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Content

The Cotton Innovations Newsletter is published twelve times annually. Contributed articles, pictures, cartoons, and feedback are welcome at any time. Please send contributions to the General Editors (see below). The editors reserve the right to edit. The deadline for contributions is one month before the publication date.

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DEAR COLLEAGUES

GREETINGS FROM THE ICRA. I AM DELIGHTED TO ANNOUNCE THAT THE WEBSITE FOR THE WORLD COTTON RESEARCH CONFERENCE-8 (WCRC-8) IS NOW ACCESSIBLE AT HTTP://WWW.ICAC-WCRC.COM/INDEX/HOME.
INTERNATIONAL COTTON RESEARCHERS ASSOCIATION-ICRA AND INTERNATIONAL COTTON ADVISORY COMMITTEE-ICAC IS OPEN TO OFFERING FINANCIAL SUPPORT TO YOUNG RESEARCHERS, PARTICULARLY FROM LEAST DEVELOPED AND DEVELOPING COUNTRIES, TO ATTEND THE CONFERENCE. SPONSORSHIP WILL BE GRANTED BASED ON THE MERIT OF SUBMITTED FULL PAPERS, PARTICULARLY THOSE SELECTED FOR ORAL PRESENTATION.

WE EAGERLY ANTICIPATE THE OPPORTUNITY TO MEET YOU IN TASHKENT.

DR. MOHAMED NEGM
ICRA CHAIRMAN

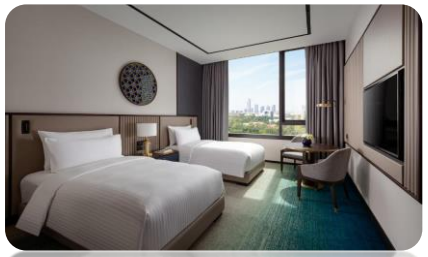
WORLD COTTON RESEARCH CONFERENCE-8 TASHKENT
7-11 OCTOBER 2024

Conference 2024
INTERNATIONAL
cotton genome
INITIATIVE

WCRC-8 TASHKENT-UZBEKISTAN
THE LIST OF HOTELS FOR PARTICIPANTS

★★★★★

Intercontinental Tashkent



Adress: 2, Shahrisabz street
Prices: 250-500

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Hyatt Regency

Adress: 1A, Navoi street
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City Palace Hotel

Address: 15, Amir Temur street
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Prices: 116-260

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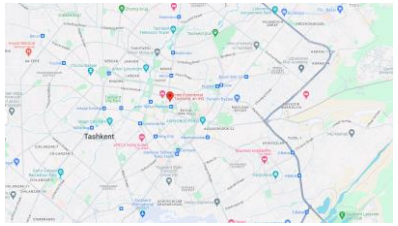

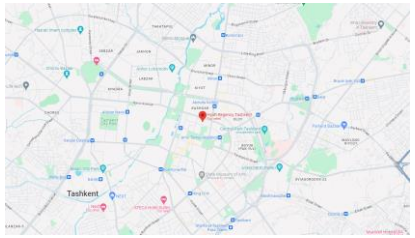

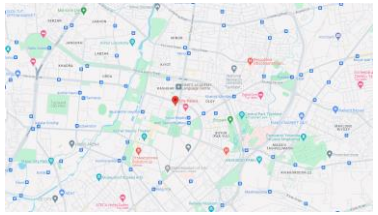

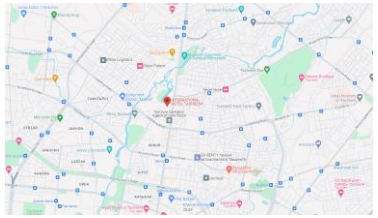

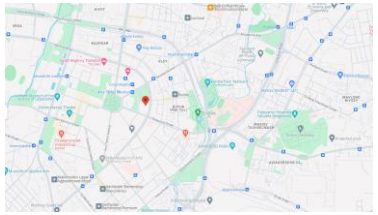

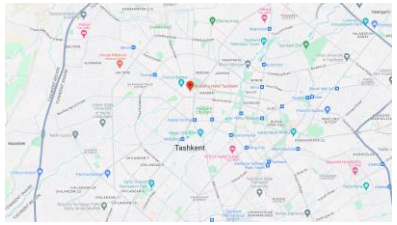

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https://citypalace.uz/

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https://legrandeplaza.com

https://www.booking.com/hotel/uz/mulberry-toshkent.ru.html

Tashkent

Tour of Tashkent

One day

10:00

- Starting excursion in Tashkent. Visit to the New City of Tashkent by bus with a guide

Independence Square

Amir Temur Square

Tashkent Subway

Beginning of a sightseeing tour of the old city of Tashkent.

13:00 - Lunch at a restaurant

14:00-18:00 - Continuation of the excursion

Kukeldash Madrasah

Alisher Navoi Park

Madrasah Abdul Qasim

Small mosque Chorsu bazaar

Samarkand

Tour of Samarkand

One day

07:24

- **Departure to Samarkand by high-speed train “Afrosiab” (economy/sedentary)**
- ***train schedule is subject to change*

09:37

Arrival in Samarkand,
(2 hours 13 minutes)

As exiting the station, guests are met by a bus and a guide.

10:00-18:00

- **Tour of Old Samarkand**

13:00

Dinner

Registan Square

Mausoleum Gur-Emir (Tomb of Amir Timur)

Shahi Zinda

Bibi Khanum Mosque

Ulugbek Observatory

Mausoleum of St. Daniel

18:32-20:47

18:32 Departure to Tashkent by high-speed train "Afrosiab"
***train schedules are subject to change*

Alginate calcium/low-quality cotton fibers composite beads as biosorbent for dye removal: the role of plasma treatment

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Abstract

This study presents the preliminary investigation of composite hydrogels comprising alginate calcium and low-quality cotton fibers, with and without plasma treatment, as novel absorbents for methylene blue (MB) dye. It was found that plasma treatment enhanced the homogeneity of crushed cotton fibers and facilitated their entrapment in the alginate calcium hydrogel matrix. Fourier Transform Infrared spectroscopy (FTIR) results suggested that plasma treatment introduced more carboxylate groups on the fiber surface. As a result, a slight enhancement of MB dye adsorption capability was achieved. Our current work lays the groundwork for further exploration into the dynamics of hydrogel systems and dye adsorption mechanisms, as well as potential applications in wastewater treatment.

Introduction

Cotton (*Gossypium* spp.) stands as a primary natural fiber crop globally and is the cornerstone of textile industries worldwide (Kohel et al., 2001). However, an inherent drawback of cotton is its variable quality, where low-quality cotton exhibits lower tensile strength and poses challenges during processing. This results in materials that are costly to handle but offer limited value (Bradow & Davidonis, 2000). Despite this challenge, global cotton consumption is predicted to be 116.2 million bales by 2024, according to a recent report (Soley, 2023), which increased by 6.6 million bales compared to 2023. This rise in demand will generate a surplus of low-quality cotton fibers (LCF), underscoring the critical need to repurpose these low-value materials effectively (Kamble & Behera, 2021; T. Zhang et al., 2021). Though low-quality fiber materials can be regenerated into cellulosic bioproducts, the wet chemistry method inevitably involves the toxicity of the solvents and the instability of the dissolving process (Liu et al., 2019). Therefore, a green, sustainable, simple, and effective approach is strongly demanded.

Hydrogels, polymers that form a three-dimensional network known for their high-water content and porosity, have garnered significant attention for their versatile applications across various fields, from biomedical engineering to environmental remediation (Gao et al., 2023; Zhang et al., 2023). In the realm of environmental remediation, hydrogels were usually utilized as adsorbents for removing hazardous substances (i.e., dye, heavy metal ions) from wastewater due to their excellent characteristics such as separability from effluent, recyclability and capability to host functional adsorbents (Kalia, 2021). Among the hydrogel family, alginate, a seaweed-derived polymer, has been widely used due to its excellent gelling properties, strong affinity to adsorb cationic hazardous materials, and efficient immobilization towards functional adsorbents (Soliman et al., 2017). In our previous works, we have successfully repurposed cotton linter waste (Zhang et al., 2023) and LCF (Zhang et al., 2024) in the form of alginate/cotton fibers composite hydrogel biosorbent for dye removal.

Plasma treatment has emerged as a promising and environmentally friendly technique for enhancing the properties of cotton products (Jelil, 2015). By subjecting cotton fibers to controlled plasma discharge, various surface modifications can be achieved, such as increased hydrophilicity (Bhat et al., 2011) and improved solubility (Rumi et al., 2022). In this work, we investigated the role of plasma treatment in composite hydrogel preparation and dye adsorption. Specifically, we utilized plasma to treat the LCF and employed an alginate calcium hydrogel matrix to host LCF with/without plasma treatment for methylene blue dye removal. To the best of our knowledge, this work is the first report elucidating the effect of plasma treatment on alginate/LCF hydrogel systems. Different characterizations, including Fourier Transform Infrared spectroscopy (FTIR), scanning microscopy (SEM), and ultraviolet-vis spectroscopy (UV-vis), were employed to characterize the structure-properties relationship and evaluate dye adsorption capability.

Experimental

Materials

Calcium chloride ($CaCl_2$, ≥95%, ACROS), sodium alginate (SA, MP biomedical, Inc), and methylene blue (MB, ACROS) were used without any further purification. Milli-Q water was used as the basic solvent for every experiment. Bulky low-quality cotton fibers (Micronaire value of 2.4, Average M_w of 394899, PDI of 2.09) were provided by the Fiber and Biopolymer Research Institute in Lubbock, Texas. The bulky fibers were then crushed into fiber powders using a Wiley mill passing a 60-mesh screen. The as-prepared fiber powders were utilized as the starting materials for plasma treatment and hydrogel fabrications. For plasma-treated LCF, LCF were oven-dried at 105°C overnight and subsequently subjected to microwave oxygen plasma treatment in a plasma chamber (PLASMATECH Inc., Erlanger, KY) (flow rate = 60 mL/min, pressure = 25 Pa, generator frequency = 2.45 GHz, time = 40 min). The plasma-treated LCF was then crushed into fiber powders in the same way.

