns for the KATI-Af sample.

The correlation between the degree of orientation angle and tensile strength of different cotton-producing regions was positively correlated having a value of correlation coefficient as R 0.5603.

The breaking strength of cotton fibers for different cotton areas is negatively correlated with the crystallinity having a correlation coefficient value 0.8135, which reveals a strong correlation. Correlation shows that the higher the tensile strength of cotton fibers, the lower the degree of crystallinity. This causes better mechanical properties of textile material. BG-Tu, BG-Au, and Pakistani cotton samples that have maximum tensile strength will have better mechanical properties. Micronaire value and crystallinity of cotton fiber were in negative correlation with $R = 0.6129$, a bit stronger correlation, which reveals the higher the Micronaire value, the smaller will be the crystallinity.

Correlation Analysis between Macroscopic Properties of Cotton Fibers

This section of the article focuses on correlation analysis between breaking strength and the upper half of the mean length and Micronaire of several areas of cotton fiber physical properties. The

Table 9. Crystallinity data of unmercerized and mercerized samples.

Sample	Crystallinity (%)	
	Mercerized	Unmercerized
BG-Tu	73.49	68.08
KATI-Af	74.01	62.91
KATI-Ma	75.78	64.95
BG-Au	74.90	66.96
KATI-Ug	76.89	60.40
KATI-Sa	77.52	68.37
Pakistan	72.75	64.80
India	72.89	66.65
Xinjiang-Cn	77.37	64.90

Table 11. Tensile strength and degree of crystallinity.

Cotton	Tensile strength	Degree of crystallinity
KATI-Ma	27.4	75.78
KATI-Af	28.4	74.01
KATI-Ug	27.6	76.89
KATI-Sa	26.3	77.52
BG-Tu	29.3	73.49
BG-Au	30.4	74.90
India	29.4	72.89
Xinjiang-Cn	27.3	77.37
Pakistan	29.7	72.75

correlation regression equation of breaking strength of different cotton-producing regions and Micronaire value is as follows, with $R^2 = 0.2313$,

$$y = -0.0693x + 6.1486.$$

Moreover, the correlation between the breaking strength and Micronaire value is negative, but the correlation coefficient value is not so strong, which is 0.4809.

The regression equation correlation for breaking strength of different cotton-producing regions and upper half average length of the cotton fibers is given in the following equation, with $R^2 = 0.2442$,

$$y = 0.3753x + 17.96.$$

In addition, breaking strength for different region samples and the upper half are in positive correlation, the correlation coefficient is $R = 0.4941$ (Table 14).

The correlation graph between the breaking strength of different cotton-producing regions and upper half mean length and graph between breaking strength of different cotton-producing regions is given in Fig 8.

From all these results, we conclude that the correlation between macroscopic properties is not very strong, so they are not significantly related to each other. But somehow, they can impact each other as the coefficient correlation value is 0.4809. In the case of the correlation between Micronaire value and tensile strength, there is a positive correlation, but not very strong. It does show that the lower the Micronaire value of cotton fibers (fine fibers), the higher the tensile strength of cotton fibers.

CONCLUSIONS

This study covers the five origins of China, Africa, Australia, India, and Pakistan which are considered as major countries in cotton fiber production raw cotton. HVI testing system is used

Table 10. Crystalline orientation of mercerized cotton fiber.

Cotton	Degree of orientation (%)	
	Raw cotton	Alkalized cotton
KATI-Ma	61.7	66.7
BG-Tu	64.8	72.9
KATI-Sa	67.0	70.2
Xinjiang-Cn	62.2	69.2
BG-Au	67.2	73.8
KATI-Af	64.2	72.7
India	69.1	72.7
KATI-Ug	68.4	74.5
Pakistan	65.8	71.8

Table 12. Micronaire value and degree of crystallinity.

Cotton samples	Micronaire value	Degree of crystallinity
India	4.13	72.89
KATI-Ma	4.10	75.78
KATI-Af	4.34	74.01
BG-Tu	4.35	73.49
Pakistan	4.40	72.75
BG-Au	4.13	74.90
KATI-Ug	4.32	76.89
KATI-Sa	4.07	77.52
Xinjiang-Cn	3.78	77.37

Table 13. Tensile strength and degree of orientation.

