

TEXTILE TECHNOLOGY

Cotton Fiber Chemical Differences and Their Effect on Friction Behavior: A Comparison of Two Crop Years in the ATMI/ARS Leading Cultivars Study

Gary R. Gamble

ABSTRACT

A comparison of two crop years from the USDA-ATMI Leading Cultivars Study indicated a substantial increase in processing difficulties in CY 2002 compared with CY 2001, but HVI properties were unable to adequately predict these differences. With the goal of complementing HVI with alternative measurements in order to better predict processing performance, this research attempted to determine whether the observed differences in processing between years are attributable to fiber chemical factors. Chemical and physical measurements were performed on 46 cotton (*Gossypium hirsutum* L.) samples with a range in genetic diversities and from different growing locations from two consecutive crop years. Samples from the two crop years exhibited marked differences in moisture and electrolyte content as a result of different environmental conditions subsequent to boll opening and prior to harvest. Infrared spectroscopy was unable to distinguish differences in crystallinity indices within or between the two crop years. Differences in moisture content appear to be related to differences in hygroscopic surface salts and sugars. Micronaire-normalized frictional measurements indicate that fiber friction increases moisture and electrolyte content decreases, indicating that moisture in conjunction with surface salt content affects the surface characteristics of the fiber, possibly through an anti-electrostatic effect.

One aspect of the USDA-ATMI Leading Cultivars Study being conducted at the Cotton Quality Research Station is to ascertain the effect of environmental factors on cotton fiber processing performance. In a comparison of the 2001 and 2002 crop years (CY 2001 and CY 2002, respectively), CY 2002 cottons in general were more difficult to

process than CY 2001 cottons, but that traditional HVI measurements gave little indication of potential differences in processing (D.D. McAlister III, personal communication, 2004). CY 2002 cottons, regardless of growing location, experienced higher rainfall amounts in the field subsequent to boll opening and prior to harvest than CY 2001 cottons. Since the greater difficulty in processing these cottons were not adequately predicted using HVI measurements, this research was begun in an attempt to determine whether any chemical factors may be indicative of or responsible for the observed processing differences.

Several chemical factors, including surface electrolytes, sugars, and pectins, have been correlated with the ability to process the fiber as measured by draft force and Rotorring friction measurements (Gamble, 2004a). In addition, surface wax did not play a significant role in fiber processing when present in naturally occurring amounts. The high rainfall experienced by CY 2002 may have led to a decrease in soluble surface components, which in turn led to the observed processing differences. This study examined the differences in surface electrolyte constituents among cotton samples from CY 2001 and CY 2002, and subsequently attempted to relate any observed differences in electrolyte constituents with the ability to process the fiber as determined by friction measurements performed on the Rotorring (Ghosh et al., 1992).

Fiber moisture content is another chemical factor potentially related to processing. Previous research has shown that moisture content is non-linearly correlated to micronaire values for the CY 2001 cottons of the Leading Cultivars Study (Gamble, 2004b). One implication of this correlation between micronaire and moisture content is that cellulose may undergo progressive structural changes as the fiber matures, leading to changes in moisture content and consequently other fiber properties, such as cellulose crystallinity, that are not currently measured on HVI instrumentation but have an impact upon fiber processing. The potential differences in cellulose crystallinity between cotton samples may have a direct bearing on

processing performance due to resultant differences in the degree of fiber convolution. Increases in fiber convolution have resulted in concomitant increases in fiber friction (Basu et al., 1978; Hussain et al., 1998). A previous study showed that cellulose subjected to wet-dry cycling exhibits decreasing moisture content (Kongdee et al., 2004). This wet-dry cycling process is similar to conditions experienced by CY 2002 cottons due to extended periods of rainfall. Additional research (Mihriyan et al., 2004) demonstrated that increasing cellulose crystallinity is accompanied by decreasing moisture content for fiber samples conditioned at 65% relative humidity. The possibility exists that cellulose crystallinity is a function of cotton fiber maturity. The fact that CY 2002 was subjected to wet-dry cycles suggests the possibility that moisture content may consequently be affected by possible changes in cellulose structure. In addition to examining surface chemical components, this study examined the differences in moisture content among the samples from CY 2001 and CY 2002 and attempted to determine whether any moisture differences are accompanied by changes in cellulose crystallinity, as measured by Fourier Transform Infrared Spectroscopy, and in the ability to process the fiber, as determined by friction measurements performed on the Rotorring.