Sample preparation

SA was first dissolved in Milli-Q water after an overnight magnetically stirring process to make a 1 wt.% SA solution. The obtained SA solution then served as a "parental solvent" to disperse the LCF or plasma-treated LCF via overnight magnetic stirring, where SA/LCF composite suspension was obtained. The weight ratio between SA and cotton fibers was fixed to be 1/1. The SA solution, as well as

composite suspension, were then transferred into the syringe and extruded into the $CaCl_2$ bath (1 wt.% water solution) at an extrusion rate of 1 ml/min. Once the droplets contacted the bath, the gelation occurred immediately. Upon accomplishing the gelation, the hydrogel beads were withdrawn from the $CaCl_2$ bath and washed thoroughly using excessive Milli-Q water. These hydrogel beads were then freeze-dried for FTIR and SEM tests. For simplification, pure alginate, alginate/LCF, and alginate/plasma-treated LCF were denoted as AC0, AC5-U, and AC5-P, respectively.

Characterizations

The water content of different hydrogel beads was tested based on a weighting method following equation 1:

$$WC = \frac{W_f - W_d}{W_d} * 100\% \quad (1)$$

where W_f and W_d denoted the weight of fresh and freeze-dried hydrogel beads, respectively.

The surface morphology of LCF with and without plasma treatment and freeze-dried gel samples was observed in the scanning electron microscope (SEM, TM-1000, Hitachi). Each sample was tested under the operation voltage of 15 kV. Neither staining nor chemical etching techniques were introduced. The chemical structures were analyzed in a Fourier-transform infrared spectroscopy (FTIR, Spotlight 400, PerkinElmer) in an attenuated total reflectance mode. The testing condition was under 32 scans and a resolution number of 4 cm^{-1}. For dye adsorption tests, the pre-weighted hydrogel beads (~0.5 g) were placed in an MB dye solution (10 ml, 30 mg L^{-1}) for at least two days to reach the equilibrium adsorption state. Then, the dye solution was tested by a UV-vis spectrometer (Lambda 650, PerkinElmer), and the dye adsorption capacity (q_e) was calculated using the following equation:

$$q_e = (C_0 - C_e)V/m \quad (2)$$

where V, C_e, $C_{0,\ and\ m\ refer}$ to the volume of methylene blue solution, methylene blue concentrations in the equilibrium and initial state, and the dry gel weight, respectively.

Results and Discussion

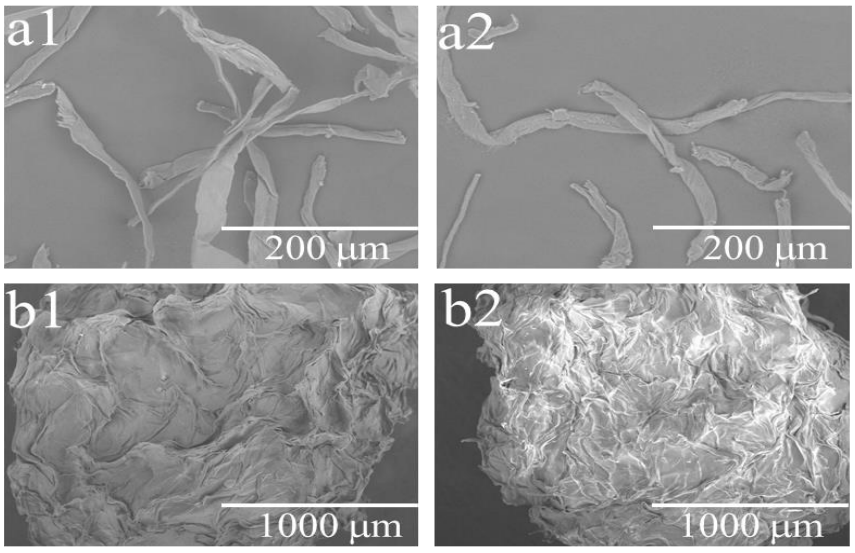

Figure 1. SEM morphological results of different materials: (a1) cotton fibers without treatment; (a2) cotton fibers treated by plasma; (b1) AC5-U; (b2) AC5-P.

Figure 1 illustrates the morphological results of low-quality cotton fibers and their composite gels. One can see in Figure 1 (a1) and (a2) that LCF without and with plasma treatment did show some interesting disparities. In Figure 1 (a1), untreated LCF displayed a less homogeneous diameter distribution compared to those in LCF with plasma treatment (Figure 1 (a2)), where certain fibers with larger diameters were evident. This disparity might be accounted by the fact that the plasma treatment introduces functional groups, mitigating the strong inter- and intra-molecular interactions within cellulose chains (Alonso-Montemayor et al., 2022; Vandenabeele & Lucas, 2020) and thereby promoting a more uniform appearance after post-mechano-treatment in the Wiley mill. In addition, the removal or etching effect caused by plasma might also played a role in it (Bhat et al., 2011). With respect to composite systems, a "mountain ridge" morphological character was observed in both AC5-U and AC5-P, which was similar to the results found in our previous work (Zhang et al., 2023). In addition, visible cotton fibers were distributed uniformly within the alginate matrices (Figure 1 (b1) and (b2)), indicating that the post-processing steps (solution mixing, gelation, and freeze-drying) barely affected the character of fibers. It should be noted that Figure 1 (b2) displayed a whiter sampling area, indicating a "charging" phenomenon suggestive of increased surface charges in AC5-P.

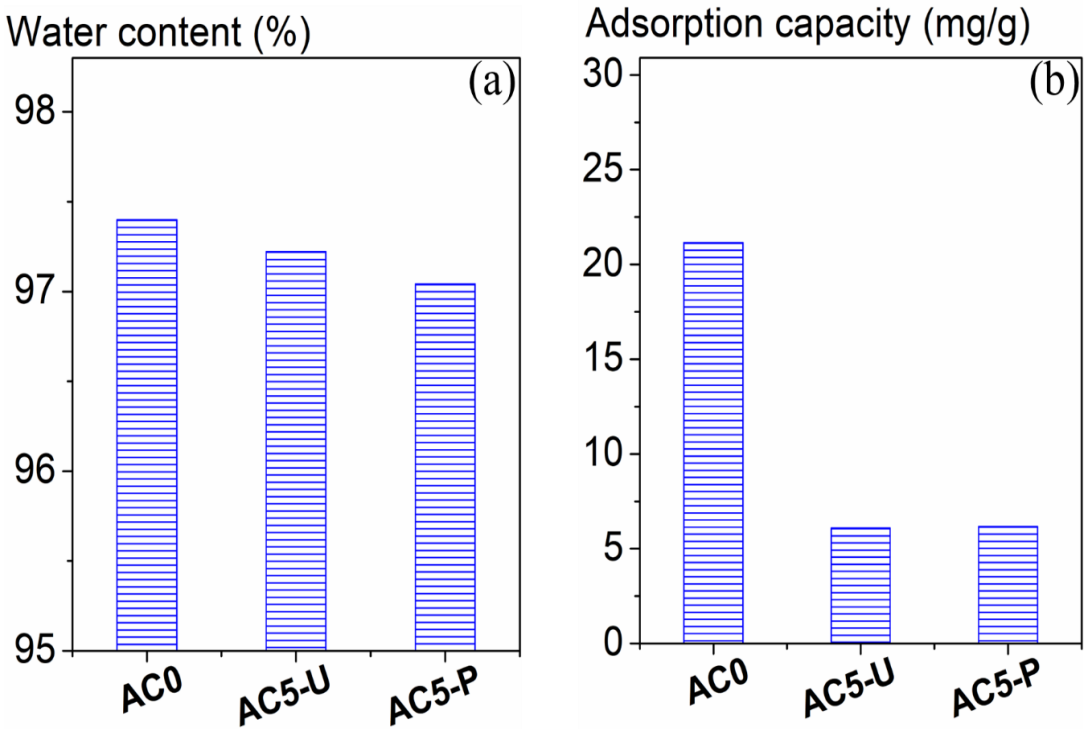

Figure 2. Water content (a) and methylene blue dye adsorption capacity (b) of hydrogel beads systems.

Figure 2 (a) displays the water content of different hydrogel systems. From Figure 2 (a), it was evident that the introduction of cotton fibers inevitably led to a reduction in water content. Such a trend was anticipated, considering the relatively limited water adsorption capacity of LCF, which acts more as invaded fillers than contributors to the hydrogel network. However, it is interesting to note that AC5-P displayed the lowest water content value. This might be again due to the surface functional groups brought by the plasma treatment, which facilitate the immobilization of cotton fibers within the alginate matrix.

Figure 2 (b) displays the MB adsorption results. One can see that the introduction of cotton fibers decreased the MB dye adsorption capacity. While such a decrease was expected due to the low dye adsorption capability of LCF, it contradicted our previous work (Zhang et al., 2024), where the LCF almost exhibited no decrease in dye adsorption capacity in an alginate acid system. This discrepancy might be attributed to their different crosslinking structures. Further investigation into such interesting phenomenon will be considered. Compared to AC5-U, the MB dye adsorption capacity of AG5-P was slightly higher, which might be due to its higher amount of surface charge groups.

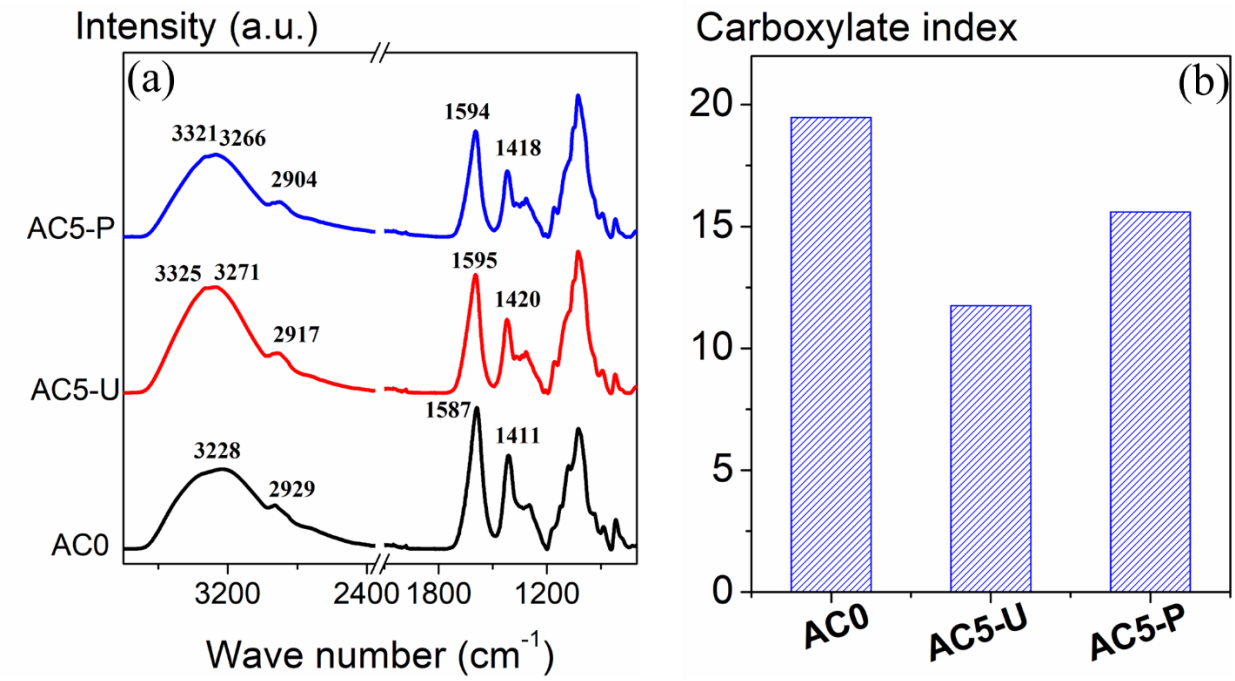

Figure 3. FTIR curves (a) and carboxylate index (b) of different materials.

