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IN SEARCH OF THE MYSTIC COTTON FIBER MATURITY: A VIEW FROM THE MICROSCOPE

Wilton R. Goynes
Southern Regional Research Center
New Orleans, LA

Abstract

Development of simple, accurate, economical methods for determining fiber maturity has been an ongoing quest because fiber properties such as strength, reactivity and processability depend on fiber secondary wall thickness or maturity. Because cotton is a natural product and all harvested fibers do not reach maximum wall thickness simultaneously, no single method seems to provide definitive measurements. This work compares results of maturity measurements by nine processes currently being used or that are in development, in order to determine which methods best characterize the sample maturity. The samples were evaluated by a differential dyeing procedure to visually show gross maturity differences, and cross sections of the dyed fibers were observed using light microscopy to relate differences found in the measured data. Neps in all samples were examined using scanning electron microscopy. Two Fibermax varieties grown in three geographical areas were examined. Both bale samples and hand-combed samples were dyed and sectioned. Obvious differences in sample maturity became evident by dyeing. These differences were not obvious in all instrumental methods. One problem in comparing data from the various measuring systems is that they do not all report maturity in the same values. Maturity comparisons within each system were somewhat consistent, however, some systems failed to recognize extremely immature samples as well as other systems did. Dyeing and microscopic observations indicated that differences between measured values from different instruments could be due to 1) sampling, 2) choice of measuring bale or cleaned samples, 3) method of cleaning, or 4) consistent selection of area along length of fiber to be measured. The work is continuing using a broader set of samples with additional measured data.

Introduction

Measurement of cotton fiber maturity has been important to the cotton industry throughout the history of commercial cotton textiles. Because maturity, a measurement of completion of secondary wall development, varies with growing conditions and locations, it is never constant even within the same variety. Additionally, fibers on the same plant and even on the same seed do not develop at the same time or at the same rate. However, maturity, wall thickness, affects properties such as strength, reactivity, and processability. It follows then that maturity affects value, and the great interest in measuring maturity relates to the economic relationship. Although there has always been great interest in measuring cotton fiber maturity, the current interest in developing an accurate, fast, and simple maturity measurement probably stems from factors that include changing harvest methods that remove both mature and immature bolls from the plant at the same time, competition in import-export markets that require standardized testing with documentation of fiber quality for improved sales, farmers' desires for maximum value from their crop which increases with improved properties, and advances in instrument technologies that should allow better measurements.

In textile laboratories worldwide there are ongoing attempts to develop the ultimate method for determining fiber maturity that is quick, accurate, and inexpensive. This is evidenced by the number of current presentations and publications related to maturity. They explore possibilities either by direct methods, based on microscopic measurements of actual fiber dimensions, or indirect methods based on measurement or observation of some related secondary characteristics such as air permeability, dyeing properties, birefringence, or optical transmission. All methods produce some bias, and unfortunately, do not give the same results when applied to the same samples. Analysis of why results are different, and which are more reliable, would be helpful in understanding these methods. Such analyses require understanding of cotton fiber structures that affect wall thickness as related to maturity. Some maturity measurement methods do not distinguish between wall thickness, a growth characteristic and fineness/coarseness, a varietal property. To understand wall thickness it is helpful to understand development of the secondary fiber wall. Figure 1 is a cotton fiber montage, computer compiled, of electron micrographs of various layers of the cotton fiber. The figure does not represent a realistic view of the cotton structure since all layer micrographs are not shown at the same magnification. Layer images are used that allow viewing of the microstructures of the layers from the surface to the lumen. Thickness of the portion of the fiber that is shown as secondary wall is the determining factor in fiber maturity