MATERIALS AND METHODS

Cotton samples. For this study, a total of 21 upland cotton (*Gossypium hirsutum* L.) samples from CY 2001 with a range of genetic diversities and micronaires from different growing regions were analyzed (Table 1). Twenty-five samples from CY 2002,

some of which are of the same cultivars and locations as CY 2001, were similarly analyzed (Table 2). CY 2001 and CY 2002 experienced substantially different environmental conditions subsequent to boll opening and prior to harvest. For the three growing regions (Texas High Plains, Georgia, and Mississippi Delta), harvest normally takes place from mid-October to the first or second week of November. October rainfall for the three growing regions for CY 2001 and CY 2002 are shown in Table 3 (NOAA-NWS, 2005). CY 2001 did not experience any unusual conditions during this period, while CY 2002 experienced unusually high rainfall at each of the three growing locations.

Moisture determination. Duplicate moisture determinations on the 46 cotton samples were performed according to standard test methods (ASTM, 2001).

Micronaire. Duplicate measurements of micronaire were performed on each sample by high volume instrumentation (HVI) according to standard test methods (ASTM, 1997).

Conductivity measurements. Cotton samples were extracted using 20 ml deionized water per gram of cotton. Three replicates were performed for each cotton sample. Each sample was agitated with a glass rod in order to promote wetting of the cotton surface. The resultant wetted sample was allowed to sit for 15 min before being wrung out. The resulting extract was then subjected to conductivity measurements. Conductivity measurements, reported in microsiemens per centimeter ($\mu\text{S}^{-1} \text{cm}^{-1}$), were performed on a Myron L Company Model EP conductivity meter (Carlsbad, CA).

Modification of surface electrolyte content. Pre-washed samples of cotton cultivar FiberMax 958 (Bayer Cropsciences; Research Triangle Park, NC) from Mississippi (FM958M) were immersed in 0.0M, 0.1M, 0.2M, 0.3M, 0.4M, and 0.5M sodium acetate (SigmaChemical Co.; St. Louis, MO). No wetting agent was used to effect moisture absorption, instead the samples were repeatedly agitated manually to remove air and allow wetting of the cotton surface by the immersion solution. Subsequent to thorough wetting, each sample was squeezed manually to remove most of the retained solution. Typically, the resultant cotton took up twice its own weight of the solution following this process. The sample was opened by hand and completely dried in a forced draft oven at 105°C for 2 hr, after which the sample was reconditioned at 65% relative humidity and 21°C for at least 8 hr prior to subsequent testing.

Table 1. October rainfall in 2001 and 2002 for the three growing regions

Location	Rainfall (mm)	
	2001	2002
Texas High Plains		
Lubbock, TX	0.5	133.4
Amarillo, TX	1.3	85.1
Georgia		
Macon, GA	3.1	122.2
Cordele, GA	25.2	114.3
Mississippi Delta		
Tupelo, MS	115.3	159.5
Memphis, TN	175.3	210.3

Table 2. Fiber properties of 21 cotton samples from CY 2001 distinguished according to genetic cultivar and growing location