FTIR spectra of different materials are displayed in Figure 3 (a). AC0 exhibited the characteristic peaks at 3228, 2929, 1587, and 1411 cm^{-1}, which corresponded to -OH stretching, C-H stretching, carboxylate (COO^-) asymmetric vibration, and COO^- symmetric vibration (Lawrie et al., 2007), respectively. In the case of composite systems, both AC5-U and AC5-P displayed similar spectra. However, compared to AC0, dual peaks in the -OH stretching region were observed, which were attributed to the cellulose in the LCF (Zhang et al., 2023). Such features

suggested that the attachment of cotton fibers to the surface of the gel bead correlated well with the previous SEM result (Figure 1 (b1)-(b2)). In addition, there existed shifts of -OH stretching as well as COO^- symmetric and asymmetric vibrations, suggesting the existence of possible special interactions between alginate and cotton fibers (Song et al., 2015).

To assess the charge status, the carboxylate index that is proportional to the amount of carboxylate groups was calculated using eq. 3:

$$\boldsymbol{Carboxylate\ index} = \boldsymbol{I_{carboxylate\ asymmetric}/I_{C-H\ stretching}} \quad \textbf{(3)}$$

where $I_{carboxylate\ asymmetric}$ and $I_{C\text{-}H\ stretching}$ refer to the peak intensity of carboxylate asymmetric and C-H stretching characteristic peaks, respectively.

One can see from Figure 3 (b) that a considerable decrease in carboxylate index was observed with the incorporation of cotton fibers. Such a feature was anticipated since the cotton fibers themselves did not contain carboxylate groups. Comparing AC5-U and AC5-P, the latter displayed a higher carboxylate index, suggesting that the plasma treatment introduced more carboxylate groups. Based on the aforementioned results, the dye adsorption mechanism was attributed to the electrostatic forces between negative charges (mainly carboxylate groups) and cationic groups in MB dye. Due to the higher amount of anionic groups (COO^-) contributed by the plasma treatment, the MB dye adsorption capacity of AC5-P was relatively higher than AC5-U, albeit with marginal improvement.

Conclusions

In this work, we preliminary investigated the composite hydrogels consisting of alginate calcium and low-quality cotton fibers with/without plasma treatment as dye absorbent for the first time. Plasma treatment not only improved the homogeneity of cotton fibers and promoted the immobilization of cotton fibers within the alginate matrix but also introduced more carboxylate groups and, thereby, the slight enhancement of dye adsorption capacity. Systematical investigations of understanding hydrogel systems and dye adsorption dynamics, as well as potential application exploration, will be considered in the future.

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An Investigation of Fiber-to-Fiber Friction

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Abstract

Based on our observations with various spinning trials, we have noticed that cottons with similar fiber properties may produce different yarn quality. Our hypothesis is that the difference in friction among fibers could be a possible explanation for this observation. Earlier researchers have used only small sample sizes to study fiber friction due to the limitations of the test method. An ASTM standard method (designated as ASTM D2612-99) exists for measuring fiber cohesion. In this study, we have used a similar method to the ASTM standard with some modifications. Our proposed method employed the Mecmesin MutliTest 2.5-dV force tester to pull apart a cotton sliver and measure stress versus strain. We have tested the friction of various types of regular cotton slivers (card, DI, DII, intermediate, and vortex-finished) as well as slivers produced by the Micro Dust and Trash Analyzer (MDTA) slivers from seven commercial bales. MDTA slivers make sample preparation simpler and allow for testing small quantities of fiber (~10 g of lint). Results on seven bales show that differences among cottons vanish as the slivers are processed through more drawing steps. Also, MDTA slivers show some trends that may relate to yarn quality, though the sample size is small. Currently, we are in the process of testing the friction of 300 commercial samples produced in the U.S. using the proposed test method with MDTA slivers to examine the correlation between friction and other fiber properties.

Introduction

As a result of prior spinning tests (not detailed here), we have observed that some cottons with similar fiber properties exhibited quite different yarn qualities that are not explained by slight differences in fiber quality. As such, we hypothesized that it is possible that the friction between fibers could be a contributing factor to the differences that we have observed in the spinning process. Several researchers have studied various methods to measure fiber friction. The fiber fringe method was used by Lord and Bayes (Bayes, 1955), A. Viswanathan (Viswanathan, 1973), and others (Subramaniam et al., 1981). The fiber beard method was used by other researchers to measure inter-fiber friction (El Mogahzy & Broughton, 1993). Besides these, X. L. Cui (Xiaoliang et al., 2002) performed the cohesion test of 36 cotton samples, while A. Sinoimeri (Sinoimeri, 2009) and Y. Zhang (Y. Zhang et al., 2016) developed

a device to measure inter-fiber friction and fiber-to-metal friction, respectively. Following the measurement of inter-fiber friction, some researchers have identified different degrees of correlation between friction and other fiber and yarn properties, though tests have been limited to small quantities of samples—no more than a few dozen.

Currently, there exists an ASTM standard method to measure fiber cohesion, designated as ASTM D2612 – 99: Standard Test Method for Fiber Cohesion in Sliver and Top (Static Tests) (American Society for Testing and Materials (ASTM), 2018). The method we propose to measure cotton fiber friction is very similar to the ASTM test method with a slight modification to be able to test small quantities of fiber. The ASTM method recommends testing card or drawing slivers, which is impossible to do for small qualities of lint, such as with breeder samples. Furthermore, there is very little published research using this ASTM method, particularly on a large set of samples. Finally, the ASTM method itself states that there is no determined mathematical relationship between the cohesion test and yarn quality. As a long-term goal, we are interested in establishing that relationship if one exists.

Materials and Methods

For our initial experiments, we tested various sliver types from seven U.S. commercial bales. These bales were part of a prior spinning test for which we already had fiber quality (HVI and AFIS) as well as rotor, ring, and airjet (vortex) yarn quality (Textechno Statimat DS and USTER Tester 5). As part of the processing of these bales, from each bale, we set aside various types of slivers for friction testing: card, DI, DII, intermediate, and vortex-finished. These sliver types generated as part of yarn processing (card, DI, DII, etc.) require a significant amount of work to produce and, as such, are impractical as a fiber testing material. Therefore, as a more practical sample preparation method, we propose using slivers created with the USTER Micro Dust and Trash Analyzer 3 (MDTA-3). These slivers can be produced with 5 g of raw cotton lint and compressed using the trumpet and calender roller of a carding machine, creating a sliver similar in appearance to a card sliver with far less effort, as shown in Figure 1. Furthermore, slivers can be processed twice through the MDTA potentially improving the distribution of the fibers throughout the sliver. Along with the aforementioned sliver types, we generated MDTA slivers (both 1-pass and 2-pass slivers) for each of the seven cottons. All samples were conditioned at 21±1°C and 65±2% relative humidity for at least 48 hours prior to testing.

(a) (b)

Figure 1. (a) Fluffy cotton sliver removed from an MDTA-3 after testing. (b) Compressing the MDTA sliver using the trumpet and calender roller on a carding machine.

Tests were conducted using a Mecmesin MutliTest 2.5-dV force tester. To prepare the sample, first, a cotton sliver is carefully laid flat on a surface and cut into 9 cm-long sections using scissors. Then, on each end of a 9 cm section, we place a 4 cm × 2.5 cm piece of 100-grit sandpaper folded in half with the sandpaper side facing the fibers to prevent fiber slippage near the clamps (Figure 2(a)). With the two pieces of sandpaper folded in half, they cover 2 cm of each end of the sliver, leaving 5 cm in the middle. The distance between the grips must be such that no single fiber is clamped on both ends. Therefore, we believe 5 cm (1.96 in) should be sufficient. Next, the sliver is put into place on the force tester, with each sandpaper-covered end being fully inserted into its respective jaw grip with no sandpaper visible outside of the jaws of the grips (Figures 2(b) and (c)). The grips are then tightened, and the sample is ready for testing. With the sample sliver in place, there is typically some slack in the sliver, as shown in Figure 2(d). The initial slack is important because it ensures that no force begins to pull the sliver apart before the measurement begins. Throughout the sample preparation and clamping process, special attention was given to maintaining the fiber alignment in the sliver specimens.

Figure 2. Sample preparation and clamping of the specimen to the MultiTest 2.5-dV(u) test system. (a) Clamping the ends of the 9 cm section of sliver with folded sandpaper. (b) Inserting the sliver into the grips of the force tester. (c) Measuring the distance between the grips. (d) View of the sliver before the test begins.

For each test, the force tester outputs the stress versus strain curve. We then extracted three measurements from each stress-strain curve: maximum stress (MaxF), the area under the curve up to MaxF (Work-to-MaxF), and the maximum derivative (MaxD). We conducted the speed tests with a 10 N load cell and chose a pulling speed of 50 mm/min.

Results and Discussions

In our first experiment, we tested card, DI, DII, intermediate, and vortex-finished slivers using the proposed test method. The results showed a clear difference among the different sliver types, with the trend being that more drawing requires less force to pull the slivers apart because the fibers are more parallelized. In Figure 3, we could also see a clear difference among the

different samples with the card sliver, while the vortex-finished slivers (which have been processed through three drawing steps) showed very little difference among the samples. In short, as the samples were processed via drawing, the differences among samples vanished. We added a half twist to the slivers shown in Figure 4 and repeated the tests. Overall, the results of the half-twisted slivers followed the same general trend as the slivers without a twist. However, Figure 5 illustrates that more force was required to pull the sliver apart (as expected), and the variance among replications was significantly higher.

