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ANALYSIS OF COTTON FIBER CROSS SECTIONS

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Abstract

Our goal was to create a set of reference cottons for maturity measurements. To achieve this we selected 104 cotton bales representing the two principal cultivated species. The vast majority of the bales originated in the U.S.A., but some foreign-grown cotton bales were also selected (Egypt, Uzbekistan, Pakistan, Cameroon, Syria, Benin, and Australia). A representative sample of approximately 30 kg (70 pounds) was taken from each bale. Each sample was homogenized according to the protocol used by the ICCSC (International Cotton Calibration Standard Committee) to produce reference cottons. Eight sub-samples per bale were taken and a minimum of 500 cross sections per sub-sample was analyzed. The average values of fiber perimeter and fiber maturity obtained for the 104 bales revealed that very wide ranges of variation were obtained. Therefore, this population of bales constitutes a good base for the calibration of the indirect measurement instruments for maturity and fineness.

Introduction

The effect of maturity on the dye uptake is well known (that is the basis of the Goldthwait test, 1947). Similarly, we know that fine and mature fibers make it possible to spin a finer yarn. But maturity and fineness of cotton fibers are also essential qualitative characteristics if one wants to better understand the facility of rupture of fibers when they are subjected to stress. It is intuitively obvious to hypothesize that immature fibers (having a thin, poorly developed secondary wall) will be fragile. Thus, they are likely to break during multiple mechanical stresses involved in transforming the fibers from the field to the yarn. These generate short fibers and neps (entanglement of fibers). An increase in the short fiber content will result in yarn defects and decreased productivity.

In spite of the importance of maturity and fineness for the textile industry, there is not a direct or indirect measurement method that is both fast and reliable. The lack of standards of reference for maturity has made it impossible to calibrate the existing instruments (air flow instruments with double compression, Advanced Fiber Information System - Maturity module). Even the dominant qualitative criterion for fineness/maturity, the micronaire, cannot be further refined without a current reference method.

The definitional reference for the measurement of maturity and fiber perimeter is the microscopic analysis of cotton fibers cross sections. Lord (1956) established on 100 cottons the relationship between micronaire and the product maturity x fineness. It should be noted that maturity was determined by the BSM method (British Standard Method, Pierce and Lord, 1939) on swollen fibers (sodium hydroxide treatment) and the gravimetric fineness by the "cut and weigh" method; i.e., forming a parallel fiber bundle, cutting a 1-cm section in the median portion, weighing the cut fibers, and counting the number of fibers (Pierce and Lord, 1939). Both of these methods introduce bias into the measurements. The BSM method swells the fibers with sodium hydroxide and the degree of swelling is not independent of the quantity of cellulose. The "cut and weigh" method is not independent of fiber length because only fibers longer than 1 cm are evaluated.

Given that the reference cottons created by Lord are no longer available, it becomes necessary to create a new set of reference cottons. In doing this, it is critical that the intra-standard variability be as low as possible and that the quantity of reference materials be large enough to meet the needs of the international scientific community for at least 10 years.

Materials and methods

Bale selection and sampling

We selected 104 cotton bales representing the two principal cultivated species (Gossypium hirsutum and Gossypium barbadense, C. W. Smith and J. T. Cothren, 1999). The vast majority of the bales originating in the U.S.A., but some foreign-grown cotton bales were also selected (Egypt, Uzbekistan, Pakistan, Cameroon, Syria, Benin, and Australia). The bales were opened and ten samples per bale were taken. Each sample was tested using a HVI Uster

900A. For each bale a total of 40 micronaire tests and 100 length and tenacity tests were done. This allowed a determination that the intra-bale variability was acceptable and that we had a wide range of micronaire, length, and tenacity.

A representative sample of approximately 30 kg (70 pounds) was taken from each bale (Figure 1). Each sample was homogenized according to the protocol used by the ICCSC (International Cotton Calibration Standard Committee) to produce reference cottons. From the card web produced, 20 samples were taken. Samples 1 to 5 were re-sampled (8 pinches per sample). This new sample was delicately mixed manually then 2 fibrograph combs were formed. We chose to sample with the fibrosampler because this method, unlike the BSM method, is not length biased (Chu and Riley, 1997). This procedure was repeated for samples 6-10, 11-15 and 16-20. A sample was then taken from each of the 8 combs produced (Figure 2). For each comb, a minimum of 500 cross sections was analyzed. When the CV% of the averages between combs was higher than 2%, eight other combs were produced and 500 additional cross sections (per comb) were analyzed. The original CV% was confirmed in almost every case.