Cultivar (location) ^z	Micronaire ($\mu\text{g in}^{-1}$)	RotorRing fiber-fiber friction (J)	Moisture fraction (w/w)	Conductivity ($\mu\text{S cm}^{-1}$)	Wax fraction (w/w) x1000
FM832(T)	2.89 n	21998 a	0.0720 a	1025 a	6.45 b
PM2800(T)	3.44 j	18646 cd	0.0688 e-g	963 b	5.53 cd
PM2200(T)	3.38 j	18416 cd	0.0693 d-f	967 b	5.73 cd
FM819(T)	3.15 l	19573 c	0.0707 bc	900 c	6.01 b-d
FM989(T)	3.05 m	19576 c	0.0713 a-c	1000 ab	7.08 a
FM958(T)	3.24 k	18835 cd	0.0701 cd	990 ab	5.89 b-d
FM966(T)	3.20 kl	18234 cd	0.0698 c-e	980 ab	6.02 bc
PM2326(T)	3.78 i	15072 fg	0.0692 d-f	990 ab	4.54 e
DP491(G)	4.06 g	17641 de	0.0680 g-i	692 de	3.83 f
PHY355(G)	4.76 c	15092 fg	0.0670 i	650 ef	4.91 e
FM966(G)	4.35 e	17919 d	0.0677 hi	642 ef	4.77 e
DPPearl(G)	4.28 f	17611 de	0.0675 hi	625 fg	5.70 cd
FM832(G)	4.04 g	19568 c	0.0673 hi	625 fg	4.40 ef
SG747(G)	4.75 c	14603 gh	0.0672 hi	742 d	4.32 ef
DPPearl(M)	4.55 d	16289 ef	0.0675 hi	500 i	3.84 f
PHY355(M)	4.80 c	14374 gh	0.0683 f-h	717 d	4.41 ef
FM832(M)	3.97 h	21683 b	0.0670 i	350 k	4.15 ef
DP491(M)	4.05 g	19529 c	0.0683 f-h	408 j	5.65 cd
FM966(M)	4.53 d	17680 de	0.0678 g-i	550 hi	3.75 f
SG747(M)	4.98 b	14424 gh	0.0670 i	583 gh	3.07 g
PM1218(M)	5.56 a	13372 h	0.0693 d-f	600 f-h	2.89 g

^z Cultivar: DP = Deltapine, PM = PayMaster, SG = SureGrow (Delta Pine and Land Co.; Scott, MS) FM = Fibermax (Bayer Cropsciences, Research Triangle Park, NC), PHY = Phytogen (Phytogen Seed Co., Indianapolis, IN). Location: T = Texas High Plains, G = Georgia, M = Mississippi Delta.

^y Means within a column followed by the same letter are not significantly different according to Duncan's multiple range test ($P \leq 0.05$).

Wax content. Wax content, reported as the fraction (w/w) present on the conditioned fiber, was determined by Soxhlet extraction. Six replicates of 2.5 grams each were performed for each of the 46 cotton samples. Extractions were performed using trichloroethylene as the solvent. The resultant solutions were evaporated to dryness at 105°C, which left a wax residue that was subsequently weighed.

Fiber friction. Four replicates for each of the 46 cotton samples were conditioned at 65% relative humidity and 21°C prior to evaluation for fiber-fiber friction according to published procedures (Ghosh et al., 1992) using the Rotorring 580 (Spinlab; Knoxville, TN). Slivers were approximately 14.70 g/m and results are reported in joules (J).

Mid-infrared spectroscopy. Six infrared spectra for each cotton sample were obtained using a DuraSamplIR (SensIR Technologies; Danbury, CT) attenuated total reflectance (ATR) device in an Excalibur FTS 3000 (Digilab; Randolph, MA) Fourier transform infrared (FTIR) bench. Mats of conditioned cotton fiber were placed on the surface of the diamond ATR crystal and pressure applied to insure full contact of the fibers with the diamond surface. Spectra were obtained at a resolution of 4 cm^{-1} over 7111 scan points, covering the range of 4000 to 600 cm^{-1} . No apodization or baseline correction functions were applied. The peak centered at 1427 cm^{-1} was integrated between 1492 cm^{-1} and 1392 cm^{-1} , and the peak centered at 901 cm^{-1} was integrated between 917 cm^{-1} and 863 cm^{-1} .