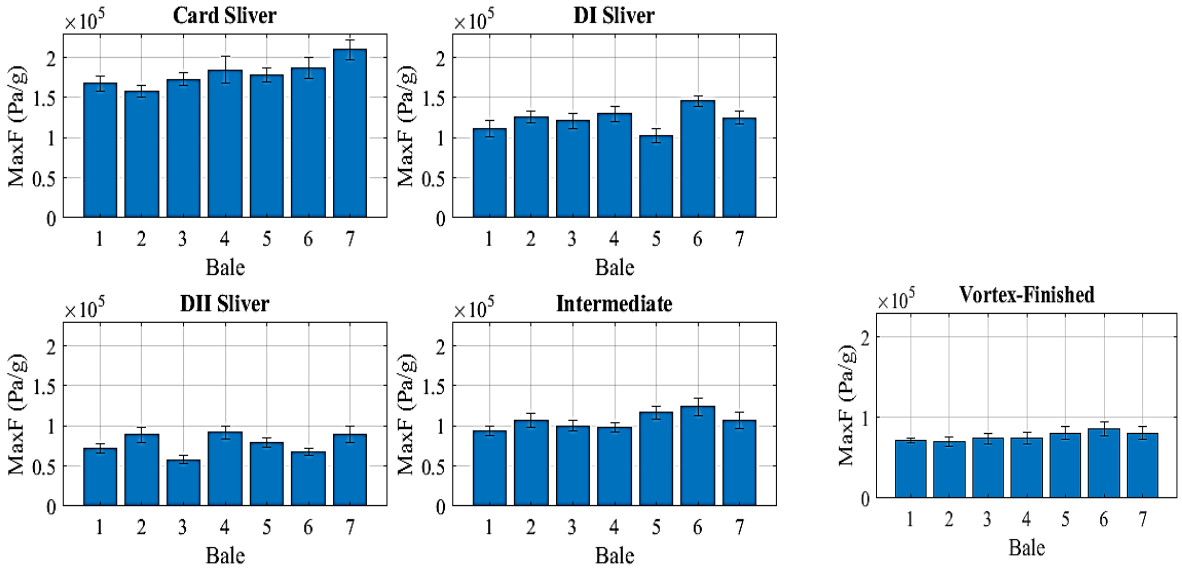

Figure 3. Means and 95% CIs for MaxF among all sliver types and bales. Each bar is the mean of 10 reps, and the error bars represent the 95% CI of the mean. Note that the y-axis is the same scale across all graphs.

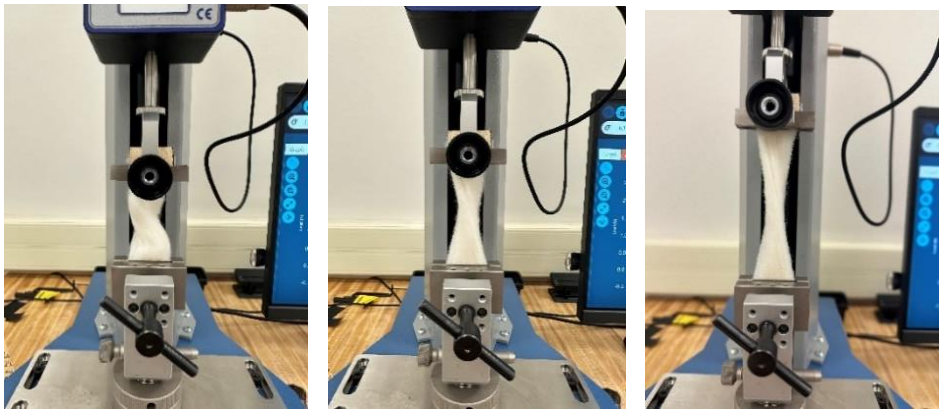

Figure 4. An example of a sliver with a half-twist being tested.

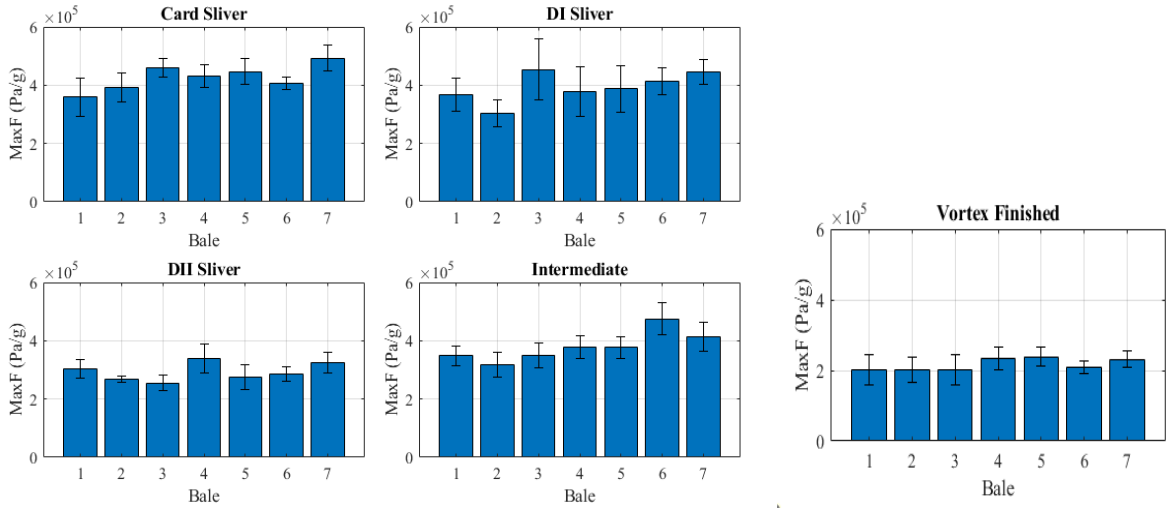

Figure 5. Means and 95% CIs for MaxF among all sliver types and bales with an added twist to the sample.

Finally, Figure 6 shows that the friction test results of the 1-pass MDTA slivers are similar to those of the card sliver (Figure 3), which indicates that perhaps the 1-pass MDTA sliver

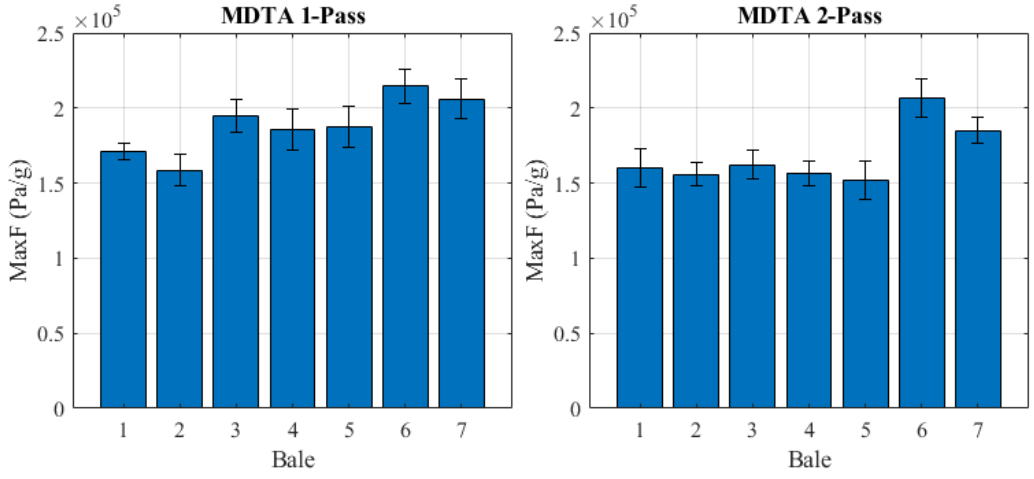

Figure 6. Means and 95% CIs for MaxF among MDTA slivers and bales.

We are currently conducting tests on 300 commercial samples using the proposed test method with MDTA slivers. Previous studies have identified varying degrees of correlation between friction and the properties of both fiber and yarn. However, these studies used relatively small sample sizes. Our study aims to verify these correlations using a larger sample size.

Conclusion

In our study, we used a method similar to the ASTM standard method with some modifications for testing smaller amounts of fiber. Using our proposed method, we tested the friction, or cohesion, of different

types of cotton slivers generated during yarn processing (card, DI, DII, intermediate, and vortex-finished) from seven commercial bales and found that card slivers produced the highest friction value. We also discovered that Fmax increases with an added twist but at the cost of higher variance among replications. Later, to make sample preparation easier, we made MDTA 1-pass and 2-pass slivers from the same seven bales and measured friction. Results showed similar differences among the samples as with card sliver, meaning the MDTA sliver could be a useful alternative for small quantities of fibers. Using the proposed test method with the MDTA slivers, we are currently testing the friction of 300 samples to verify the correlation between friction and fiber properties. We should point out that this type of study on a large number of samples is practically feasible only with the MDTA sample preparation method as it is much simpler, quicker, and cheaper than the other sliver types we tested.

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A new tool for measuring the ribbon width of cotton fibers

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Abstract

Fiber diameter is a crucial factor in textile processing and significantly influences the quality of yarn and fabrics. Various methods, such as cross-sectional image analysis, Cottonscope, and AFIS, are utilized to measure this fundamental parameter in direct or indirect ways. Yet, these methods are indeed time-consuming and impractical for large commercial samples. Recently, the Optical Fiber Diameter Analyzer (OFDA) 4000 was introduced to measure the diameter and length of multiple types of fibers. However, no information is available on the use of this instrument to measure ribbon widths of cellulosic fibers. In this study, the OFDA 4000 was evaluated for measuring the ribbon width of cotton fibers. First, the stability of the measurement was confirmed, ensuring the reliability of the result. Next, a set of 104 samples were used and tested with a standard protocol of 6 replications. The OFDA 4000 ribbon width strongly correlates with the AFIS diameter, explaining 81% of the OFDA's ribbon width variability. These findings could have significant implications in improving efficiency and accuracy in fiber fineness measurements and provide valuable insight into its applicability to natural cellulosic fibers.

Introduction

Fiber diameter plays a key role in textile processing and in determining the quality of yarn and fabric. A smaller fiber diameter enables more fibers to fit within a particular yarn cross-section, eventually contributing to the production of high-quality yarn (Kelly et al., 2015; Delhom et al., 2018). In the case of cotton, fibers tend to collapse and form ribbon-like structures along their

length when they dry naturally (Berkley, 1948). Due to the inherent variability and structural characteristics of cotton, 'fineness' or 'ribbon width' is employed as a practical means of characterizing the diameter of cotton fibers (Delhom et al., 2018; Lord, 1956; Ramey, 1982).