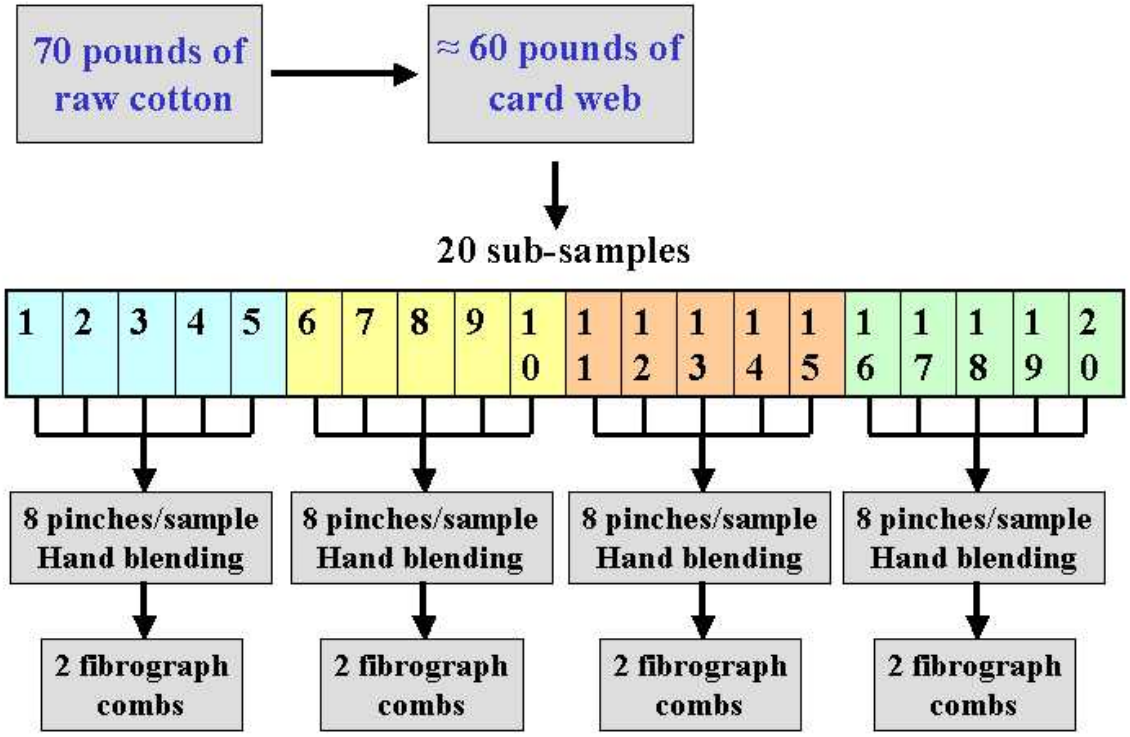

Figure 1. Sampling procedure

Figure 2. Fiber sample produced with the Fibrosampler.

Realization of the cross-sections

The method used to embed the sample is a modified version of the methacrylate embedding method developed by Boylston of the USDA-SRRC New Orleans (1995). The methacrylate polymer holds the cotton fibers until they can be glued to a slide for observation and then the methacrylate polymer is dissolved in methyl ethyl ketone (MEK).

The methacrylate polymer is a mix of 60 parts methyl methacrylate, 40 parts butyl methacrylate and 2 parts benzoyl peroxide. This solution is prepared and partially polymerized (heated to 75^{o} C for about 40 minutes) into what is called the MBM Stock. A second solution of "pre-catalyst" is prepared consisting of 18 parts of methyl methacrylate and 2 parts of benzoyl peroxide. Both of these solutions are refrigerated.

To embed the cotton fibers, 5 ml of the MBM Stock and 1ml of the pre-catalyst are mixed, then the fibers are suspended from a fine wire and dipped into the solution (Figure 3). The wire is then drawn up a Teflon tube (1.6 mm internal diameter and 0.8 mm wall thickness) with the lower end of the tube below the surface of the solution. The tube is stoppered with a toothpick at the bottom and filled with solution from the top, to eliminate air, then stoppered with another toothpick at the top. Finally, the sample-filled tube is placed in the UV chamber (Ultraviolet Crosslinker, from UVP Inc., modified to hold 14 lights of 302 nm) for 20 to 30 minutes.