Table 3. Fiber properties of 25 cotton samples from CY 2002 distinguished according to genetic cultivar and growing location

Cultivar (location) ^z	Micronaire ($\mu\text{g in}^{-1}$)	RotorRing fiber-fiber friction (J)	Moisture fraction (w/w)	Conductivity ($\mu\text{S cm}^{-1}$)	Wax fraction (w/w) x1000
FM832(T)	3.29 n	34093 a-c	0.0674 ef	550 a	6.09 b
PM2200(T)	3.85 j	31209 b-f	0.0677 de	417 b	5.78 bc
FM819(T)	3.56 m	31941 b-e	0.0720 b	500 ab	5.85 b
FM989RR(T)	3.53 m	30538 b-g	0.0665 e-g	485 ab	5.49 c
FM989B/R(T)	3.30 n	32231 b-e	0.0700 c	550 a	5.97 b
FM958(T)	3.71 l	28512 e-h	0.0745 a	510 ab	5.42 c
FM966(T)	3.78 k	33741 a-c	0.0658 g-i	440 b	5.25 c
PM2326(T)	4.38 h	29744 d-g	0.0688 de	550 a	4.46 d
SG521R(T)	3.51 m	28503 e-i	0.0690 cd	410 b	6.75 a
FM5024(T)	3.70 l	27834 f-i	0.0741 a	500 ab	5.58 c
FM832(G)	4.36 h	33565 a-d	0.0627 k	217 d	4.87 cd
DP491(G)	4.48 g	25829 hi	0.0645 h-j	295 c	4.74 cd
FM958(G)	5.18 b	27465 g-i	0.0652 g-j	335 c	4.26 d
FM966(G)	4.58 f	29935 c-g	0.0661 f-h	225 d	5.23 c
SG747(G)	5.45 a	25029 i	0.0656 g-j	258 d	3.90 d
DPPearl(G)	4.74 d	30042 c-g	0.0657 g-j	195 d	4.71 cd
DP555(G)	4.39 h	27530 g-i	0.0645 h-j	235 d	4.82 cd
SG747(M)	4.78 d	30369 c-g	0.0653 g-j	245 d	3.83 d
PH355(M)	4.90 c	33085 b-d	0.0652 g-j	275 cd	4.05 d
FM958(M)	4.73 de	27019 g-i	0.0643 ij	240 d	3.92 d
DPPearl(M)	4.68 e	33424 a-d	0.0639 jk	250 d	3.75 de
FM966(M)	4.56 f	34227 ab	0.0663 e-g	260 d	3.87 d
FM832(M)	4.07 i	37211 a	0.0655 g-j	245 d	3.50 ef
DP491(M)	4.50 g	31655 b-e	0.0658 g-i	245 d	3.45 ef
DP555BR(M)	4.53 fg	30099 c-g	0.0642 jk	215 d	3.52 ef

^z Cultivar: DP = Deltapine, PM = PayMaster, SG = SureGrow (Delta Pine and Land Co.; Scott, MS) FM = Fibermax (Bayer Cropsciences; Research Triangle Park, NC), PHY = Phytogen (Phytogen Seed Co.; Indianapolis, IN). Location: T = Texas High Plains, G = Georgia, M = Mississippi Delta. Location: T = Texas High Plains, G = Georgia, M = Mississippi Delta.

^y Means within a column followed by the same letter are not significantly different according to Duncan's multiple range test ($P \leq 0.05$).

Statistics. All regressions were performed using SigmaPlot 8.0 (SPSS Science; Chicago, IL), and one-way analysis of variance were performed using SigmaStat 3.0 (SPSS Science; Chicago IL).

RESULTS AND DISCUSSION

Results of physical and chemical measurements for crop years 2001 and 2002 are presented in Tables 1 and 2, respectively. A comparison of cotton fiber

moisture content with micronaire for the 21 cotton samples from crop year 2001 and the 25 samples from crop year 2002 are shown in Figure 1. The data from CY 2002 are more scattered and are generally have lower moisture content than data from CY 2001, a result that was unexpected but not surprising given the difference in harvest conditions between the two years. During CY 2002, each growing region included in this study experienced unusual amounts of rainfall subsequent to boll opening but prior to