The cross-sectional image analysis, Cottonscope, and Advance Fiber Information System (AFIS) are commonly used tools for evaluating the fineness or diameter of cotton fibers (Kelly et al., 2015; Delhom et al., 2018). Cross-sectional image analysis represents a direct and precise method for measuring the cross-sections of fibers. This approach provides detailed and accurate data regarding the shape and dimensions of fibers, serving as a reference method for calibration indirect measurement methods (Xu & Huang, 1982; Hequet et al., 2006). Several thousand cross-sections of fibers should be taken for each sample to be fairly represented. Preparing cotton samples and processing cross-sectional images can be a time-consuming and labor-intensive process. Both Cottonscope and AFIS are indirect methods for measuring the fineness of cotton. While the Cottonscope uses a microscope and image analysis to measure the ribbon width of cotton fibers from 20,000 fibers snippets distributed in water (Delhom et al., 2018; Kim et al., 2020), AFIS uses an electro-optical sensor to provide both fineness and maturity of 3,000 individualized fibers. AFIS is widely recognized for its ability to provide detailed and reliable data on fiber characteristics, making it an essential resource for assessing cotton fineness and complete fiber quality (Bragg & Shofner, 1993; Kelly & Hequet, 2018). However, these conventional methods are time-consuming, tedious, and not feasible for large samples. As a result, the textile industries have been looking for relatively quick, precise, and reliable fiber quality measurement tools to meet the requirements of high-speed textile machines.

Recently, the Optical Fiber Diameter Analyzer (OFDA) 4000 was introduced to measure the diameter and length of various types of fibers, including wool, cotton, hemp, synthetic fibers, and others (Qi et al., 1994; Brims, 2002; Rafat et al., 2007; van der Sluijs et al., 2021). It is equipped with a digital microscope featuring LED lighting and a CCD video camera, which are essential components of the measuring system. The measurement process begins by cutting a 20 mg clean fiber sample into 2 mm fiber snippets using a guillotine. These fiber snippets are then evenly distributed on a 70 mm glass slide using a spreader machine. Then, the fibers on the slide are imaged by the digital microscope, and the images are analyzed by OFDA software to provide the ribbon width or diameter of the fibers. The OFDA 4000 offers two test modes: slide mode and top/tuft mode. In slide mode, fiber snippets are utilized to measure the diameter of the fibers, and top/tuft mode employs a fiber tuft or sliver to measure both the diameter and length of the fibers. Previous works have demonstrated the OFDA's capabilities in measuring wool fiber diameter (Rafat et al., 2007; Baxter et al., 1992). However, there is limited information available on the use of the OFDA for measuring other types of fibers. We hypothesize that the OFDA could be a potential tool for measuring the

diameter and length of cellulosic fibers. This study was designed to investigate the use of OFDA measuring ribbon-width cotton fibers. The term 'ribbon width' refers to the width of a 2D projection of fiber images.

Materials and methods

This study involved two sets of samples. The first set was used to test the repeatability of the OFDA 4000, while the second set was utilized to assess the ribbon width of the cotton fibers.

Viscose and polyester fibers were used to evaluate the repeatability of the diameter measurement. Each sample was measured using a research protocol of six replications daily for five days. Another set of 104 cotton samples was used to measure the ribbon width. These samples are well-blended carded cottons covering a wide range of fiber properties (Hequet et al., 2006). Each sample was tested with a research protocol of six replications. The data (cross-section, Cottonscope, and AFIS) obtained from past studies on the 104 samples were used to evaluate the efficacy of OFDA ribbon width measurement. Samples were conditioned at 21±1°C and 65±2% relative humidity for two days before testing.

Results and Discussion

Repeatability of diameter measurement

The repeatability of the OFDA 4000 diameter measurement for five days was evaluated. The diameters from the six replications per day were averaged for each sample and compared across the three days. Figure 1 shows the mean diameters of both samples for five days.

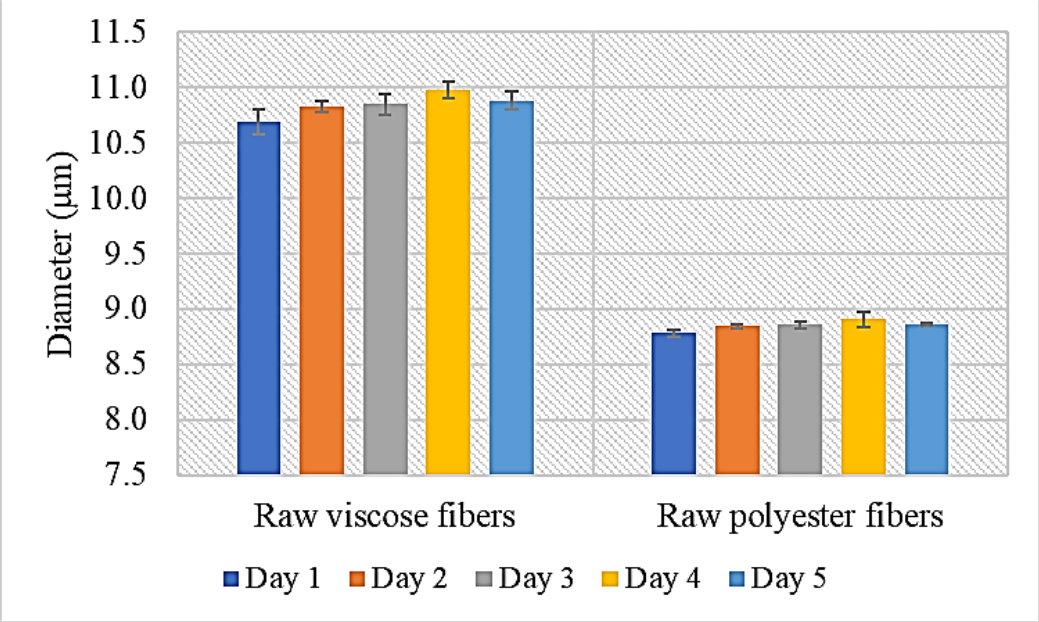

Figure 1. Stability of diameter measurements.

As shown in Figure 1, the diameter measurements of each sample do not show statistically significant differences over time at 95% confidence intervals. In addition, the coefficients of variation (CV%) within and among days were calculated to evaluate the variance of the measurements. In both cases, the CV% was very low and below the acceptable limit (i.e., CV% below 5.0%). In practice, some variation in this measurement can be expected because of the sample preparation and sample slide handling. The obtained results revealed the repeatability of OFDA 4000 diameter measurements.

Cotton fiber ribbon width measurement

After confirming the stability of the OFDA diameter measurements on synthetic fibers, the cotton fiber ribbon width measurement was performed. For each sample, the average diameter measurement of 6 replications was calculated. Then, the OFDA ribbon width was compared with the existing tools that measure similar fiber properties. For this study, the data (cross-sectional image analysis, Cottonscope, and AFIS) from the previous studies on the same set of 104 samples were obtained for comparisons. In the previous study, the relationships between the outer perimeter and standard fineness (H_s) of cotton fibers help in determining the average diameter of fibers in a sample, i.e., AFIS Diameter $=1.2044\times\sqrt{(Hs)}$, in which $H_s=H/M$ (7), where Hs is AFIS standard fineness, H is AFIS fineness, and M is AFIS maturity ratio. Figure 2 provides a graphic overview of the diameter or ribbon width measurement using these four different tools.

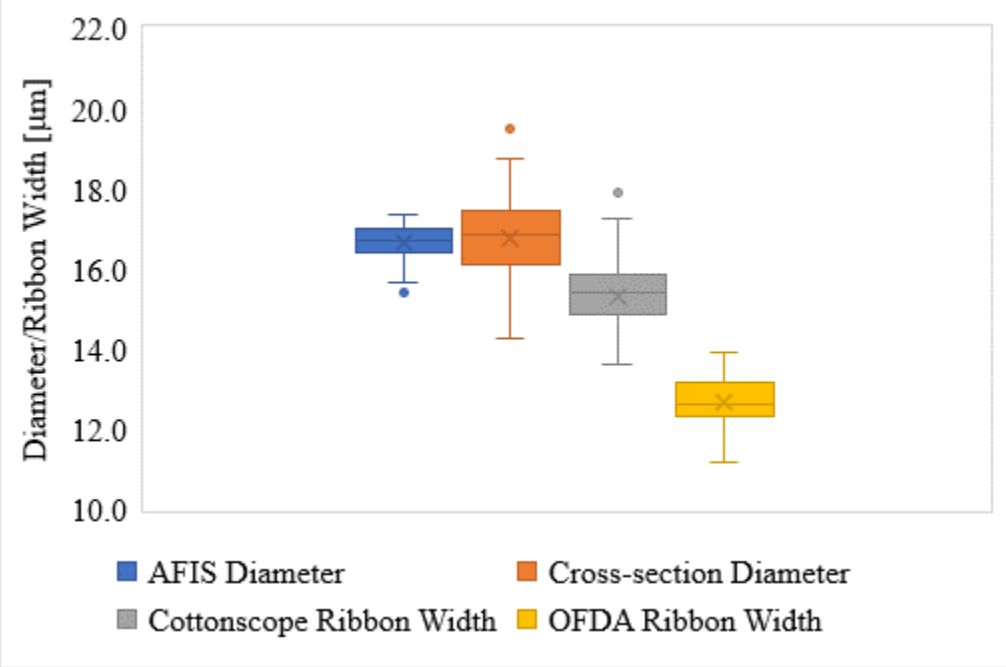

Figure 2. Comparison among tools measuring fiber fineness.

Based on the boxplot in Figure 2, the four tools used to measure cotton fiber diameter in direct or indirect ways demonstrate varying ranges and levels for a set of 104 samples. The cross-sectional diameter may capture the widest range of variations in diameter among the four methods. However, differences in sample preparation and testing procedures may have an impact on the level differences.

The establishment of relationships between the ribbon width measurements obtained by the OFDA and existing tools measuring similar properties, such as the cross-sectional method, Cottonscope, and AFIS, is a critical step in evaluating the efficacy of the OFDA 4000 for measuring cotton fiber ribbon width. Figure 3 illustrates the relationship between OFDA ribbon width and AFIS diameter.

