

## TEXTILE TECHNOLOGY

### Preliminary Assessment of Lint Cotton Water Content in Gin-Drying Temperature Studies

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#### ABSTRACT

**A new reference method for water content in lint cotton, based on Karl Fischer Titration, is highly selective for water and gives precise results. The aim of this study was to evaluate the influence of cultivar, defoliation time and gin-drying temperature on water content of lint cotton, raw and cleaned. Five cultivars were grown in the field in Mississippi in 2009 and subjected to two possible defoliation dates and gin-drying temperatures. The ginned lint underwent further processing including mechanical cleaning, and scouring and bleaching. Also, prediction models were developed to help understand how defoliation timing influences water content changes within cultivars. Overall, the mean water content across all samples analyzed was (%): raw, 7.83; cleaned, 7.69; and scoured and bleached, 8.10. Within cultivar range of the averaged water data from the various processing treatments was (%): raw, 0.19; mechanically cleaned, 0.13; and scoured and bleached, 0.08. The hypothesis that the different genetic backgrounds of the cultivars, defoliation dates and gin-drying temperatures would not affect water content of cleaned samples within cultivars was accepted as true, but only for the scoured and bleached samples. Model predictions of the change in water content with defoliation timing agreed well with experimental data. The data demonstrates that a change in water content in the botanical trash in ginned lint (raw cotton) may be confused with that due to a change in maturity. It is recommended that the correlations between water content and maturity be limited to cleaned cottons.**

Moisture measurements in lint cotton (see Appendix) can now be expressed in two distinct ways, moisture content and water content. Moisture content (ASTM D2495, 2007) refers to the weight loss by standard oven-drying and is expressed as a percentage of the moist material. The oven-drying method could be affected by other volatiles present in cotton and some water remains in the cotton sample (Cheuk et al., 2011; Montalvo et al., 2010; Rodgers et al., 2010). Water content (ASTM D7785, 2012) refers to the standard Karl Fischer Titration (KFT) method that only detects the amount of water in lint cotton and is expressed as a percentage of the moist material.

The dependence of moisture content on gin-drying treatment has been reported (Hart et al., 1955; Hessler and Workman, 1959). The results showed that as gin-drying temperature increased, moisture content of the lint cotton immediately following ginning decreased. However, when the ginned samples were brought to moisture equilibrium in a laboratory and then analyzed for moisture content, the data was found to be influenced by the drying history of the samples, the cultivar, and the extent of maturity. In other studies there is an interest in the correlation of moisture regain with micronaire and maturity as they varied widely with crop year (Rousselle and Thibodeaux, 2006). Additional research revealed essentially no relationship between moisture content and micronaire at mid micronaire values but a strong inverse correlation at low micronaire values (Gamble, 2004). The underlying mechanism to explain these findings has not been proposed.

The influence of the impurities in ginned lint (e.g., botanical trash) on moisture content has not been introduced in fundamental or applied studies. Since the moisture content method is nonspecific for water in cotton, the impurities in the samples may confound correlations between moisture content and important fiber properties.

The Southern Regional Research Center (SRRRC) developed the water content method based on Karl Fischer Titration (Montalvo et al., 2011; ASTM D7785, 2012). In practice, the specimen tested is placed in a sealed glass container and heated for a few minutes in a small oven attached to the instrument.

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Moisture released is transported by dry nitrogen carrier gas into the titration cell where it is titrated with Karl Fischer reagent. Iodine in the reagent reacts stoichiometrically with water. The end point is determined electrometrically with platinum electrodes at controlled current between the electrodes. The chemistry is described by two reactions:



where ROH is an alcohol and R'N an organic base (Scholz, 1984). Note that the first reaction is reversible while the second is irreversible. At the end point, there is slight excess of iodine. This is what the detector electrode is sensing.

Attention at SRRC has shifted to applications of the Karl Fischer Titration method. Models have been developed to predict the difference in water content before and after cleaning raw cotton with the Shirley analyzer (Montalvo and Von Hoven, 2012). The models are based on the mass fraction of trash removed in cleaning, and the water content in the trash and cleaned cotton. The measured difference in water content compared well with the predicted difference. Surprisingly, the water content in botanical trash removed from raw cotton was about twice that of the cleaned fibers, but the mass fraction of trash in ginned lint may be small.

This is a preliminary report of the influence of cultivar, defoliation timing, and gin-drying temperature on water content of ginned lint, raw and cleaned. Five commercial cultivars and two possible defoliation timings, and gin-drying temperatures were investigated. The objectives of the research were: (1) test the hypothesis that defoliation date and gin-drying temperature do not affect water content – at moisture equilibrium – of cleaned cottons within cultivars, and (2) develop models to help understand how early or late defoliation influences a change in water content within cultivars of lint cotton, raw and cleaned.

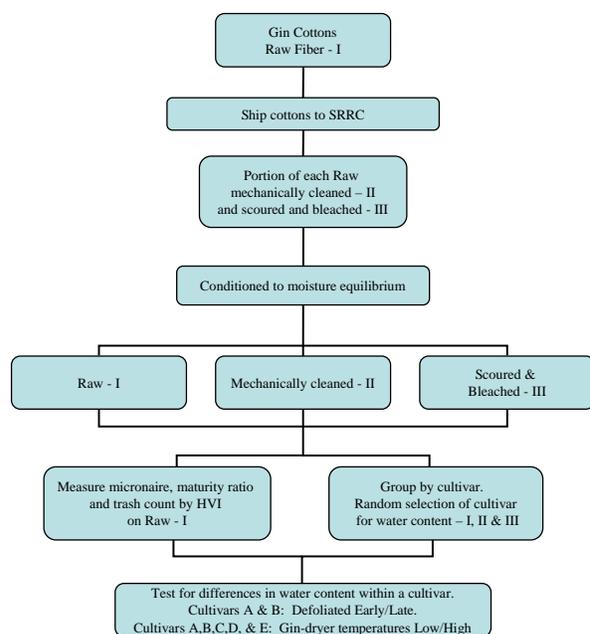
## MATERIALS AND METHODS

**Cottons and Gin-Drying Treatments.** Five cultivars were available for this study, all of which were grown in Stoneville, MS in 2009. For all five cultivars, two possible gin dryer settings were studied; 32.2°C, (Low) and 82.2°C, (High), respectively. Two of the cultivars were harvested with two possible defoliation dates (early and late), and thus six