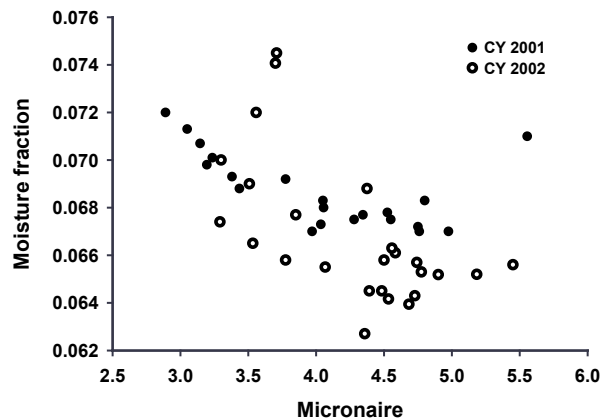


Figure 1. Comparison of micronaire with the fiber moisture fraction for CY2001 and CY2002 cottons.

harvest (Table 1). No unusual harvest conditions existed on a widespread basis during CY 2001. These conditions are reflected in data obtained on water extractable surface electrolytes from the cotton lint. When cotton fiber is extracted with water, the conductivity of the resultant solution is indicative of the proportion of electrolytes present on the fiber. Previous work demonstrated that the amount of electrolytes present on the surface for cottons harvested in conditions of low to moderate rainfall are inversely proportional to micronaire (Gamble, 2003). The results from each crop year are distinctly different. Cotton from CY 2002 had substantially lower levels of surface electrolytes than CY 2001 cotton (Fig. 2). Heavy rainfall subsequent to boll opening results in the extraction of water-soluble surface components present in or on the exposed lint. The cause of the observed moisture differences between CY 2001 and CY 2002 may be related directly to

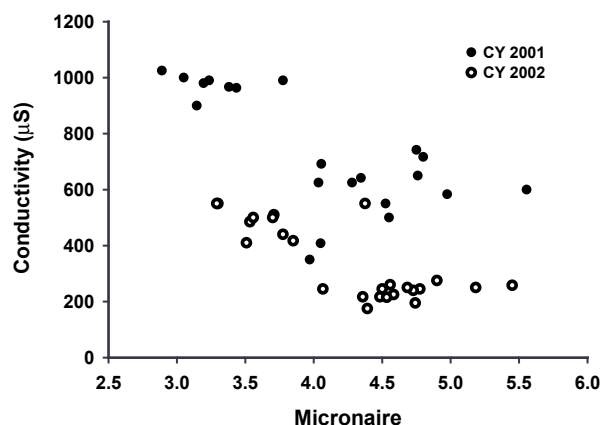


Figure 2. Comparison of micronaire with the conductivity from a 20 ml water extract of 1 gram of fiber for CY2001 and CY2002 cottons.

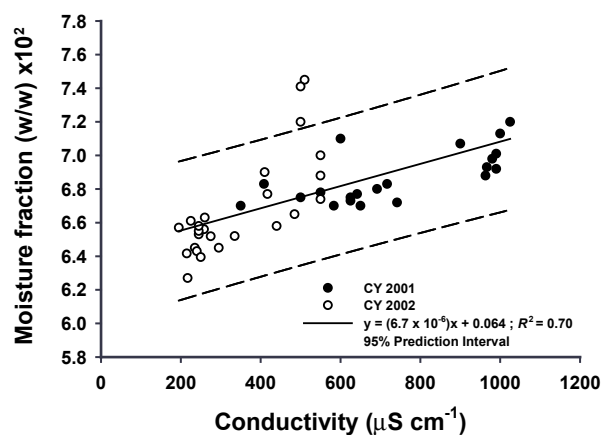
this decrease in surface electrolytes, but it also points to the possibility that CY 2002 experienced one or more wet-dry cycles prior to harvest. Wet-dry cycles have been shown to alter moisture regain values in cellulose (Kongdee et al., 2004).