Samples	Tensile strength	Degree of orientation
Xinjiang-Cn	27.3	69.2
KATI-Ma	27.4	66.7
KATI-Ug	27.6	74.5
India	29.4	72.7
Pakistan	29.7	71.8
BG-Au	30.4	73.8
BG-Tu	29.3	72.9
KATI-Sa	26.3	70.2
KATI-Af	28.4	72.7

to compare the physical properties of cotton fibers by several indicators as tensile strength, elongation at break, upper half mean length, reflectivity, short fiber content, Micronaire value, and yellowness. After testing, the analysis was performed for the selection of raw cotton from different countries for spinning performance and at the same time, several cotton fibers were processed for mercerization. Crystallinity and orientation parameters of the cotton fibers were examined by XRD, FTIR, and SEM to analyze the changes before and after the mercerization of cotton fibers. A linear regression analysis was also performed to analyze the relationship between several microstructure properties with macroqualities (physical properties) of cotton samples; a corresponding theoretical analysis is drawn. Raw material from different regions should be tested to examine the physical properties ie length, impurities, Micronaire values, etc. because of various eco-environment of different regions. For better mechanical properties, fibers having the highest values in tensile strength should be preferred because of its strong negative effect on the microstructural property ie crystallinity. A decrease in crystallinity will lead to better mechanical properties eg an increase in the elongation at break of cotton fibers will lead to yarn that is more elastic. From correlation analysis, it is observed that the higher the tensile strength of cotton fibers, the higher the values of the degree of orientation and the higher the orientation of microfibrils, the better the strength of fiber properties. The raw material with relative higher tensile properties should be segregated to obtain the highest values of mechanical properties of the end product. Micronaire value shows a negative but no strong correlation with the macroscopic property of tensile strength, the smaller the value of Micronaire, the higher the values of tensile strength. Therefore, the separation of raw material can be performed according to their different Micronaire values.

Table 14. Macroscopic properties of cotton fibers.

Samples	Tensile strength	Micronaire	Upper half mean length
Xinjiang-Cn	27.3	4.35	28.33
KATI-Ma	27.4	4.34	28.57
KATI-Ug	27.6	4.4	27.39
India	29.4	4.07	30.27
Pakistan	29.7	3.78	28.13
BG-Au	30.4	4.13	30.37
BG-Tu	29.3	4.32	27.72
KATI-Sa	26.3	4.13	28.39
KATI-Af	28.4	4.1	28.47

REFERENCES

Chen H (2014) Chemical composition and structure of natural lignocellulose. Biotechnology of lignocellulose. Springer, Dordrecht, Netherlands, pp. 25-71.

El Mogahzy YE, Broughton R Jr., Lynch W (1990) A statistical approach for determining the technological value of cotton using HVI fiber properties. Textile Res J 60(9): 495-500.

Ethridge D, Zhu R (1996) Prediction of rotor spun cotton yarn quality: A comparison of neural network and regression algorithms. Beltwide Cotton Conferences, January 9-12, 1996, Nashville, TN. Editors: C. Paul Dugger & Debbie A. Richter.

Garner WE, Shanklin EH, LaFerney PE (1970) Fiber quality and ginning performance of machine picked and stripped cotton, southeastern area, 1964-66. U.S. Dep Agric Mark Res Rep 1964–66. Marketing Research Report No. 852, USDA-ARS, Washington, DC, 1970, p. 30.

Gordon S, Hsieh Y-L (2006) Cotton: Science and technology. Woodhead Publishing, Cambridge, England.

Hearle JWS, Sparrow JT (1979) Mechanics of the extension of cotton fibers. I. Experimental studies of the effect of convolutions. J Appl Polym Sci 24:1465-1477.

Hosseinali F (2012) Investigation on the tensile properties of individual cotton (*Gossypium hirsutum* L.) fibers. Thesis for Master of Science, Texas Tech University, Texas, US.

Hussain G, Iyer KK, Patil N (1982) Influence of mercerization and crosslinking on the dynamic and static moduli of cotton yarns. Textile Res J 52(10):663-665.

Lawrence CA (2003) Fundamentals of spun yarn technology. CRC Press, New York, USA.

Li Y, Yeung K, Shen Y, Newton E, Yao L (2003) The World Trade Organization and international denim trading. Woodhead Publishing, Cambridge, England.

Liu J, Wang F (2011) Influence of mercerization on microstructure and properties of kapok blended yarns with different blending ratios. J Eng Fibers Fabrics 6(3):63-68.

Long RL, Bange MP, Gordon SG, van der Sluijs MH, Naylor GR, Constable GA (2010) Fiber quality and textile performance of some Australian cotton genotypes. Crop Sci 50(4):1509-1518.