bags of seed cotton were collected for ginning in the micro gin at the Stoneville ARS research facility. The remaining six bags of seed cotton were obtained from three late harvested cultivars, all of which experienced the two gin drying temperatures. Standard gin processing was used with dryer 1, cylinder cleaner, stick machine, dryer 2, cylinder cleaner, extractor-feeder/gin stand, and one lint cleaner. Each of the 12 bags (Gin ID 1 to 12) was ginned separately (Table 1).

**Cleaning the Lint Samples.** There were three levels of cleaning in this study: none (raw), mechanically cleaned, and scoured and bleached. One hundred g samples of cotton fibers were mechanically cleaned using the Shirley Analyzer (SDL Atlas, Rock Hill, SC). Two passes were made through the analyzer.

Three g of each of the 12 raw cottons were scoured and bleached (Figure 1). The samples were placed in separate cotton cloth bags and the bags closed by sewing with cotton thread. The bags were placed in a Werner Mathis Lab Jumbo Jet (JFO 15606) machine using the recommended solutions for scouring and bleaching. After the final rinse, the chamber was again filled with de-ionized water, the pH adjusted to 7.0 with acetic acid and drained. The 12 small sacks of cotton were removed from the chamber and allowed to dry at room temperature. Scoured and bleached fibers were removed from the cloth bags, placed in paper bags, and stored in the conditioning room.



**Figure 1.** Flow chart illustrating creation of three sets of cotton samples (I, II, and III), water content determination on all samples, and HVI analysis of set I for three fiber properties.

Table 1. Ginned samples for this study sorted by cultivar.

Cultivar & GinID Code			Defoliation & Gin		HVI Fiber Properties			
Cultivar Actual/Revised	Gin ID	Combine	Defoliation	Dryer heat <sup>z</sup>	Micronaire	Maturity Ratio	Trash Count	
STV4554B2RF	A	2	A2	Early	Low	3.5	0.84	51
		1	A1	Late	Low	4.7	0.86	38
		8	A8	Late	High	4.6	0.86	33
STV4427B2RF	B	4	B4	Early	Low	3.7	0.85	57
		3	B3	Late	Low	4.6	0.88	42
		9	B9	Late	High	4.6	0.88	44
FM960BR	C	5	C5	Late	Low	4.1	0.87	27
		10	C10	Late	High	4.1	0.87	24
DP164B2RF	D	6	D6	Late	Low	4.1	0.86	23
		11	D11	Late	High	4.0	0.86	23
PHYTO485	E	7	E7	Late	Low	3.8	0.84	48
		12	E12	Late	High	3.8	0.84	56

<sup>z</sup> Low 90°F and High 180°F.

**Conditioning Systems for Water Content Determination.** Following standard textile testing conditions, a conditioning room set to 70° ± 2°F and 65% ± 2% relative humidity was used. To control humidity to a greater degree, a glove box containing a salt solution was used within the conditioned lab to acclimatize samples to moisture equilibrium prior to measuring water content. The glove box (Coy Laboratory Products, Grass Lake, MI) had a volume of 370.4 L. Three crystallizing dishes (14.5 cm diameter) were filled with a salt solution and distributed in the box. Each dish contained a saturated solution of sodium nitrite prepared by mixing 250 mL deionized water and 250 g A.C.S. reagent grade sodium nitrite to produce 65% relative humidity at 21°C (Wink and Sears, 1950). The glove box also contained a small portable fan and a balance.

**Water Content by Karl Fischer Titration.** Following the standard conditioning period to moisture equilibrium in the glove box, water content was determined by Karl Fischer Titration, a procedure highly selective for water in cotton (Montalvo, Von Hoven, and Cheuk, 2011; Cheuk et al., 2011; ASTM D7785). The apparatus (Metrohm USA, Tampa, FL) consists of a fully automated Metrohm 774 oven sample processor held at 150°C, a 35 glass vial carousel, 800 Dosino with electronic burette, 801 stirrer, 803 Ti stand for the titration cell with platinum electrode, and the Tiamo 1.2 titration software.

Note that the Karl Fischer specimens were conditioned, weighed, placed in vials, and capped while in the glove box. Using gloved hands, 0.1000 ± 0.0003 g specimens (six replicates/sample) were

weighed, placed in KFT glass vials and immediately crimped with septum caps. To maintain the conditioned environment, the sealed vials were placed in acclimated Mason jars where they remained until just prior to being placed on the KFT carousel. Hydranal® composite 5K was used as the titration reagent and Hydranal® medium K was the solvent in the titration cell. Mean water content (%) and standard deviation were calculated from the amount of reagent consumed after correction for the blank.

**In Vitro NIR.** To ensure samples were free of moisture following KFT analysis, randomly selected sealed samples were removed from the carousel immediately after moisture testing and analyzed on a Bruker MPA near infrared instrument using OPUS 5.0 software (Bruker Optics Inc., Billerica, MA). The vial was placed on the carousel, and the spectrum read through the bottom of the vial. Three spectra were taken and averaged. Samples were considered dry if there was no visible peak in the spectrum at 1930 nm (Montalvo et al., 2010).