Previous work demonstrated that cellulose moisture content decreases as a function of increasing crystallinity index for a set of cellulose samples ranging from 45 to 95% crystalline (Mihrianyan et al., 2004). In order to determine whether the moisture content of cellulose in the cotton fiber is a function of crystallinity, the crystallinity indices all samples in both the 2001 and 2002 crops were measured by FTIR. The crystallinity index is essentially a measure of the fraction of hydroxyl (OH) groups that are hydrogen bonded in a regular crystalline manner. A measure of the crystallinity index of raw cotton is based on the ratio of IR absorption at 901 cm⁻¹ and 1427 cm⁻¹ (O'Connor et al., 1958). It has been determined that the band at 1427 cm⁻¹ decreased or disappeared as cotton was decrystallized mechanically (ball mill) or chemically (ethylamine swelling) means (O'Connor et al., 1958). Concomitantly, the band at 901 cm⁻¹ increased in intensity as crystallinity decreased. The crystallinity index (I_x) is proportional to the ratio of the peaks at 1427 and 901 cm⁻¹ in the mid-infrared spectrum:

$$I_x \propto A_{1427}/A_{901} \tag{Eq. 1}$$

There were no significant differences of the infrared ratio among the 46 samples from both crop years. The average value of these ratios ($A_{1427}/A_{901} = 3.05 \pm 0.269$) indicates a crystallinity index (I_x) of approximately 80.0 ± 2.0 %, based on an empirical correlation of infrared ratios with X-ray diffraction data (Nelson and O'Connor, 1964). Based on previous results (Mihrianyan et al., 2004), celluloses varying in crystallinity from 78 to 82% exhibited an approximately 0.5% moisture difference. The samples from the 2001 crop year ranged from 7.2 to 6.7% in moisture content, but no corresponding differences in crystallinity were determined. This may be due to the relatively low precision of the FTIR method, since the expected differences fall within the range of the standard error involved with the method. A more precise measurement of I_x is needed in order to explore the possibility of small but significant differences in crystallinity. If in fact there were no differences in crystallinity between the samples, then the moisture differences are due to other factors. The

samples from CY 2002 range from approximately 7.6 to 6.3% in moisture content, a difference which, if due to crystallinity differences, should have been measurable by FTIR. The fact that no statistical differences in crystallinity were observed in the 2002 CY suggests that a separate factor, such as surface electrolyte or glucose content, was responsible for the observed moisture differences due to the potentially hygroscopic nature of these components. The relationship between moisture content and surface electrolytes, indicated by conductivity of the water extract, from both CY 2001 and CY 2002 are presented in Figure 3. Three points falling outside the 95% prediction interval were treated as outliers. A linear fit of the remaining points gave an $R^2 = 0.70$. Though the correlation is not sufficient to infer a direct relationship, it indicates that surface electrolyte content may play a role in the overall moisture content of the cotton fiber, possibly through intrinsic hygroscopic properties of these electrolytes.



individual fibers in a cross section of the sliver can be determined by

$$\# \text{ fibers} = D_s / D_f \quad (\text{Eq. 2})$$

where D_s is the linear density of the sliver, and D_f is the linear density of the individual fibers. Micronaire is used as an approximate measure of individual fiber linear density in units of $[M (0.0000392 \text{ g/m})]$, and equation 1 thus becomes

$$\# \text{ fibers} = 375,000 / M \quad (\text{Eq. 3})$$

where M indicates micronaire. In order to ascertain the fiber friction per individual fiber, the fiber-fiber friction measurement made on the sliver are divided by the number of fibers in a cross-sectional area of the sliver

$$F_{s,rr} / \# \text{ fibers} = F_{f,rr} \quad (\text{Eq. 4})$$

where $F_{s,rr}$ is the fiber-fiber friction measured by the Rotorting on the sliver and $F_{f,rr}$ is the calculated fiber-fiber friction of an individual fiber in the sliver. Substituting equation 5 into equation 6, then

$$F_{f,rr} = (F_{s,rr})(M) / 375,000 . \quad (\text{Eq. 5})$$

Equation 5 provides a basis for a comparison of moisture content with the fiber friction of individual fibers for CY 2001 and CY 2002 (Fig. 5). The linear regression within the 95% prediction interval for both crop years results in $R^2 = 0.54$. This does not provide a strong correlation but does indicate a trend of individual fiber friction increasing as a function of decreasing fiber moisture content. Although the data from both crop years are fit by a single equation, a distinct separation between crop years was apparent. A comparison of fiber friction with conductivity also showed a separation between crop years (Fig. 6), and when the data was fit to a linear equation a correlation of $R^2 = 0.70$ was obtained, although the data appear to follow a non-linear behavior. By contrast, when fiber friction was compared with wax content (Fig. 7), the two crop years again show separation, but a linear fit ($R^2 = 0.09$) showed that there was essentially no correlation between the two parameters.