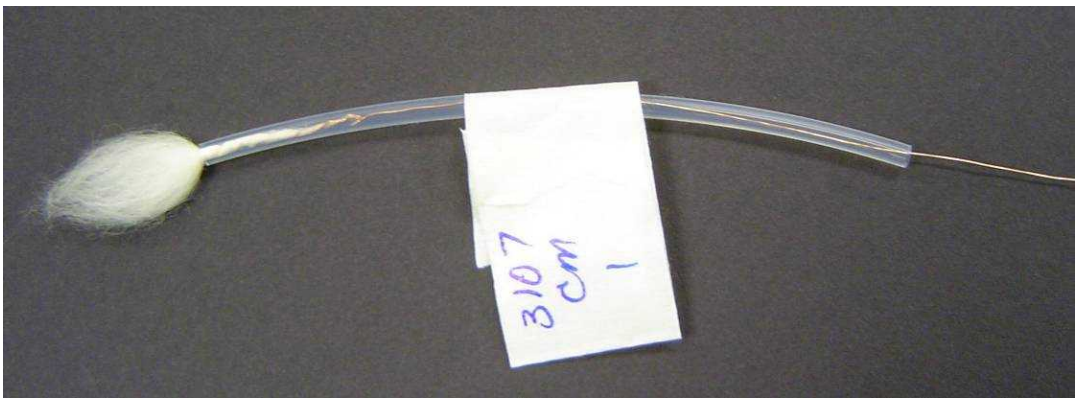

Figure 3. Fiber sample being inserted in the Teflon tubing.

After polymerization, the Teflon tube is removed. The sample (approximately 25.4 mm length) is inserted in a second Teflon tube (3.2 mm internal diameter and 0.8 mm wall thickness). The lower end of the tube is closed with a cork stopper (0000) and filled with solution. The top is then closed with the same type of stopper. Then, the sample is placed in a UV chamber to polymerize the solution (which takes another 20 to 30 minutes). The tube is

then removed. The sample is placed in a third Teflon tube (8 mm internal diameter and 1.6 mm wall thickness). The lower end of the tube is closed with a cork stopper (0) and filled with solution. The top is closed with the same type of stopper. This tube is placed in a UV chamber to polymerize (another 20 to 30 minutes), after which the Teflon tube is removed. This process produces a sample with a diameter that fits well into the sample holder of the microtome (MT 990). Before mounting the sample on the microtome, approximately 6 to 12 mm are cut with a razor blade. The glass knife of the microtome is then used to obtain a perfectly plane surface. Finally the diamond knife is used to make the cross sections (1 micron thick), which are to be observed with the microscope.

Glass slides for microscopic observation are coated with albumin glue, then dried prior to sectioning the samples. The glue is made of equal quantities of glycerin and egg white intimately mixed. The slides are coated with a thin layer of this preparation. Two water drops are deposited on a slide and 1 section is placed on each water drop. The slide is placed on a glass rod support in a Petri dish above chloroform for 3 seconds. This gives the thin polymer time to smooth and allows the cross sections to scatter slightly. This allows a better separation of the fibers, thereby facilitating the microscopic observation. The slide is then placed on a hotplate (very briefly and at low temperature) to eliminate water and to fix the cross sections in the glue. The slide is then washed with the MEK (methyl ethyl ketone) for approximately 4 hours, to eliminate the methacrylate polymer and leave the cross sections glued in place on the slide.

After the slides are prepared the images are viewed with the microscope (40x objective) and captured in the computer using a Hitachi 3CCD Camera Model HVC-20 with a Coreco Oculus TCX Frame Grabber. Finally the images are analyzed (Figure 4) by the FIAS software developed by Bugao Xu of UT Austin (Xu and Pourdeyhimi, 1994).

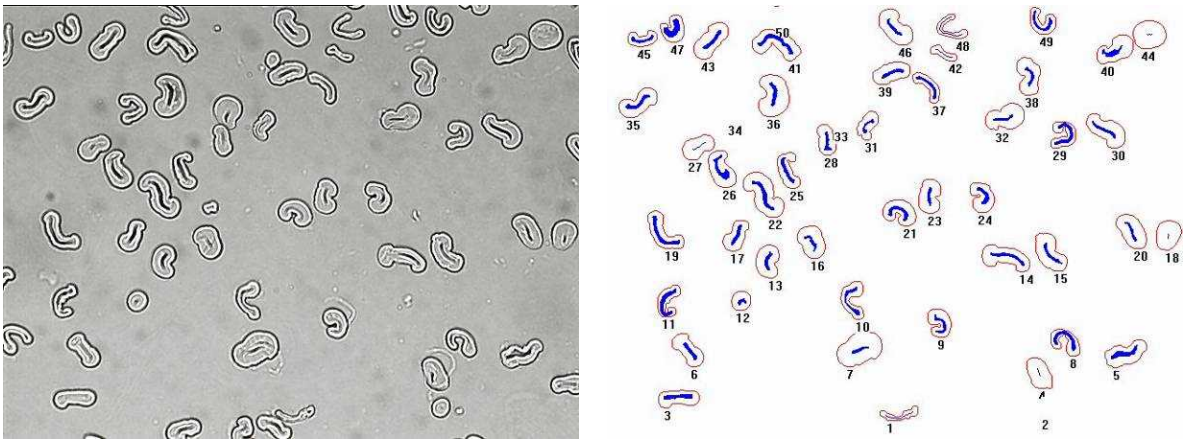

Figure 4. Cross-section analysis with the Fias software.