**HVI Fiber Properties.** Micronaire, maturity and trash count were determined by HVI (Uster, Model 1000, Knoxville, TN) at standard testing conditions (Figure 1). Mean values were calculated from five replicates.

**Data Analysis.** Descriptive statistics were performed with Excel 2007 (Microsoft Corp., Redmond, WA) to provide descriptive statistics about the basic features of the data (mean values of HVI fiber properties, means and standard deviations of water content data, and range of water content values). Analysis of variance was performed with Excel 2007 to assess

the differences in mean water content for samples within cultivars at fixed extent of cleaning: no cleaning (raw), mechanical cleaning, and scoured and bleached. Statistical significance was set at  $p < 0.05$ .

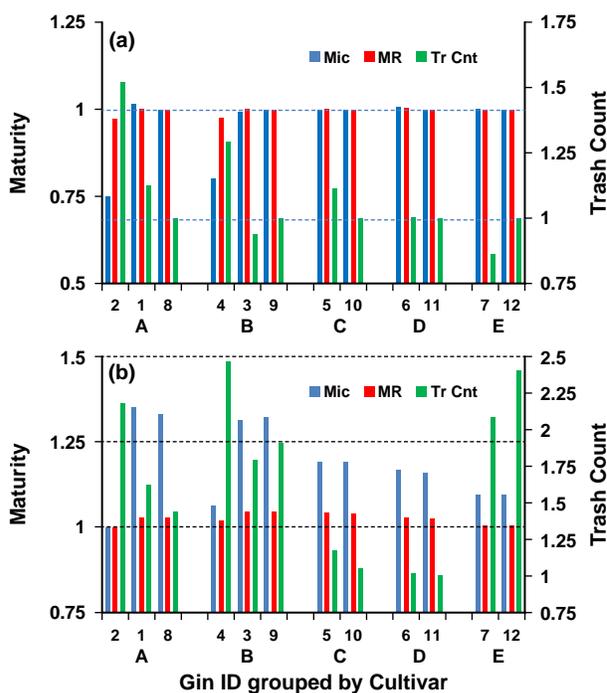
**Prediction Models for Defoliation Related Difference in Water Content and Computer Simulation.** Prediction models for differences in water content in early and late defoliated cottons within cultivars were developed. Derived models for before and after mechanical cleaning, without regard to defoliation timing, were extended to include two possible defoliation periods, early and late (Montalvo and Von Hoven, 2012). Computer simulation was accomplished with Excel 2007.

## RESULTS AND DISCUSSION

**Fiber Properties by HVI.** Moisture content of ginned lint is reported to be influenced by maturity (Gamble, 2004; Rousselle and Thibodeaux, 2006). Water content of ginned lint was found to be influenced by the water in the botanical trash in the cotton (Montalvo and Von Hoven, 2012). Consequently, micronaire, maturity ratio and trash count were analyzed by HVI to determine if changes in water content within cultivars could be explained by the variability in these parameters.

To aid in understanding the extent of change in maturity (micronaire and maturity ratio) and trash content, Table 1 data was normalized in two ways that complement each other (Figure 2). In Figure 2(a) normalization was relative to the specific cotton within each cultivar that was subjected to late defoliation and high gin-drying temperature (A8, B9, C10, D11 and E12). The micronaire of the two early-defoliated cottons gin-dried at low temperature, A2 and B4, were much lower, 25% and 20%, respectively, than the comparable late-defoliated treatments (A1 and B3). The change in micronaire in all five cottons (A1, B3, C5, D6 and E7) that were defoliated late and gin-dried at low temperature, compared to late defoliation and high gin-drying temperature, did not exceed 1.5%. There were similar trends with maturity ratio, but the changes are less. Trash counts were more variable, with a very large increase for the early-defoliated cottons: A2, 52% and B4, 29% compared to their respective counterparts A1 and B3. The change in trash count in all five cottons defoliated late and gin-dried at low temperature (A1, B3, C5, D6 and E7), compared to late defoliation and high gin-drying temperature, and did not exceed 14%.

In Figure 2(b) normalization was relative to the smallest value for each parameter across all 12 cottons (micronaire and maturity ratio, A2 and trash count, D11). The smallest micronaire values are associated with the early-defoliated cultivars (A2 and B4). The change in micronaire between early and late defoliation was dramatic. Note that the normalized micronaire value increased from 1 to 1.35 (A2 to A1) and 1.06 to 1.32 (B4 to B3). Although cultivar E was defoliated late in the season, its maturity was less than that of cultivars C and D.



**Figure 2.** HVI micronaire, maturity ratio and trash count of the 12 raw cottons normalized to: (a) within each cultivar, the specific cotton subjected to late defoliation and high gin-drying temperature (A8, B9, C10, D11 and E12) and (b) between cultivars, the smallest value for each parameter across all cottons (Mic and MR, A2 and Tr Cnt, D11). Legend: micronaire (Mic), maturity ratio (MR) and trash count (Tr Cnt). Both frames: primary y-axis – Mic and MR, and secondary y-axis – Tr Cnt.

Trash count was strongly influenced by maturity; lower maturity resulted in a greater trash count. The two early-defoliated cottons (A2 and B4) and the late-defoliated cultivar E, (7 and 12) exhibited the largest trash counts. Normalized trash counts were as follows for cultivars A, B, and E (trash count): A2 (2.19), A1 (1.62), A8 (1.44); B4 (2.47), B3 (1.79), B9 (1.91); and E7 (2.09), E12 (2.41). In the late-defoliated cottons there was no apparent trend within cultivars with either maturity or trash count versus gin-drying temperature.