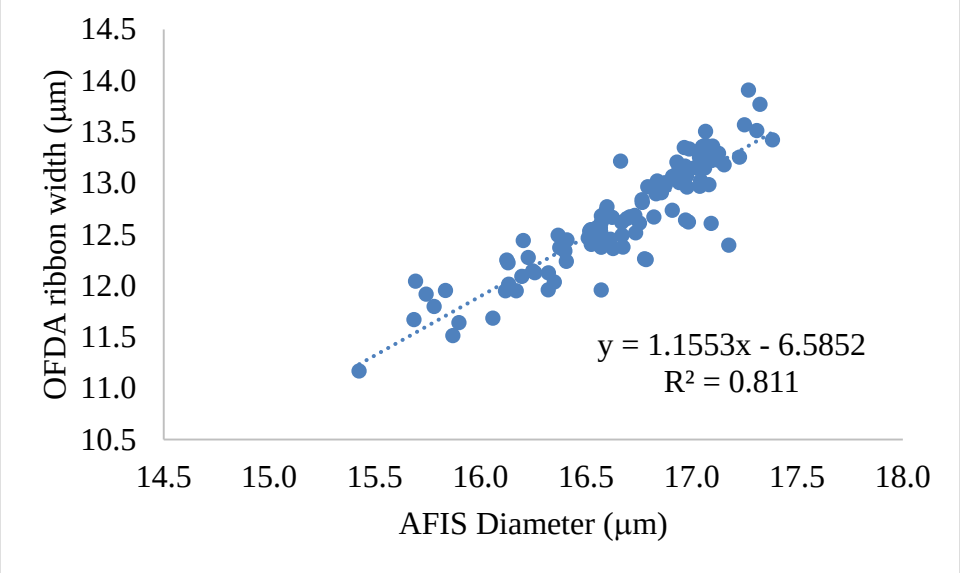

Figure 3. The relationship between OFDA ribbon width and AFIS diameter.

The strong correlation observed between the OFDA ribbon width and AFIS diameter, as depicted in Figure 3, is particularly noteworthy. This finding reveals that 81% of the variation in OFDA ribbon width can be explained by AFIS diameter. Furthermore, this finding suggests that the OFDA is effective in capturing and representing cotton fiber ribbon width in a manner that aligns closely with the AFIS, which is a well-established and widely used tool in the textile industry. However, it is also noted that the OFDA ribbon width measurements do not show good correlations with the Cottonscope (R^2=0.375) or the cross-sectional method (R^2=0.385). The difference in measurement medium in which the tests are performed (i.e., aqueous for the Cottonscope and air for the OFDA) may contribute to the observed poor relationships.

Conclusion

The ribbon width of cotton fibers is an important characteristic, as it can provide an estimate of the fiber's diameter, which is a critical parameter in assessing the fiber quality. The OFDA 4000 is a tool that has been used to measure the diameter of wool fibers and is now being evaluated for its

effectiveness in measuring the ribbon width of cotton fibers. The repeatability of the diameter measurement was confirmed, which is essential for ensuring that the results are reliable. Comparisons were made between the OFDA ribbon widths and data from past studies, including cross-sectional measurements, Cottonscope measurements, and AFIS data, to evaluate the proposed method. There is a strong correlation between the ribbon width measurements obtained by the OFDA and the calculated diameter measurements from the AFIS, with the AFIS diameter explaining 81% of the variability in the OFDA's ribbon width measurements. The poor relationship between the Cottonscope and OFDA is likely related to the medium in which the measurements are performed, i.e., aqueous for the Cottonscope and air for the OFDA. This suggests that the OFDA 4000 can be a valuable tool for measuring cotton ribbon widths, particularly for researchers and cotton development studies. These findings could also have significant implications in improving efficiency and accuracy in fiber fineness measurements and provide valuable insight into its applicability to natural cellulosic fibers.

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Establishing a Small Set of Reference Material for Cotton Fiber Maturity Measurements

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Abstract

This study addresses the critical need for rapid and accurate measurement of cotton fiber maturity and fineness in the textile industry. Recognizing the lack of a standardized method for assessing these qualities, several years ago, a collaboration with Cotton Incorporated led to the creation of a reference set of 104 cotton bales from various origins for maturity assessment through fiber cross-section analysis. The goal was to develop calibration equations for quick-testing instruments like AFIS and Cottonscope. We are now in the second phase of the project, which involves creating a smaller set of calibration cottons (nine bales) for routine instrument calibration, ensuring minimal variability and long-term availability. Preliminary results from the analysis of eight out of nine bales show strong correlations between cross-section and AFIS-reported maturity measurements, with an R^2 value of 0.88. Once testing is complete, the final set of nine reference cottons will serve as a valuable resource for researchers within the cotton industry in the development of new methods for measuring cotton fiber maturity.

Introduction

The impact of fiber maturity on dye absorption is well-established. Additionally, mature and fine fibers enable the production of finer yarn. As such, the qualitative characteristics of cotton fibers, such as maturity and fineness, are also crucial in understanding how fibers break under stress. It is reasonable to hypothesize that immature fibers, which are thin and have a poorly developed secondary cell wall, are fragile and prone to breaking during the various mechanical stresses involved in the transformation of fibers into yarn. This breakage results in the

generation of short fibers and neps, which are entanglements of fibers. An increase in the amount of short fibers leads to defects in the yarn as well as reduced productivity.

In the textile sector, the significance of fiber maturity and fineness cannot be overstated, yet the industry lacks a quick and accurate method for measuring these attributes directly or indirectly. To address the absence of reference material for maturity, a collaboration with Cotton Incorporated led to the creation of a set of reference cottons for maturity assessment via fiber cross-section analysis (Hequet et al., 2006). This collection comprised 104 bales from the two main cotton species primarily sourced from the U.S. with additional selections from Egypt, Uzbekistan, Pakistan, Cameroon, Syria, Benin, and Australia. From each bale, a 30 kg (about 70 pounds) sample was extracted and processed following the International Cotton Calibration Standard Committee's (ICCSC) protocol to create reference material. For each bale, eight sub-samples were analyzed, with at least 500 cross-sections per sub-sample examined using FIAS software (Bugao & Pourdeyhimi, 1994). While image analysis of cotton fiber cross-sections offers a reliable method for assessing maturity and fineness, this slow and tedious process renders it impractical for commercial use. The primary aim behind establishing this reference set of 104 cottons was to develop calibration equations for rapid-testing instruments like AFIS and Cottonscope.

Current Progress

We are now in phase two of this project, i.e., creating a small set of calibration cottons (nine bales) for the day-to-day calibration of instruments. It is important that these standards exhibit minimal variability and have enough material to be used for a decade. The examination of the results obtained on the initial set of 104 cottons allowed us to build a general abacus relating maturity, standard fineness, and micronaire (Figure 1).

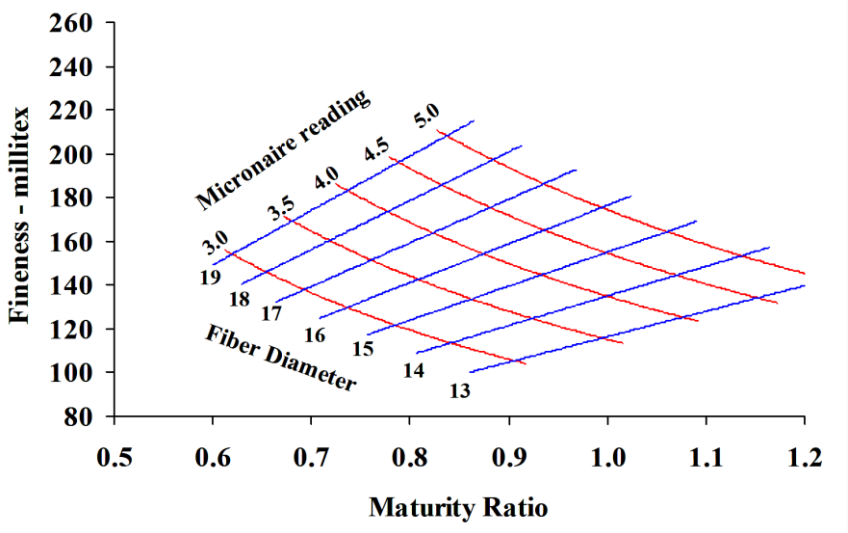

Figure 1. Relationship among maturity, fineness, diameter (equivalent to standard fineness), and micronaire.

From Figure 1, we can easily conclude that to be effective, a calibration for maturity and standard fineness needs to be performed with a set of calibration cottons that do not exhibit a high correlation between maturity and standard fineness. Therefore, a minimum of three cottons (minimum to check for linearity) per level of standard fineness are necessary (Table 1).

Table 1. Candidate standards to be identified.

		Maturity Ratio		
		Low	**Medium**	**High**
Standard Fineness	**Low**	**X**	**X**	**X**
	Medium	**X**	**X**	**X**
	High	**X**	**X**	**X**

At this time, eight bales have been identified. They have been blended, and the card web produced following the ICCSC (International Cotton Calibration Standard Committee). We processed the full bale for each cotton. Sixty samples were taken per bale. Each sample was tested on HVI and AFIS (high accuracy protocols, Figures 2 through 4). For the cross-sections, 60 subsamples are taken from each bale, and a minimum of 500 cross-sections per subsample are tested using the procedure described by Hequet et al. and analyzed using the FIAS software (Hequet et al., 2006). The cross-sections on the first six bales are complete, and testing on the seventh bale began at the end of the first quarter in 2023. The ninth bale will be acquired this year (2024) following the completion of the seventh bale.

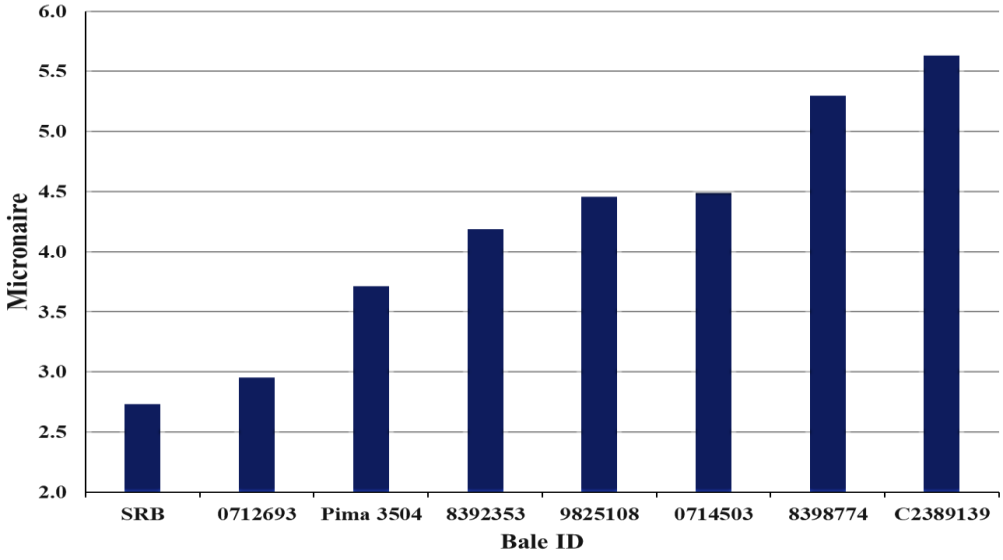

Figure 2. Micronaire of the first eight bales (card web).