The relationship of fiber friction with both moisture content and conductivity (Fig. 8 and 9, respectively) were further demonstrated using the sample FM 958(M) that was treated with various concentrations of sodium acetate. Figure 8 indicates that the treated cotton experienced a decrease in fiber friction as the moisture content increased. This increase in moisture content (Fig. 4) is a result of increasing

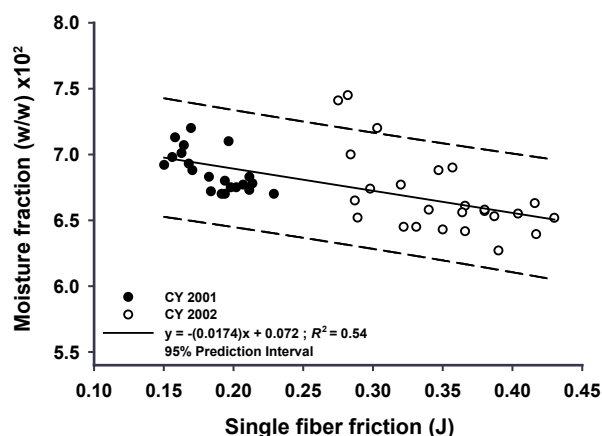


Figure 5. Relationship between fiber moisture fraction with single fiber friction for CY 2001 and CY 2002 cottons.

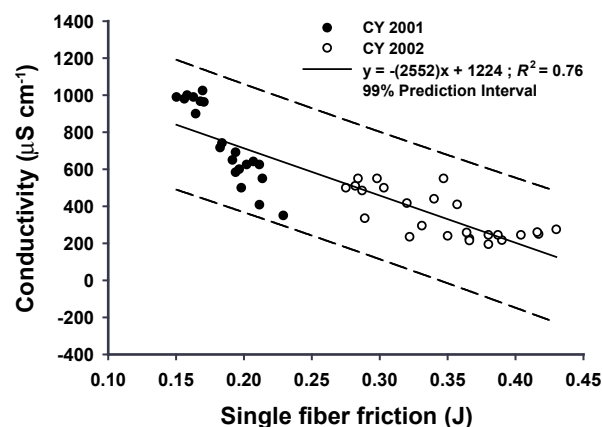


Figure 6. Relationship between conductivity of water extract with single fiber friction for CY 2001 and CY 2002 cottons.

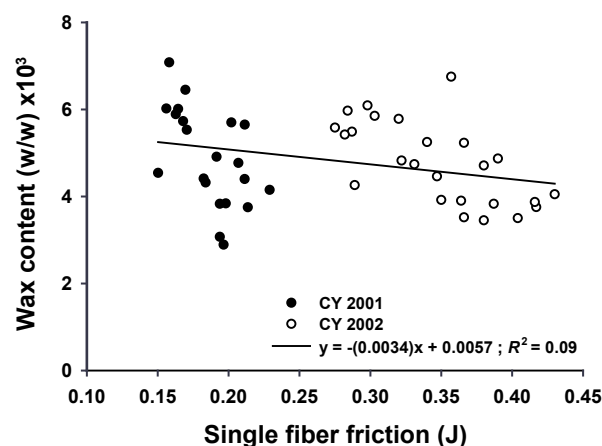


Figure 7. Relationship between fiber wax fraction with single fiber friction for CY 2001 and CY 2002 cottons.