Thus, there were impressive changes in maturity and trash count among the 12 cottons in this study. Are there corresponding significant changes in water content? The dependency of water content on defoliation date (maturity), level of trash in the cottons and gin-drying temperature is critically analyzed below followed by the use of water content prediction models to glean insight into the results.

**Hypothesis.** Tables 2 and 3 introduce the reader to the water content descriptive statistics. The grand means (Table 2) across the cottons varied with cleaning regimen (%): raw, 7.83; mechanically cleaned, 7.69; and scoured and bleached, 8.10. Table 3 displays the range of the averaged water content values, within and between cultivars.

The hypothesis that the different genetic backgrounds of the five cultivars, the range of micronaire and maturity levels, and two gin-drying temperatures, would not affect KFT water content of cleaned samples within cultivars was developed for this study. Note that none of these treatments would be considered excessive, but the early defoliation resulting in low micronaire would be unusual for cotton grown in the Mississippi delta.

**Table 2. Water Content by Karl Fischer Titration.**

Sample code	Mean ± std dev (%) <sup>z</sup>		
	Raw	Mech Cl	S&B
A2	7.79 ± 0.04	7.65 ± 0.08	8.09 ± 0.05
A1	7.89 ± 0.10	7.76 ± 0.07	8.16 ± 0.06
A8	7.92 ± 0.05	7.78 ± 0.06	8.09 ± 0.05
A <sup>y</sup>	7.87 ± 0.07	7.73 ± 0.07	8.11 ± 0.06
B4	7.84 ± 0.09	7.73 ± 0.05	8.00 ± 0.10
B3	7.94 ± 0.06	7.79 ± 0.04	8.08 ± 0.07
B9	7.86 ± 0.06	7.72 ± 0.04	8.03 ± 0.09
B	7.88 ± 0.07	7.75 ± 0.04	8.04 ± 0.09
C5	7.80 ± 0.06	7.67 ± 0.03	7.99 ± 0.06
C10	7.64 ± 0.03	7.60 ± 0.08	7.96 ± 0.10
C	7.72 ± 0.05	7.64 ± 0.06	7.98 ± 0.08
D6	7.75 ± 0.08	7.61 ± 0.04	8.12 ± 0.07
D11	7.74 ± 0.08	7.49 ± 0.09	8.17 ± 0.03
D	7.75 ± 0.08	7.55 ± 0.07	8.15 ± 0.06
E7	8.01 ± 0.08	7.76 ± 0.06	8.21 ± 0.06
E12	7.82 ± 0.05	7.73 ± 0.03	8.24 ± 0.04
E	7.92 ± 0.07	7.75 ± 0.05	8.23 ± 0.05
<b>Grand mean ± pooled std dev<sup>y</sup></b>	<b>7.83 ± 0.07</b>	<b>7.69 ± 0.06</b>	<b>8.10 ± 0.07</b>

<sup>z</sup> Ginned lint (Raw), mechanically cleaned (Mech Cl), and scoured and bleached (S&B).

<sup>y</sup> Mean ± pooled std dev

**Table 3. All Cultivars – range of avg. values within and between cultivars.**

Cultivar	Water Content Range (%)		
	Raw <sup>z</sup>	Mech Cl	S&B
<b>Within:</b>			
A (3) <sup>y</sup>	0.13	0.13	0.07
B (3)	0.10	0.07	0.08
C (2)	0.16	0.07	0.03
D (2)	0.01	0.12	0.05
E (2)	0.19	0.03	0.03
<b>Between:</b>	<b>0.20</b>	<b>0.20</b>	<b>0.25</b>

<sup>z</sup> Ginned lint (Raw), mechanically cleaned (Mech Cl), and scoured and bleached (S&B).

<sup>y</sup> No. cottons within a cultivar.

Table 4 details the p values used to test the hypothesis for statistical significance. First, for the raw cottons, only the B cultivar (20 % of the cultivars) showed the sample averages within the cultivar were not significantly different. Second, for the mechanically cleaned cottons, 40 % of cultivars (C and E) showed the sample averages within a cultivar were not significantly different. Finally, for the scoured and bleached cottons, 100% of cultivars tested showed the sample averages within a cultivar were not significantly different. Thus, the hypothesis is accepted as true but only for the scoured and bleached cottons. Scouring and bleaching fibers removed the impurities from the samples and the gin-drying history of the cottons was negated by the contact with liquid water in the cleaning process.

**Table 4. Statistical significance test of water contents at p < 0.05**

Cultivar	p value		
	Raw <sup>z</sup>	Mech Cl	S&B
A (3) <sup>y</sup>	0.014	0.011	0.083
B (3)	0.086	0.017	0.372
C (2)	0.0005	0.128	0.535
D (2)	0.0003	0.006	0.105
E (2)	0.00002	0.090	0.355
<i>No. of cottons: 12</i>	<i>3</i>	<i>4</i>	<i>12</i>

<sup>z</sup>Ginned lint (Raw), mechanically cleaned (Mech Cl), and scoured and bleached (S&B).

<sup>y</sup>No. cottons within a cultivar.