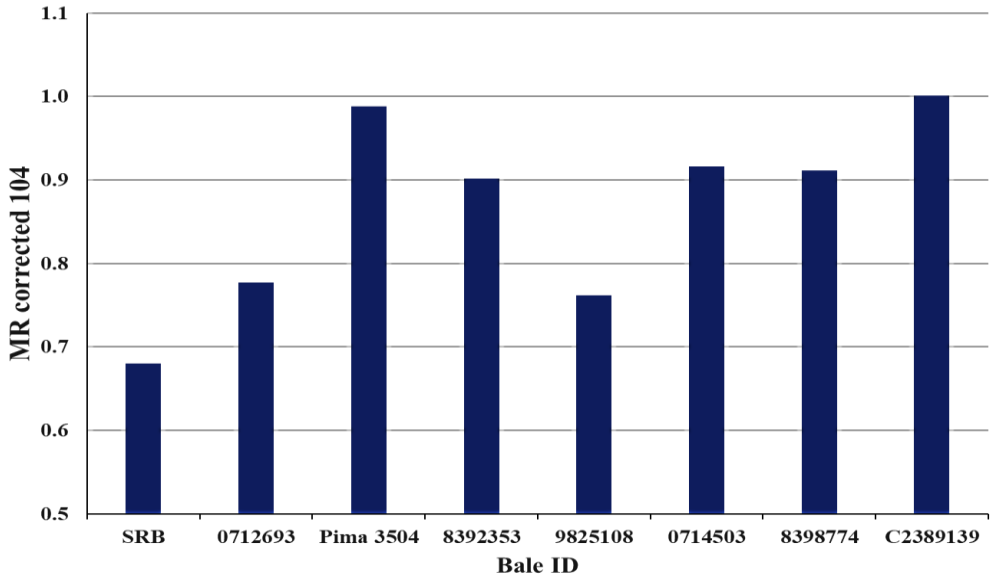

Figure 3. AFIS Maturity Ratio (corrected with the reference values of the 104) of the first eight bales (card web).

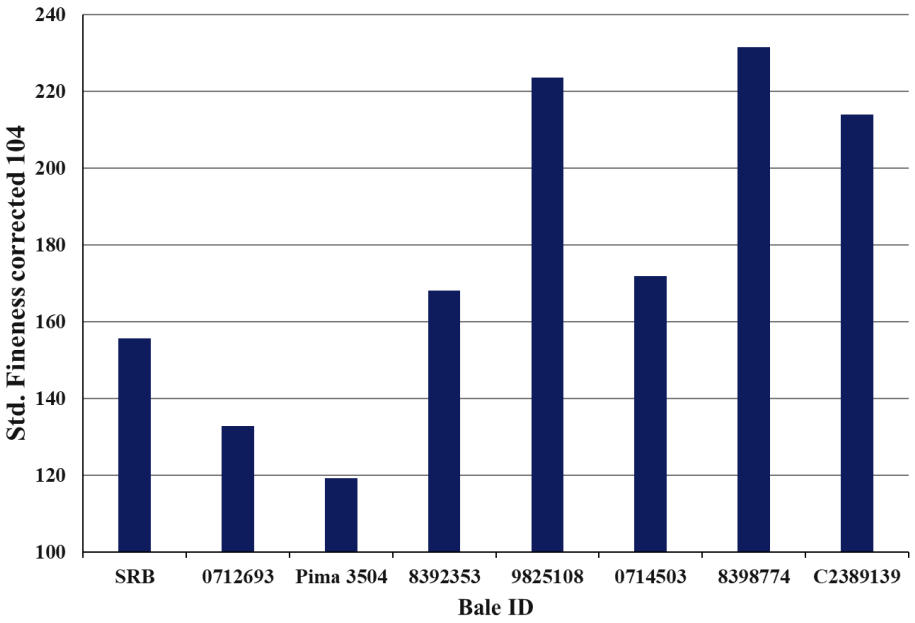

Figure 4. AFIS Standard fineness (corrected with the reference values of the 104) of the first eight bales (card web).

The results obtained are in line with the observations made on the original set of 104 cottons in terms of CVs (among fibers and among replications) as well as in terms of the shapes of the distributions. The average theta values obtained from cross-sections and AFIS are close, R^2=0.881. (Table 2). (Theta from AFIS is calculated as 0.577 × Maturity Ratio as per Pierce and Lord (Peirce & Lord, 1939).) For very low

maturity cottons (SRB and 0721693, for example), AFIS theta tends to be lower than cross-sections theta. This was expected as the AFIS fiber individualizer tends to break more of the less mature fibers. One long immature fiber can be broken into two shorter fragments, while long mature fibers would not break. This results in a lower average theta. These results are very encouraging and better than expected. We would have been satisfied with an R-squared of 0.8.

Table 2. Theta from cross-sections and AFIS (for the first seven bales).

	Cross-sections	AFIS calculated
8392353	0.511	0.520
8398774	0.535	0.526
C2389139	0.581	0.578
9825108	0.489	0.440
Pima 3504	0.558	0.570
SRB	0.459	0.393
0712693*	0.453	0.448
R^2	**0.881**	

*Results after 34 out of 50 subsamples.

The primary purpose of this set of nine reference cottons will be to provide reference material that can assist researchers in the development of fast and reliable methods of measuring cotton fiber maturity. Once the ninth bale has completed testing (sometime in 2026), we can provide small samples of the nine standards along with any necessary testing data to the research community upon request. These standards can then be used to assess the accuracy of newly proposed methods and, ideally, provide a source of calibration material for these new methods for years to come.

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Recent developments in the cotton fiber length measurement using the High Volume Instrument

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Introduction

Over the last several years, the cotton fiber quality research group at the Texas Tech University Fiber and Biopolymer Research Institute (FBRI) has spent a significant amount of time investigating cotton fiber length, particularly as it pertains to length obtained from the USTER High Volume Instrument (HVI). Our central hypothesis has been that more information regarding length is being captured by the HVI than what is currently being reported. This information could be used by breeders for the improvement of the cotton fiber length distribution for new germplasm as well as for spinners seeking better information regarding the spinnability of their raw materials. This article provides a high-level overview of our progress and summarizes several papers published in recent years.

Measurement of cotton fiber length

Precisely characterizing the complete within-sample variation in fiber length is crucial for every sector of the cotton industry. Cotton breeders use the cotton fiber length information along with other fiber quality parameters to develop improved cotton varieties. Textile industries use the fiber length information to purchase the cotton bales for their desired end product. Without proper information, it would be difficult for both groups to achieve their goal. Typically, fiber length is determined through instruments like the HVI or the Advanced Fiber Information System (AFIS). The HVI can provide several fiber quality parameters such as length, strength, micronaire, color, and trash within 30 seconds, which makes it very fast (1,000 samples in classing offices per 8 hours) and relatively cheap (~$3.0 per sample). The AFIS measures the length of 3,000 individual fibers from a sliver prepared with 0.5 gm of raw cotton and provides a complete fiber length distribution along with fiber maturity and fineness (Bragg and Shofner, 1993). However, AFIS is too slow (50 samples per 8 hours) and expensive ($15 to $20 per sample), which makes it impractical for the cotton industry to use it commercially.

HVI fiber length measurement

The HVI is the instrument that is being used to classify every bale of cotton

produced in the U.S. The HVI bases its fiber length measurement on the fibrogram principle, which was originally proposed by Hertel in 1940 (Hertel, 1940). Later, Chu and Riley modified Hertel's theory to account for the sampling assumptions of the HVI fibrosampler (Chu and Riley, 1997).

The HVI combs grab fibers from the fibrosampler to prepare a fiber beard. The fiber beard is then brushed to remove loose fibers and extraneous materials and to make the fibers as straight as possible. Then, the beard is scanned over a red-light beam beginning at 3.81 mm (0.15 in) away from the comb and then moving toward the tip of the fiber beard. A sensor above the beard records the amount of light attenuated by the beard. The maximum amount of light blocked occurs at the base of the beard near the comb, which is considered 100% attenuated light. As the beard is scanned towards the tip of the longest fibers, the amount of light attenuated by the beard decreases as fewer fibers are present. Where no fibers are present, beyond the tip of the longest fiber, the amount of attenuated light is considered to be 0%. The function of distance versus the amount of light attenuated by the beard generates a curve called a fibrogram. From this curve, the HVI reports two length parameters: upper half mean length (UHML) and uniformity index (UI). UI is the ratio of mean length (ML) to the UHML. In 2021, Sayeed et al. demonstrated that instead of measuring the UHML and ML, the current HVI system measures two span lengths from the fibrogram, 1.8% and 7.8%, respectively, and simply refers to these as "UHML" and "ML" (Figure 2) (Sayeed et al., 2021). Sayeed et al. also demonstrated that these two-span lengths are highly correlated as they share 95% of their variation.

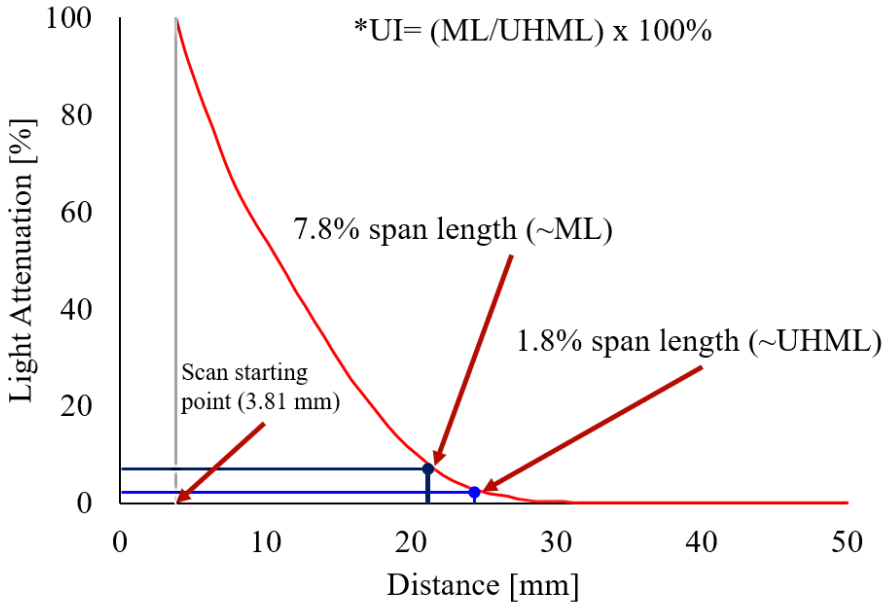

Figure 2: A typical fibrogram showing current HVI length measurement points.