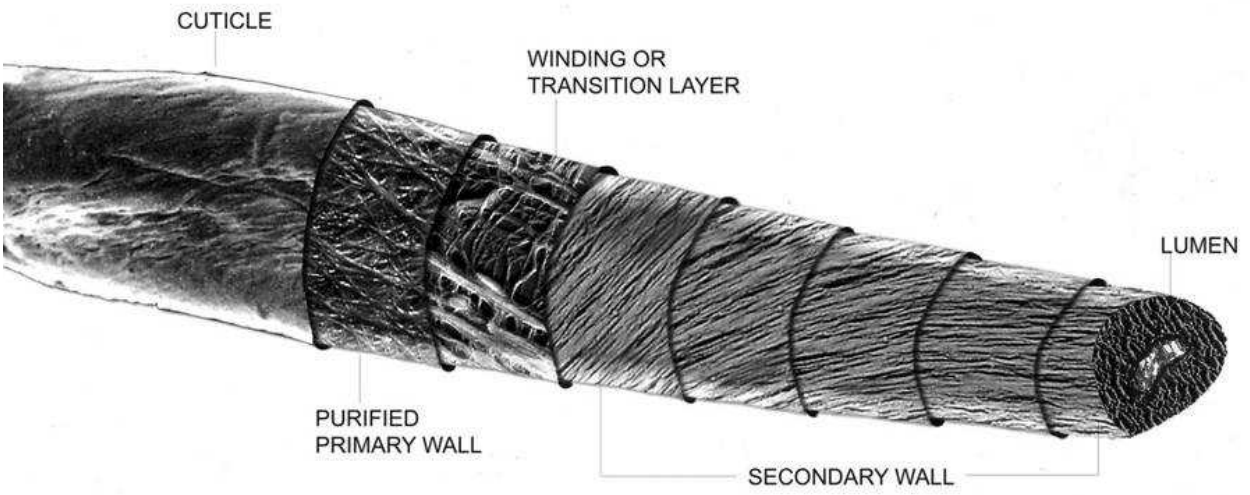

Figure 1. Cotton Fiber Montage

Cross sections of a bundle of cotton fibers that have been dyed to differentiate mature and immature fibers are shown in figure 2 (Goldthwait, 1947). Green fibers are considered to be immature and red fibers are mature. Variations in secondary wall thickness within both the green and red fiber sections are obvious. Some green fibers are more mature (have thicker walls) than others. The question in maturity measurements is "at what point in wall development does the fiber become mature rather than immature". How is maturity defined so that a particular wall thickness is considered to be mature?

Peeters states that "maturity is based on the ratio between wall thickness and the cross-sectional dimensions of the cell such as diameter and cross-sectional area." (Peeters, 1986) Raes and Verschraege define maturity as the "average relative wall thickness expressed as the mean of two times the wall thickness divided by the fiber diameter deduced from the real perimeter." (Raes, 1981) Thibodeaux says that "maturity refers to the degree of development or thickening of the fiber cell wall relative to the perimeter or effective diameter of the fiber." (Thibodeaux, 2000) These definitions provide an idea of what maturity is, but do not indicate a direct method for measuring it. They also do not indicate at what point in fiber wall development the fiber is considered to be mature.