The decision to clean the cottons for this investigation by mechanical cleaning, and scoured and bleached but not water washing was based on a search of the literature for the two extremes – high and low efficiency – in cleaning. Scouring and bleaching re-

moves the waxy coating on the fiber, as well as other impurities and exposes more fibrils to the conditioned atmosphere (Goynes et al, 1984; Wakelyn et al, 2007). Thermo-gravimetric analysis (TGA) of three varieties of cotton showed that the scoured and bleached control cotton produced less thermal residue compared to simple water washing with a wetting agent (Ward et al., 1985). Chemical profiling of water-washed and mechanically cleaned cottons showed that water washing removed more impurities than mechanical cleaning (Rousselle et al., 1983). Thus, the two extremes in cleaning efficiency are mechanical cleaning (low efficiency), and scoured and bleached (high efficiency).

**Prediction Models for Defoliation Related Difference in Water Content.** Two within cultivar models (Eqs. 1 and 2) were developed (Montalvo and Von Hoven, 2012) to predict water content in ginned lint before mechanical cleaning ( $W_b$ ), and the difference in water content before and after cleaning ( $W_b - W_c$ ).

$$W_b = F_i W_i + (1 - F_i) W_c \quad [1]$$

$$W_b - W_c = F_i (W_i - W_c) \quad [2]$$

The symbols (see Appendix)  $W_b$ ,  $W_c$ , and  $W_i$  are the concentrations (%), in that order, of water in the raw cotton, cleaned fibers, and isolated botanical trash, and  $F_i$  the mass fraction of the aggregate trash

removed by mechanical cleaning. The experimental difference in water content ( $W_b - W_c$ ) compared well with that predicted from the modeling using the Shirley analyzer to clean the cottons.

Now consider extending Eq. 1 to models associated with early ( $E$ ) and late ( $L$ ) defoliated cottons (i.e., low and high maturity) within cultivars (see Appendix):

$$W_{Eb} = F_{Ei} W_{Ei} + (1 - F_{Ei}) W_{Ec} \quad [3]$$

$$W_{Lb} = F_{Li} W_{Li} + (1 - F_{Li}) W_{Lc} \quad [4]$$

There are six possible prediction models for defoliation related difference in water content:  $W_{Eb} - W_{Ec}$ ,  $W_{Lb} - W_{Lc}$ ,  $W_{Eb} - W_{Lb}$ ,  $W_{Ec} - W_{Lc}$ ,  $W_{Eb} - W_{Lc}$ , and  $W_{Ec} - W_{Lb}$ . These differences occur due to a change in maturity and/or change in the physical form of cotton – before and after mechanical cleaning.

In this preliminary study the prediction models are derived for five of the combinations (Table 5):  $W_{Eb} - W_{Ec}$ ,  $W_{Lb} - W_{Lc}$ ,  $W_{Eb} - W_{Lb}$ ,  $W_{Ec} - W_{Lc}$ , and  $W_{Eb} - W_{Lc}$ . These combinations are sufficient to help understand the effect of defoliation time on the predicted change in water content. (The reader may wish to derive the remaining subset,  $W_{Ec} - W_{Lb}$ . Hint: Solve for  $W_{Ec}$  in Eq. (3), subtract Eq. (4) and simplify.)

**Table 5. Prediction models for difference (%) in water content of within cultivar cottons subjected to defoliation and cleaning treatments.**

$W_{Eb} - W_{Ec} = \int (F_{Ei}, W_{Ei}, W_{Ec})$	[5]
$W_{Eb} - W_{Ec} = F_{Ei} (W_{Ei} - W_{Ec})$	[6]
$W_{Lb} - W_{Lc} = \int (F_{Li}, W_{Li}, W_{Lc})$	[7]
$W_{Lb} - W_{Lc} = F_{Li} (W_{Li} - W_{Lc})$	[8]
$W_{Eb} - W_{Lb} = \int (F_{Ei}, F_{Li}, W_{Ei}, W_{Li}, W_{Ec}, W_{Lc})$	[9]
$W_{Eb} - W_{Lb} = F_{Ei} (W_{Ei} - W_{Ec}) + F_{Li} (W_{Lc} - W_{Li}) + (W_{Ec} - W_{Lc})$	[10]
$W_{Ec} - W_{Lc} = \int (F_{Ei}, F_{Li}, W_{Ei}, W_{Li}, W_{Eb}, W_{Lb})$	[11]
$W_{Ec} - W_{Lc} = \left( \frac{W_{Eb} - F_{Ei} W_{Ei}}{1 - F_{Ei}} \right) - \left( \frac{W_{Lb} - F_{Li} W_{Li}}{1 - F_{Li}} \right)$	[12]
$W_{Eb} - W_{Lc} = \int (F_{Ei}, F_{Li}, W_{Ei}, W_{Li}, W_{Ec}, W_{Lb})$	[13]
$W_{Eb} - W_{Lc} = F_{Ei} (W_{Ei} - W_{Ec}) + W_{Ec} - \left( \frac{W_{Lb} - F_{Li} W_{Li}}{1 - F_{Li}} \right)$	[14]
<b>Reduction of prediction models at <math>F_{Ei}</math> and <math>F_{Li} = 0</math>:</b>	
$W_{Eb} - W_{Ec} = 0$	[6']
$W_{Lb} - W_{Lc} = 0$	[8']
$W_{Eb} - W_{Lb} = W_{Ec} - W_{Lc}$	[10']
$W_{Ec} - W_{Lc} = W_{Eb} - W_{Lb}$	[12']
$W_{Eb} - W_{Lc} = W_{Ec} - W_{Lb}$	[14']

Note, for example, that the predicted difference in water content ( $W_{Eb} - W_{Lb}$ ; Eq. 10) comes from four sources of water in the raw cotton: botanical trash and fibers in early-defoliated ginned lint, and trash and fibers in late defoliated lint. In contrast, the difference in water content ( $W_{Ec} - W_{Lc}$ ; Eq. 12) is due to change in maturity between the early and late defoliation; the trash had been removed by mechanical cleaning. As to ( $W_{Eb} - W_{Lc}$ ; Eq. 14), this predicted difference in water content is equal to the sum of the water concentration in the trash and fibers in early-defoliated lint subtracted by that in late-defoliated lint after mechanical cleaning.