Results and Discussion

The averages obtained for the 104 bales (Figure 5) show that, for the measured parameters, the ranges of variation obtained are very broad. Therefore, this sample of bales constitutes a good base for the calibration of the indirect measurement instruments for maturity and fineness.

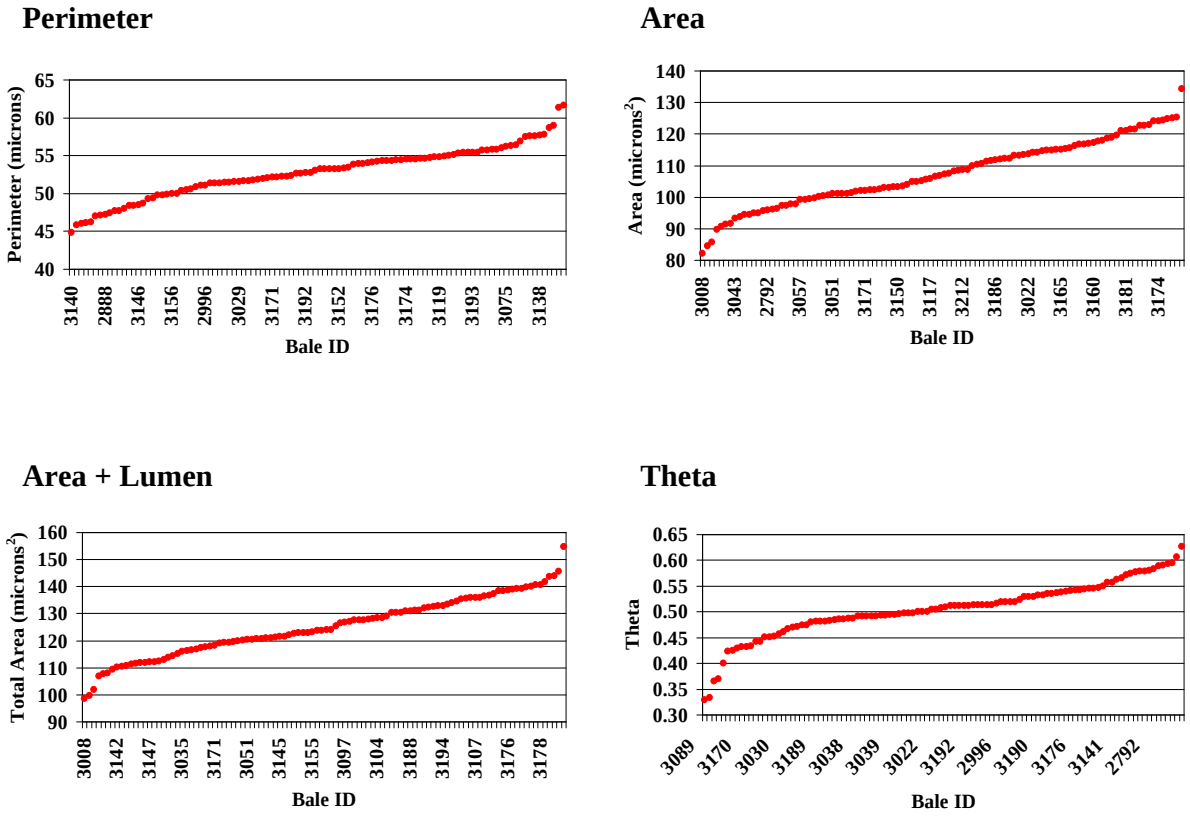

Figure 5. Bale averages for the 104 reference cottons.

Table 1 shows the expected confidence intervals for 8 and 16 replications of 500 fiber cross sections.

Table 1. Expected confidence intervals for 8 and 16 replications of 500 fiber cross sections.

	8 replications of 500 sections	16 replications of 500 sections
Perimeter	± 0.78 µ (± 1.5 %)	± 0.55 µ (± 1.0 %)
Area	± 3.2 μ^2 (± 3.0 %)	± 2.2 μ^2 (± 2.1 %)
Theta	± 0.009 (± 1.8 %)	± 0.006 (± 1.3%)

Conclusions

We now have 104 reference cottons, which will enable us to calibrate double compression testers (Lintronics, CSIRO) and the AFIS (Uster). The calibration of the AFIS will probably be a difficult task. While calibration could be done on the average values, the main advantage of an apparatus such as the AFIS is to have access to the fiber distributions. This implies the necessity of calibrating the distributions, as well as the averages.

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