Characterize complete fiber length information using the whole fibrogram curve

After observing that a large portion of the fibrogram curve is unused, Sayeed et al. investigated whether the complete fibrogram curve captures additional fiber length information that is not being captured by the current HVI length measurements, UHML and UI (Sayeed et al., 2021). By extracting new features from the fibrogram curve using principal component analysis (PCA), they demonstrated that the fibrogram holds additional fiber length information that can predict yarn quality better than currently reported HVI length parameters, as well as AFIS mean length by number (Figure 3). Later, they developed a method to calibrate the whole fibrogram as a curve and demonstrated that it is possible to bring fibrogram curves from different HVIs to a similar level by applying a calibration method (Sayeed et al., 2022). This means that the information captured by the whole fibrogram curve could be used across the cotton industry.

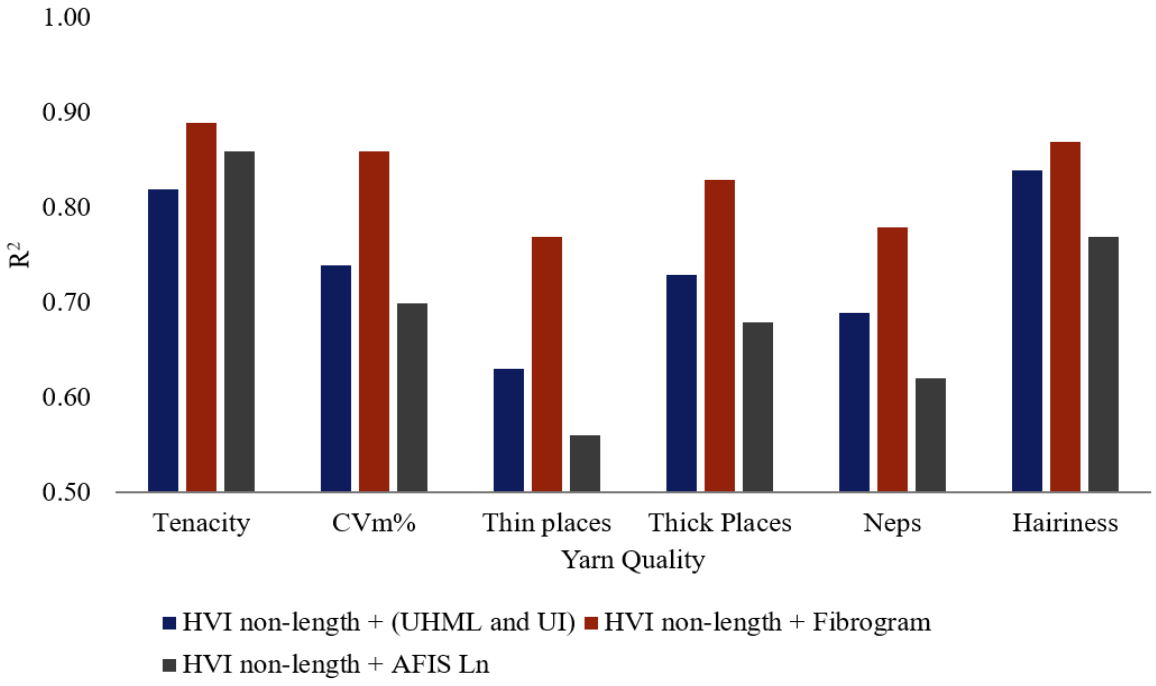

Figure 3: Prediction power of yarn quality parameters by fibrogram fiber length information compared to HVI UHML and UI, as well as AFIS mean length by number.

Using span lengths across the fibrogram

While the approach proposed by Sayeed et al. using PCA to extract features from the fibrogram showed promising results, it is impractical to provide such data (i.e., values calculated from principal components) to breeders and spinners as it lacks an intuitive interpretation. As such, the research group at FBRI began looking for an alternative representation of the

information captured by the fibrogram. Along those lines, Tesema et al. selected ten span lengths across the fibrogram curve (Tesema et al., 2023). The first thing they did was to demonstrate that these ten span lengths were stable over a short-term (one week) and a long-term period (two months). Next, they investigated a method to calibrate the selected span lengths. The results demonstrated that it is possible to bring the selected span length measurements to a similar level across multiple HVIs, which supported the previous results obtained by Sayeed et al. in 2021. Finally, they developed yarn quality prediction models where they used different combinations of the selected span lengths. The results demonstrated that the use of two span lengths, namely the 2.5% and 15% span lengths, can predict yarn quality better than the current HVI-reported measurements. It should be noted that these two span lengths are different from the span lengths currently used by the HVI (1.8% and 7.8%) and are farther apart.

Reconstructing the fiber length distribution from the fibrogram

According to Hertel's fibrogram theory, the fibrogram curve is a type of cumulative distribution function (Hertel, 1940). As stated previously, Chu and Riley later modified Hertel's theory to account for the sampling assumptions of the HVI fibrosampler (Chu and Riley, 1997). In 2023, Turner and his colleagues at the FBRI used the theory provided by Chu and Riley to create an algorithm that reconstructs the fiber length distribution from an HVI fibrogram (Turner et al., 2023). The algorithm was initially validated on synthetic distributions and then examined using the fibrograms from raw cotton as well as man-made fibers, which showed promising results. With the complete fiber length distribution, one can calculate any length parameter of interest, i.e., UHML, ML, or even short fiber content. Currently, AFIS is the fastest instrument capable of measuring the complete fiber length distribution of a cotton sample. However, it is possible that this new method could provide information similar to the AFIS at the speed of the HVI, though more investigation is needed.

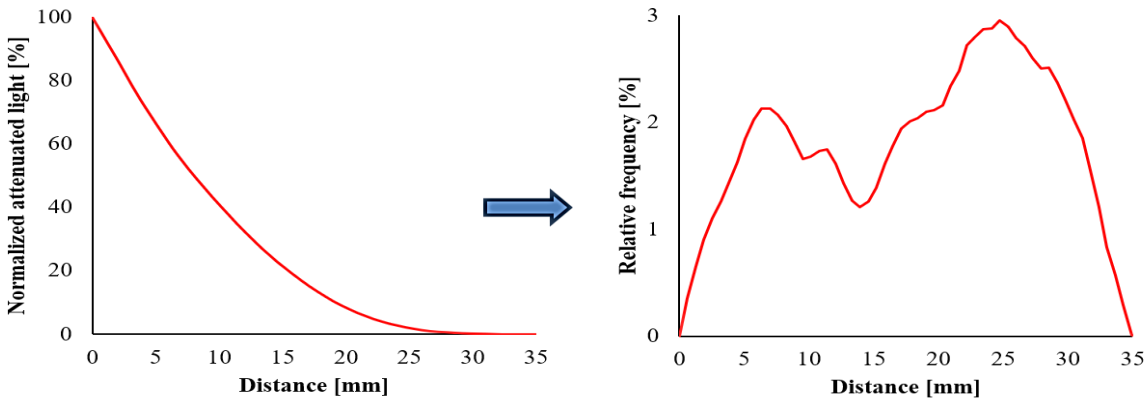

Figure 4: Reconstructed fiber length distribution from the fibrogram curve.

Developing calibration methods for the new length parameters

With the advent of new length parameters based on Turner's algorithm, there now exists a need to develop calibration methods so that the measurements can be compared across different HVIs as well as different labs. In 2023, we started the work on developing calibration methods for several fiber length parameters calculated from the reconstructed fiber length distribution. Currently, we have selected the following length parameters for the calibration study: UHML, ML, upper quartile length (UQL), lower half mean length (LHML), lower quartile length (LQL), and short fiber content (SFC).

The first challenge was to find appropriate cotton bales that could be used as calibration standards for all the selected length parameters. To that end, we analyzed the fibrogram data obtained from approximately 200 commercial cotton bales. From those bales, we identified three bales representing ranges in values for each length parameter that are comparable to the U.S. cotton crop (Table 1).

Table 1: Fiber length parameters for the selected candidate calibration bales. (Note that parameters derived from the reconstructed distribution from the fibrogram are suffixed with the letter "f" to avoid confusion with similarly-named parameters from HVI or AFIS.)

Bale ID	UHMLf (mm)	MLf (mm)	UQLf (mm)	LQLf (mm)	LHMLf (mm)	SFCf (%)
Bale 1	29.21	21.34	28.45	14.73	14.22	21.77
Bale 2	22.61	15.49	24.38	7.87	9.14	39.23
Bale 3	25.40	18.03	24.64	10.41	10.92	31.21

After purchasing the three candidate calibration bales, we investigated the within-bale variability. While the within-bale variability was confirmed to be sufficiently low to serve as calibration standards, we chose to blend each bale to reduce the variability further. Though the blending slightly reduced the variability, we concluded that blending would not be necessary in the future so long as the initial investigation of within-bale variability showed good results. In 2024, the fiber quality research group at the FBRI is collaborating with the Cotton Incorporated fiber testing lab in Cary, North Carolina, U.S.A., to perform a round test to develop the calibration methods for each of the parameters.

Concluding Remarks

In general, the fiber quality research group at the FBRI works towards developing or improving cotton fiber phenotype measurements. As fiber length is one of the most crucial physical properties, we have worked on improving the fiber length measurement using additional information obtained from the HVI fibrogram. The

most recent developments of providing a complete fiber length distribution from an HVI fibrogram could serve as a tool to enhance germplasm selection for cotton breeders as they seek to improve fiber length distribution. Currently, we are working to establish calibration methods for these new parameters. While we believe that these new length parameters offer new information about the quality of the length distribution of a sample as it pertains to spinnability, that still needs to be validated. Our future goal is to perform large-scale spinning tests (at least 100 commercial bales) to confirm that hypothesis, but it will take time. Until then, we are working with breeders to determine whether selecting new lines using these new parameters versus the traditional HVI length measurements can improve overall length distribution in future generations.

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