concentration of hygroscopic surface electrolytes, in this case sodium acetate. The trend was similar to that observed in Figure 5 for the CY 2001 and CY 2002 cottons, although the linear fit parameters are different. Comparison of Figure 9 with Figure 6 also indicated a similarity in a trend toward decreasing fiber friction with increasing surface electrolyte concentration. It appears plausible that the processing difficulties experienced with the CY 2002 cottons relative to CY 2001 cottons are due at least in part to the washing of the fiber surface free of electrolytes by rainfall. The part that these hygroscopic electrolytes play in decreasing fiber friction is possibly related to an anti-electrostatic effect, which is comparable to widely marketed quaternary ammonium salts used as anti-static agents. Identification and quantification of naturally occurring electrolytes on cotton fiber, as well as an exploration of their potential role as anti-electrostatic agents, will be the subject of ongoing investigation.

CONCLUSIONS

Samples from both crop years (CY 2001 and CY 2002) exhibited moisture contents that varied inversely to micronaire, although the moisture values for CY 2002 were for the most part systematically lower and more variable. This difference between the two crop years was related to the fact that CY 2002 samples experienced substantially higher amounts of rainfall subsequent to boll opening and prior to harvest than CY 2001, a difference reflected in the relative amounts of surface electrolytes present. No measurable changes in cellulose crystallinity were observed as a function of moisture content differences for either crop year, whereas moisture content appeared to be correlated to the amount of surface electrolytes present. This relationship was elucidated through a controlled treatment of cotton fiber with varying amounts of sodium acetate, which is somewhat hygroscopic in nature. These treated samples exhibited a similar trend of increasing moisture as a function of electrolyte content. The effect of moisture and electrolyte content on fiber processing was determined using the Rotorring as a measure of the ability to process the fiber. Though the correlations were not sufficient for predictive purposes, they indicated a substantial impact of electrolyte and moisture content on ability to process the fiber, and may provide the basis of a method for the prediction of ability to process the fiber not currently available with standard HVI measurements.

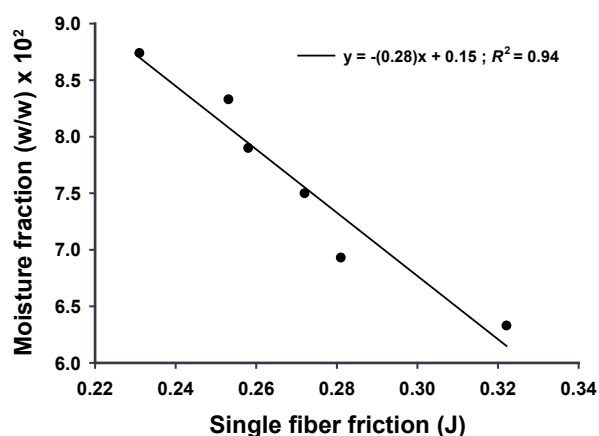


Figure 8. Relationship between fiber moisture fraction with single fiber friction for a cotton sample from FM 958(M) (CY 2002) treated with different concentrations of sodium acetate.

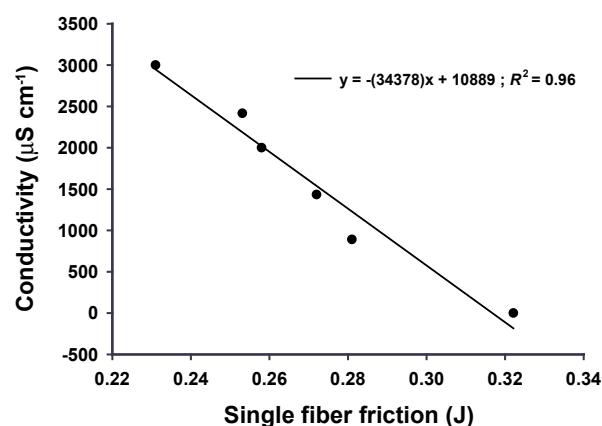


Figure 9. Relationship between conductivity of water extract with single fiber friction for sample FM 958(M) (CY 2002) treated with various concentrations of sodium acetate.

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