To eliminate in the models the contribution of water in the trash to the water content of cottons within a cultivar, let the trash mass fractions equal to zero (i.e.,  $F_{Ei}$  and  $F_{Li} = 0$ ). The ensuing models, Table 5 (Eqs. 6' to 14'), are reduced in complexity.

**Simulation of the Prediction Models.** Assumptions and assigned default input values for the within cultivar simulations are tabulated in Table 6. Default values close to the experimental data and data contained in Montalvo and Von Hoven (2012) were used. Note that the possible sources of the water in the cottons within a cultivar are the botanical trash and the fibers. The water concentration in the trash is about twice that of the cleaned fibers (Montalvo and Von Hoven, 2012). Additionally, some values were set equal to each other (7.75 % water) to initialize the simulations at reasonable values. Unless stated otherwise, equal weight was given to a change in water content (positive 0.2 % water) due to introduction of trash or a change in maturity (e.g., 7.75 % water + 0.2 % water = 7.95 %).

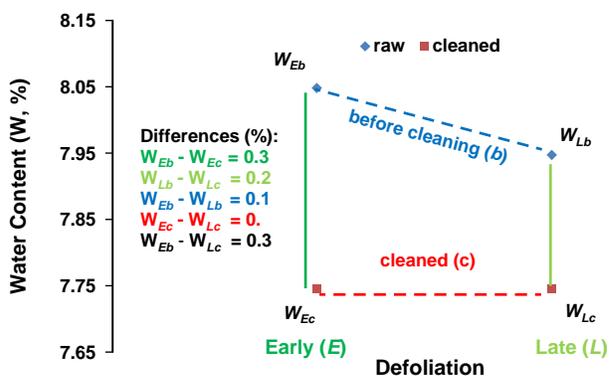
Four cases were simulated in this study: case I – no trash in the early and late defoliated cottons, and no change in maturity between defoliation periods; case II – no trash and a change in maturity; case III – the same mass fraction of trash, and water concentration in the trash, in early and late defoliated cottons, and no change in maturity; and case IV – 1.5 x more trash in early compared to late defoliation and no change in maturity.

Results are summarized in Table 7. The third column from the left in the table gives the difference results by direct calculation from the input data in Table 6. For example, in case I, the assigned default values in Table 6 are  $W_{Eb} = W_{Ec} = 7.75$  % water so that by direct calculation  $W_{Eb} - W_{Ec} = 0$ . The case I calculations for all of the models (Table V) – Eqs. 6 to 14 and 6' to 14' – produced results that matched those by direct computation.

With no trash in the cottons within a cultivar (cases I and II), the only possible source of water is the cotton fibers. A change in water content between the early- and late-defoliated cottons (case II) is due to a change in maturity. Case II calculations for all of the models produced results that matched those by direct calculation.

Cases III and IV simulations are concerned with trash in the cottons and no change in maturity between early- and late-defoliations within a cultivar. The difference in the amount of water in the hypothetical cottons was predicted by Eqs. 6, 8, 10, 12 and 14. Results match those by direct calculation. In contrast, the reduced models ignored the trash by assigning the trash mass fractions  $F_{Ei} = F_{Li} = 0$  (Eqs. 6', 8', 10', 12' and 14'). These models generally gave incorrect predictions, especially Eq. 14' wherein  $W_{Eb} - W_{Lc}$  was too low a forecast: 0.4 % (case III) and 0.5 % (case IV).

Graphic analysis of case IV simulation is shown in Fig. 3. The early-defoliated cotton contained 1.5 x more trash than late-defoliation (Table 6). (Also see that in Table 1, A2 and B4, respectively, contained 1.44 x and 1.33 x higher trash count compared to late defoliation.) The order of the five differences in water content listed in Figure 3 ( $W_{Eb} - W_{Ec} = 0.3$  % to  $W_{Eb} - W_{Lc} = 0.3$  %) corresponds to that in the direct calculation column in Table 7. Four of the five differences in the figure contain at least one term related to water content before cleaning ( $W_{Eb}$  and/or  $W_{Lb}$ ). After cleaning, water content was the same in the early- and late-defoliated fibers since there was no maturity change in this simulation ( $W_{Ec} - W_{Lc} = 0$  %).



**Figure 3. Graphic analysis of Case IV simulation of raw cotton. Trash level in early defoliated cotton 1.5 x that in the late defoliated cotton. No maturity difference between defoliation periods. See Appendix for glossary of water content differences.**

**Table 6. Input hypothetical data for simulation of the models within cultivar.**

**Assumptions:**  
**Case I.** No trash in the early and late defoliated raw cottons. No change in maturity between the early and late defoliations. Default values for defoliation related before and after cleaning are all 7.75 %. (See default values summary below.)  
**Case II.** No trash in the early and late defoliated raw cottons. A change in maturity between early and late defoliation. The maturity change resulted in an increase in water content in the early defoliated cotton, before and after cleaning, by 0.2 %. Default values in the early defoliated cotton are 7.95 % water compared to 7.75 % for late defoliation.  
**Case III.** Trash in the early and late defoliated raw cottons. No change in maturity between early and late defoliation. Same trash mass fraction and same water concentration in the trash in the early and late defoliated cottons. The trash resulted in an increase in water content in the early and late defoliated cottons before cleaning, by 0.2 %. Default values before cleaning are 7.95 % water compared to 7.75 % after cleaning.  
**Case IV.** Same as Case III except the mass fraction of trash in the early defoliated cotton is 1.5 x that in late defoliation cotton. Default values before cleaning are 8.05 % ( $W_{Eb}$ ) and 7.95 % ( $W_{Lb}$ ) compared to 7.75 % after cleaning.