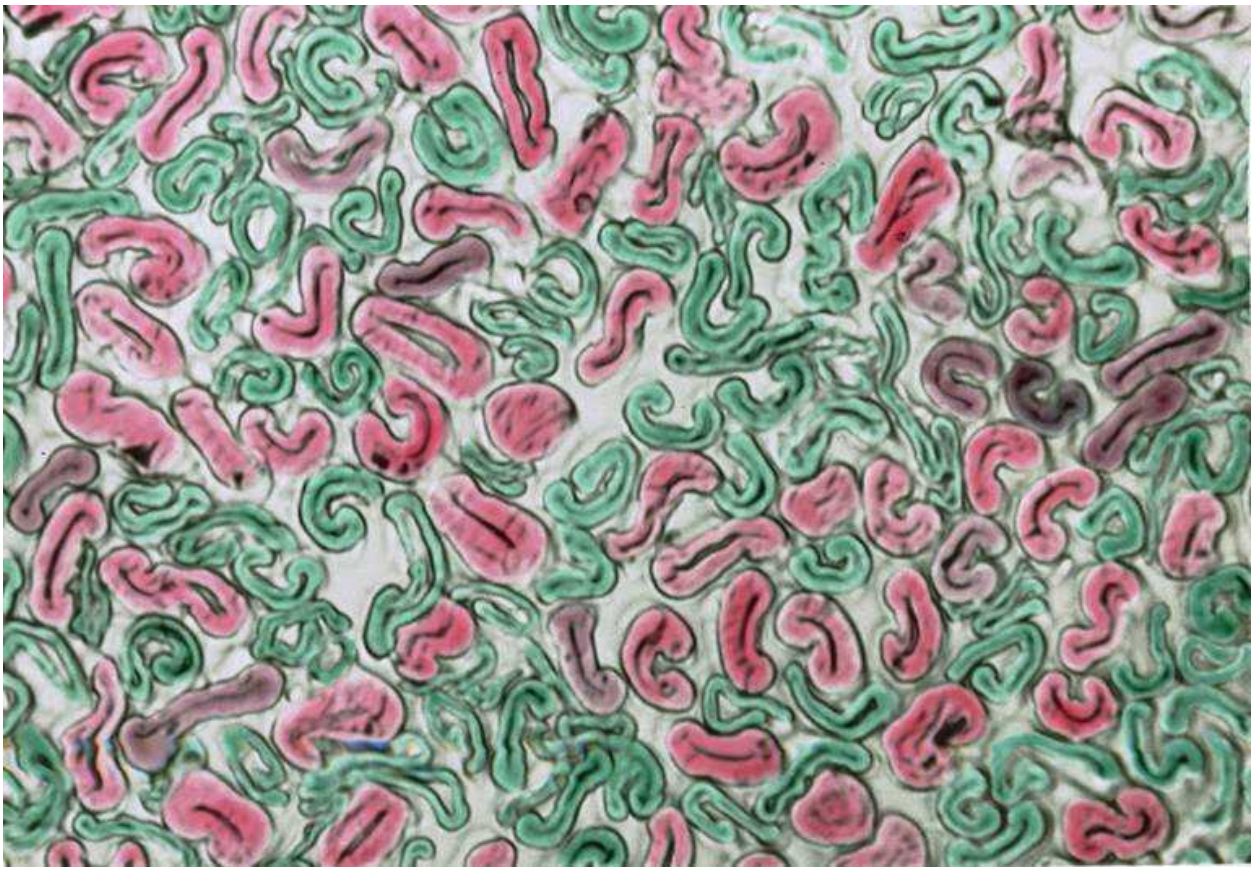

Figure 2. Cross sections (light micrograph) of cotton fibers dyed to show mature and immature fibers

ASTM Method D1442-75 says that a fiber is mature when wall thickness is greater than 25% of the ribbon width measured on NaOH swelled fibers using microscopic observations. This definition has several limitations, in that maturity methods currently being developed do not use swelled fibers, and measurement of wall thickness of such fibers in a light microscope means that an average ribbon width has to be determined on a convoluted fiber. The definition indicated that when half of the secondary wall thickness from primary wall to lumen is developed the fiber is mature.

Materials and Methods

In attempts to better illustrate the concept of secondary wall development to maturity and to determine which of the currently developed methods best show this, we have compared results of maturity measurements of a series of fibers grown in several USA locations (Rousselle). Data from recently developed methods such as Image Analysis and NIR were compared with results from The Micromat Fineness/Maturity Tester (FMT) and HVI micronaire (Montalvo et al., 2004). These data are shown in Table I. While some of the data shows generally good correlation, there are inconsistencies that need to be resolved. In our attempt to determine reasons for these inconsistencies we developed maturity information as directly measured by differential dyeing and light and scanning electron microscopy. In the dyeing study, Fibermax-832 and Fibermax-966 grown in Georgia, Mississippi, and Texas were examined to compare fiber maturity in the same variety grown in different locations. While other fibers in this broad general study of fiber characterization were also examined microscopically, only the two Fibermax varieties are compared here.