Majumdar PK, Majumdar A (2004) Predicting the breaking elongation of ring spun cotton yarns using mathematical, statistical, and artificial neural network models. Textile Res J 74(7):652-655.

Memon H, Ali Khoso N, Memon S (2015) Effect of dyeing parameters on physical properties of fibers and yarns. Int J Appl Sci Eng Res 4(4):401-407.

Negm MA, Sanad S, Kugler G (2015) A comparison of HVI, AFIS and CCS cotton testing method. 12th Meeting of the Inter-Regional Cooperative Research Network on Cotton for the Mediterranean and Middle East Region, Sharm El-Sheikh, Egypt, October 6-10, 2015.

O'sullivan AC (1997) Cellulose: The structure slowly unravels. Cellulose 4(3):173-207.

Okano T, Sarko A (1984) Mercerization of cellulose. I. X-ray diffraction evidence for intermediate structures. J Appl Polym Sci 29(12):4175-4182.

Okano T, Sarko A (1985) Mercerization of cellulose. II. Alkali–cellulose intermediates and a possible mercerization mechanism. J Appl Polym Sci 30(1):325-332.

Öztürk HB, Potthast A, Rosenau T, Abu-Rous M, MacNaughtan B, Schuster KC, Mitchell JR, Bechtold T (2009) Changes in the intra-and inter-fibrillar structure of lyocell (TENCEL®) fibers caused by NaOH treatment. Cellulose 16(1):37.

Patel G, Iyer PB, Sreenivasan S, Iyer KK (1990) Reversals in cotton: A study with scanning Electron microscopy. Textile Res J 60(12):771-774.

Saville BP (1999) Physical testing of textiles. Woodhead Publishing, Cambridge, England.

Ugbolue SCO (1990) Structure/property relationships in textile fibres. Textile Prog 20(4):1-43.

Wakelyn PJ, Bertoniere NR, French AD, Thibodeaux DP, Triplett BA, Rousselle M-A, Goynes WR Jr., Edwards JV, Hunter L, McAlister DD (2006) Cotton fiber chemistry and technology. CRC Press, Boca Raton, FL. 95 pp.

Wang X, Zhang L, Evers JB, Mao L, Wei S, Pan X, Zhao X, van der Werf W, Li Z (2014) Predicting the effects of environment and management on cotton fibre growth and quality: A functional–structural plant modelling approach. AoB Plants 6:plu040.

Yang S, Gordon S (2016) A study on cotton fibre elongation measurement. 33rd International Cotton Conference, Bremen, Germany, March 16-18, 2016.

Yue Y, Han G, Wu Q (2013) Transitional properties of cotton fibers from cellulose I to cellulose II structure. BioResources 8(4):6460-6471.

Zeronian SH, Alger KW, Cabradilla KE (1976) Tensile properties of cotton treated with alkali metal hydroxides. J Appl Polym Sci 20:1689-1693.

Zhou Y, Zhao M, Ma H, Xia X-m (2008) Effect of verticillium wilt with different grades on main cotton yield characters. Shandong Agric Sci 9:81-83.

Appendix

Appendix Table 1. Cotton fiber length grading (Negm et al 2015).

Length level (mm)	Upper half average length (mm)
25 mm	25.9 mm and below
26 mm	26.0-26.9 mm
27 mm	27.0-27.9 mm
28 mm	28.0-28.9 mm
29 mm	29.0-29.9 mm
30 mm	30.0-30.9 mm
31 mm	31.0-31.9 mm
32 mm	32.0 mm and above

Appendix Table 2. Cotton fiber length uniformity index standard (Negm et al 2015).

Grading	LUI (%)
Very low	<77.0
Low	77.0-79.9
Medium	80-82
High	83-85
Very high	>85.0

Appendix Table 3. Classification standard of cotton fiber Micronaire value (Saville 1999).

Scale	Micronaire value
Very fine	up to 3.1
Fine	3.2-3.9
Average	4.0-4.9
Coarse	5.0-5.9
Very coarse	6.0 and above

Appendix Table 4. Tensile strength index (Lawrence 2003).

Degree of strength	Strength (cN/tex)
Weak	<24.0
Intermediate	24.0-25.9
Average	26.0-28.9
Strong	29.0-30.9
Very strong	>31.0

Appendix Table 5. Elongation at break index (Lawrence 2003).

Grade	Elongation at break (%)
Very low	5.0 And below
Low	5.0-5.8
Medium	5.9-6.7
High	6.8-7.6
Very high	7.7 above