Simulation Case	Trash mass fraction				Water (%)			
	$F_i$		$W_i$		$W_b$		$W_c$	
	$F_{Ei}$	$F_{Li}$	$W_{Ei}$	$W_{Li}$	$W_{Eb}$	$W_{Lb}$	$W_{Ec}$	$W_{Lc}$
I	0	0	0	0	7.75	7.75	7.75	7.75
II	0	0	0	0	7.95	7.75	7.95	7.75
III	0.02857	0.02857	14.75	14.75	7.95	7.95	7.75	7.75
IV	0.02857x1.5	0.02857	14.75	14.75	8.05	7.95	7.75	7.75

**Table 7. Prediction results (% water) from simulation of the models within cultivars.**

Tr	Cnt	MR <sup>z</sup>	Model	Direct Calc.	[6]	[8]	[10]	[12]	[14]	[6']	[8']	[10']	[12']	[14']
<b>Case I:</b>														
N <sup>y</sup>	N <sup>x</sup>		$W_{Eb} - W_{Ec}$	0.	0.					0.				
			$W_{Lb} - W_{Lc}$	0.		0.					0.			
			$W_{Eb} - W_{Lb}$	0.			0.					0.		
			$W_{Ec} - W_{Lc}$	0.				0.					0.	
			$W_{Eb} - W_{Lc}$	0.					0.					0.
<b>Case II:</b>														
N	Y		$W_{Eb} - W_{Ec}$	0.	0.					0.				
			$W_{Lb} - W_{Lc}$	0.		0.					0.			
			$W_{Eb} - W_{Lb}$	0.2			0.2					0.2		
			$W_{Ec} - W_{Lc}$	0.2				0.2					0.2	
			$W_{Eb} - W_{Lc}$	0.2					0.2					0.2
<b>Case III:</b>														
Y	N		$W_{Eb} - W_{Ec}$	0.2	0.2					0.				
			$W_{Lb} - W_{Lc}$	0.2		0.2					0.			
			$W_{Eb} - W_{Lb}$	0.			0.					0.		
			$W_{Ec} - W_{Lc}$	0.				0.					0.	
			$W_{Eb} - W_{Lc}$	0.2					0.2					-0.2
<b>Case IV:</b>														
Y	N		$W_{Eb} - W_{Ec}$	0.3	0.3					0.				
			$W_{Lb} - W_{Lc}$	0.2		0.2					0.			
			$W_{Eb} - W_{Lb}$	0.1			0.1					0.		
			$W_{Ec} - W_{Lc}$	0.				0.					0.1	
			$W_{Eb} - W_{Lc}$	0.3					0.3					-0.2

<sup>z</sup> Trash count (Tr Cnt) and maturity ratio (MR).

<sup>y</sup> N is no trash in the hypothetical cotton.

<sup>x</sup> N is no change in MR in the hypothetical cotton between early and late defoliations.

Clearly, the water in the botanical trash in raw cotton may confound correlations with micronaire and maturity. The correlations should be limited to mechanically cleaned samples to prevent confusion due to water in the trash.

**Evaluation of Prediction Models with Experimental Data.** The three cottons selected in this preliminary study to evaluate the models (B4/B3 and E12) represent the extremes in defoliation date and gin-drying temperature. Note that the B cultivar had been subjected to early- (B4) and late- (B3) defoliation, and a low gin-drying temperature. In contrast, both of the E cultivar cottons were defoliated late in the season and ginned at a low (E7) and high (E12) gin-drying temperature; only E12 was included in this study. For convenience, the input experimental data and the prediction results are detailed in Table 8.

For the B cultivar, the difference in water content, as predicted by Eqs. 6, 8, 10, 12 and 14, agreed well with results derived by direct calculation. The reduced models ignored the trash by assigning the trash mass fractions  $F_{Ei} = F_{Li} = 0$  (Eqs. 6', 8', 10', 12' and 14'). These models generally gave the same trends between that by direct calculation and the abridged models as observed in the hypothetical simulations, in particular Eq. 14' wherein  $W_{Eb} - W_{Lc}$  was too low a forecast by 0.26 % (B4/B3). As regards the E cultivar results, only the  $W_{Lb} - W_{Lc}$  models (Eqs. 8

and 8') are of interest since the other models are not applicable (they contain one or more terms related to early defoliation). Prediction results by Eq. 8 agreed well with that by direct calculation; again, ignoring the trash in the cotton (Eq. 8') gave too low a forecast.

### CONCLUSIONS

Commercial cultivars of various stages of maturity were subjected to different gin-drying temperatures in this preliminary study. Sub-samples of the ginned lint were mechanically cleaned, and scoured and bleached. After conditioning to moisture equilibrium, water content was determined by ASTM D7785 (Karl Fischer Titration). Additionally, micronaire, maturity ratio and trash count were measured to decide if variability in these parameters matched changes in water content.

The hypothesis that the different genetic backgrounds of the cultivars – subjected to two possible defoliation dates and gin-drying temperatures – would not affect water content of cleaned samples within cultivars was accepted as true for the scoured and bleached samples. Compared to late defoliation, micronaire and maturity ratio of the early-defoliated cottons were reduced and trash count was considerably higher. Nonetheless, variability of these fiber properties did not result in within cultivar changes in water content of the scoured and bleached fibers.