Table I.

Maturity Data Table

ID	State	Mic[3]	Mic[4]	Micron[1]	Theta[1]	Theta[2]	Mat Ratio[4]	400-2500 nm[5]	1100-2500 nm[5]	Mat Ratio[6]
		HVI	FMT	AFIS V2	AFIS V2	IA	FMT	NIR Mat	NIR Mat	AFIS Pro
FM832	GA	4.03	3.97	3.718	0.471	0.48	0.79	0.787	0.806	0.92
FM832	MS	3.97	3.89	3.605	0.460	0.59	0.81	0.821	0.806	0.90
FM832	TX	2.89	2.79	3.042	0.415	0.44	0.65	0.647	0.642	0.85
FM966	GA	4.34	4.28	3.940	0.473	0.48	0.82	0.813	0.827	0.92
FM966	MS	4.53	4.49	4.253	0.493	0.50	0.85	0.855	0.849	0.91
FM966	TX	3.19	3.18	3.279	0.432	0.53	0.70	0.687	0.690	0.87

1 AFIS V2	G Davidonis/K Pusateri/G Richard
2 Image Analysis	D Thibodeaux/ J Moraitis
3 HVI	D McAllister/ L Cui
4 FMT	J Montalvo/ T Von Hoven
5 NIR	J Montalvo/ T Von Hoven
6 AFIS Pro	J Campbell/ K Blakes

Data in this table were provided by various investigators as indicated in the figure.

For microscopic examinations, samples were dyed using the differential dye technique of Goldthwait as prescribed in ASTM D 1464-90. This procedure requires that the samples be boiled in the dye mix, rinsed, then dipped into boiling water briefly. Theoretically both dyes penetrate cotton fibers. The green dye, a larger molecule than the red, is less easily removed from the fibers than the red dye. Red dye is differentially removed from the fibers with thinner walls. Thus, thin-walled immature fibers remain mainly green, and the thicker-walled mature fibers retain more of the red dye and appear red. This difference in color can be seen in the bulk fibers, especially when there are bundles of adhering immature fibers distributed through the sample. The blend of red and green colors give the dyed samples a deep burgundy color, but areas of red, green, and even undyed fibers can be found. In this study, samples directly from the bale were used to compare types of maturity, as shown by dyeing, that are present in the harvested fibers. Because of the imprecise nature of dye procedures, it is essential that all samples to be compared be dyed in the same bath. After dyeing, samples were taken from the dyed fibers and bundle cross sections were made using the Hardy hand sectioning device (Hardy, 1935). In addition, undyed clumps or dark neps were removed from the samples, mounted on sample stubs for the scanning electron microscope (SEM) and examined using scanning electron microscopy. Both bale cotton and hand-combed samples from these cottons were dyed and examined.

Results and Discussion

Examination of maturity data in Table I indicates the type problems involved in comparing maturity data from various measurement systems. It is obvious that all systems do not report results in the same terms. HVI and FMT report Micronaire. FMT also reports maturity ratio, as does AFIS Pro. NIR data is reported as maturity measured at two wave length ranges. AFIS V2 reports Micron-AFIS, and well as Theta, and Image Analysis (IA) reports Theta. While it is possible to inter-relate these data, correlations are not readily apparent without a thorough knowledge of what the values represent. It should be possible, however, even with casual observations to compare trends of higher or lower maturity within each system as compared to the trends in others. Comparison of the two varieties that were grown in Georgia, Mississippi and Texas showed different maturities, and most data generated from these samples indicate the Texas sample to be much less mature. These trends are apparent in most of the data. However, image analysis Theta values and AFIS Pro maturity ratios do not follow this trend. Data from the other systems shown in the table indicate lower values for both Texas varieties, but it is not possible to correlate exact ratings of the samples since variations in relationships between the different systems is not known. The two systems, Image Analysis and AFIS Pro, failed to rate the samples in the same order as the other systems. A more direct evaluation of system parameters, such as methods of sample preparation and selection may indicate reasons for these differences.

Bale Samples

The differential dye test of Goldthwait provides a visual method to evaluate quantities of mature and immature dyed fibers in a bulk sample. When these samples were examined after dyeing, differences in maturity were obvious. Both varieties grown in Texas were much greener than the Georgia and Mississippi-grown samples. What is more interesting is that even though red and green fibers were blended throughout much of the samples, all samples had bundles of green fibers that have remained separate. This is even apparent in the most mature samples. In addition, all samples had small, undyed clumps and neps. The green fiber bundles and undyed white clumps have an effect on maturity measurements. Some of these, especially the undyed clumps, become white specks on dyed fabrics

When these dyed cottons were randomly sampled, and cross sections were cut, micrographs showed distributions of green and red fibers, as well as fiber wall thicknesses. Georgia and Mississippi samples had more red than green cross sections, but even in these more mature samples there were bundles of unseparated green fibers that can affect both maturity measurements and yarn and fabric quality.

Trash and undyed neps that were separated from the dyed bundles were studied using the scanning electron microscope. All samples, even those that had the most mature fibers, contained these types of defects. Typical micrographs in figure 3 show their structures. The micrograph at lower magnification on the left shows an unidentifiable mass. Higher magnification as on the right micrograph shown this clump to be composed of extremely thin-walled fibers bound together.

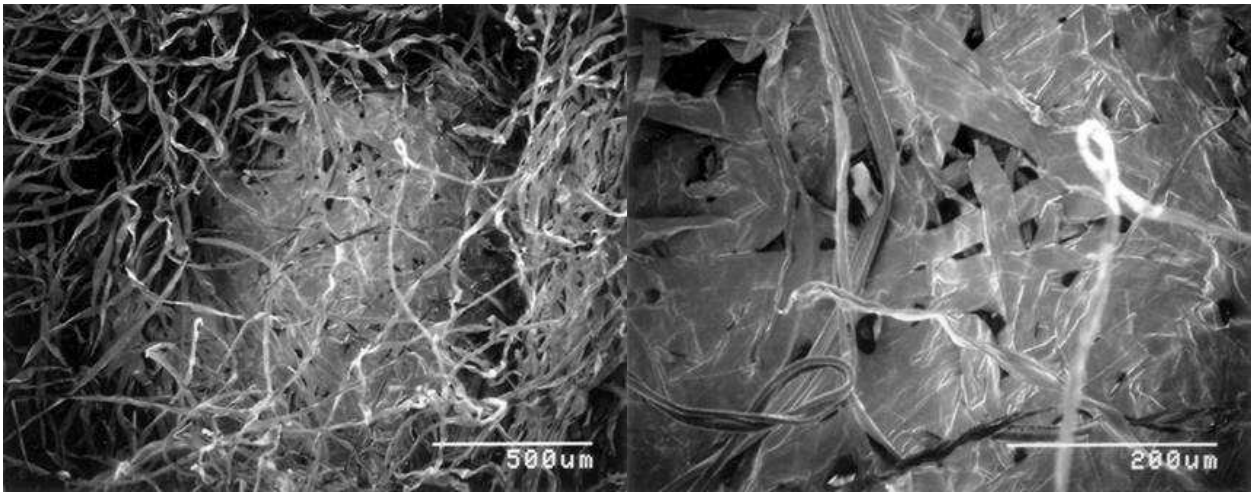

Figure 3. Scanning electron micrographs showing an unidentified clump entangled in fibers on the left, and at higher magnification on the right the components of the clump are seen to be thin-walled fibers

Combed Samples

Because bale samples were used in the first dyeing experiment we next attempted to determine what effect cleaning the samples before dyeing would have on the results. Unfortunately all methods for measurement of maturity do not use the same methods for cleaning samples. For this test we hand combed the samples using a single-line clamp comb, with the goal of leaving trash and immature fiber clumps in one bundle and the more developed individual fibers in the other. These separated samples were weighed and then dyed using the differential dye test.