**Table 8. Input experimental data representing sample code B4/B3 and E12, and the prediction results<sup>z</sup>.**

Data description: The within cultivar numerical code B4 (early defoliation and low gin-drying temperature) and B3 (late defoliation and low gin-drying temperature); and E12 (late defoliation and high gin-drying temperature).											
Sample Code	Trash mass fraction				Water (%)						
	$F_i$		$W_i$		$W_b$		$W_c$				
	$F_{Ei}$	$F_{Li}$	$W_{Ei}$	$W_{Li}$	$W_{Eb}$	$W_{Lb}$	$W_{Ec}$	$W_{Lc}$			
B4	0.0186		14.58		7.84		7.73				
B3		0.0135		15.04		7.94		7.79			
E12		0.0180		15.04		7.82		7.73			
Prediction results (% water)											
Model	Direct Calc.	Model									
		[6]	[8]	[10]	[12]	[14]	[6']	[8']	[10']	[12']	[14']
B4/B3	$W_{Eb} - W_{Ec}$	0.11	0.13				0.				
	$W_{Lb} - W_{Lc}$	0.15	0.10				0.				
	$W_{Eb} - W_{Lb}$	-0.1		-0.03				-0.06			
	$W_{Ec} - W_{Lc}$	-0.06			-0.13				-0.1		
	$W_{Eb} - W_{Lc}$	0.05				0.01				-0.21	
E12	$W_{Lb} - W_{Lc}$	0.09	0.13				0.				

<sup>z</sup> Source: Tables 1 and 2 ( $W_b$ , before cleaning and  $W_c$ , after mechanical cleaning). Water content in the isolated trash ( $W_i$ ) measured by Karl Fischer Titration for B4 and E12 (Montalvo and Von Hoven, 2012); the value for B3 assumed equal to that for E12. Trash mass fraction ( $F_i$ ) value for B3 taken as the averaged value of six cottons in a comprehensive study (Montalvo and Von Hoven, 2012; Montalvo and Mangialardi, 1983); for B4 and E12 computed as the ratio 0.0135 x Trash Count (Table 1)/Trash Count B3.

Prediction models were derived and simulated to help understand the change in water content at different defoliation times. The two sources of water in the models are the botanical trash in the raw cottons and the cleaned fibers. The predictions agreed with experimental data except when the models were simplified by assuming there was no trash in the ginned lint.

This study has demonstrated that the contribution of water in botanical trash to the water content of raw cotton may be measured, and also predicted by models, as the difference in water content between the raw and mechanically cleaned fibers. Correlations of maturity with water content should be done on cleaned cotton data to prevent confounding of results due to the water in the trash.

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### DISCLAIMER

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### APPENDIX: GLOSSARY OF TERMS AND SYMBOLS

#### Terms

**ginned lint:** cotton fibers that have been separated from their seeds by ginning but not subjected to any further processing after ginning (ASTM D123, 2009)

**lint cotton:** loose cotton fibers in any form, either raw or processed, free of seeds and not bound together in yarn or fabric (ASTM D123, 2009)

**raw cotton:** ginned lint that has not been subjected to any textile manufacturing process (ASTM D123, 2009)

#### Symbols

$F_i$ : mass fraction of the aggregate botanical trash removed from lint cotton in a specific cleaning operation (Shirley analyzer in this paper)

$F_{Ei}$ : mass fraction of the aggregate botanical trash removed from early-defoliated cotton in a specific cleaning operation (Shirley analyzer in this paper)

$F_{Li}$ : mass fraction of the aggregate botanical trash removed from late-defoliated cotton in a specific cleaning operation (Shirley analyzer in this paper)

$W_i$  (%): concentration of water in the trash removed from cotton by mechanical cleaning and followed by manual separation of entrained lint

$W_{Ei}$  (%): concentration of water in the trash removed from early defoliated cotton by mechanical cleaning and followed by manual separation of entrained lint

$W_{Li}$  (%): concentration of water in the trash removed from late defoliated cotton by mechanical cleaning and followed by manual separation of entrained lint

$W_b$  (%): concentration of water in the cotton before mechanical cleaning

$W_{Eb}$  (%): concentration of water in the early-defoliated cotton before mechanical cleaning

$W_{Lb}$  (%): concentration of water in the late-defoliated cotton before mechanical cleaning

$W_c$  (%): concentration of water in the mechanically cleaned fibers

$W_{Ec}$  (%): concentration of water in the early-defoliated cotton after mechanical cleaning

$W_{Lc}$  (%): concentration of water in the late-defoliated cotton after mechanical cleaning

#### Water Content Differences (%) Within Cultivar

$W_{Eb} - W_{Ec}$ : difference in water content in early-defoliated cotton before and after mechanical cleaning; due to trash removal in the early-defoliated cotton

$W_{Lb} - W_{Lc}$ : difference in water content in late-defoliated cotton before and after mechanical cleaning; due to trash removal in the late-defoliated cotton

$W_{Eb} - W_{Lb}$ : difference in water content between early- and late-defoliated cotton before cleaning; due to the combined effect of a change in maturity and trash in the early- and late-defoliated cottons

$W_{Ec} - W_{Lc}$ : difference in water content between early- and late-defoliated cotton after cleaning; due to a change in fiber maturity

$W_{Eb} - W_{Lc}$ : difference in water content between early- defoliated cotton before cleaning and late defoliated cotton after cleaning; due to the combined effect of a change in maturity and the trash in the early-defoliated cotton

$W_{Ec} - W_{Lb}$ : difference in water content between early-defoliated cotton after cleaning and late defo-

liated cotton before cleaning; due to the combined effect of a change in maturity and the trash in the late defoliated cotton

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