Combing removed most large neps and undeveloped fiber bundles. It also blended the fibers. Because the process removed the most undeveloped or least mature fibers, it would theoretically improve maturity measurements more in the least mature samples (those with more waste) than in the more mature samples. Weight measurements of the hand carded samples showed the least mature Texas-grown fibers to leave behind the most waste. In maturity measurements, the amount of these immature fibers removed may affect the sample maturity rating. This indicates that sample preparation before measurement is probably of prime importance in standardizing various methods.

Conclusions

We should now return to the question: What is being measured when the various maturity-related processes are used? Some methods measure air permeability, some methods measure wall thickness as related to diameter. How does fiber structure affect these types of measurements? In airflow measurements, bundles of extremely immature fibers act as a single unit, not as individual fibers. How do these bundles of thin-walled fibers with no separation between them affect the airflow measurement?

In making cross sectional measurements, definitions of terms such as diameter are important. Because of the collapsed structure of the fiber cross section, the mature fiber cross section is generally bean-shaped. This means that the fiber does not have a single diameter measurement. Assumptions in calculations of diameter may not take into effect the structural anomalies that occur that compress some areas of the fiber wall more than others, producing not only an elliptical fiber section, but an extremely elongated lumen wall. Thick and thin-walled geometry is extremely different.

Many currently-proposed maturity methods use wall thickness averages of a statistically determined number of fibers to provide a maturity-related number. This averaging is logical for the numbers obtained in the measurements, but does not represent a true picture of the wall thickness of the fibers in the sample. It is not possible to average thin-walled fibers with thick-walled fibers and have a product with properties of an average wall fiber. Properties of the sample depend on the numbers of individual thin and thick walled fibers in the sample. Looking at fiber wall structures as shown in figure 2, it appears that a fiber sample would be best characterized as an array of fiber maturities determined in the sample. Cotton samples with fewer thin-walled samples and more thick-walled fibers are more likely to provide products with better properties. While it is possible to average perimeter and area numbers to determine a presumed maturity, actual wall thicknesses of individual fibers cannot be “averaged” to a common, presumed maturity. In an array of fiber cross sections, it is important to know how many are actually considered mature, and how many are extremely immature. An average value does not necessarily represent a real wall thickness.

Another structural aspect to consider is the position along the fiber length that the measurement is taken. In general, sections should be made near the center of the fiber, since the fiber tapers at the tip end, and is thicker at the base end. Measurements made in these regions are not representative.

Finally, the dyeing studies have shown that the amount of sample preparation before measurement greatly affects apparent maturity of the sample. In the reality of the cotton commercial program, it should be determined whether maturity measurements at the bale or after cleaning are of greatest importance. While measurement at the bale can affect bale price, cleaning can improve maturity measurements. From this study, it appears that samples of low maturity and high nep-count may be improved to a greater extent than samples of higher quality. However, cleaning also reduces the usable weight of quality fibers in a bale. This may indicate that measurement of cleaned samples is a more realistic value for maturity. If cleaned samples are to be used, a standard method of cleaning should be determined since all currently proposed methods do not clean in the same way or to the same extent. Type and amount of cleaning will affect maturity measurements.

There appears to be value in many of the current systems being considered for cotton fiber maturity measurements. However, it would be profitable to the industry for some standards to be considered for all methods being developed so that results can be compared and are an actual reflection of the bio-physical structure of the fibers.

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