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TEXTILE TECHNOLOGY

Modeling of Water Content in Cotton Before and After Cleaning With the Shirley Analyzer

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ABSTRACT

The role of botanical trash in the observed water content of ginned cotton has not been elucidated. Recently, a reference method for water content in raw and cleaned cottons based on Karl Fischer Titration was evaluated. The objective of this study was to compare – by modeling and Karl Fischer Titration – the water content in lint from two cultivars before and after mechanical cleaning with the Shirley analyzer. Models were developed as a function of the mass fraction of the aggregate trash removed in cleaning, and the water contents in the trash and cleaned cotton. Water content was also measured in the isolated trash after correction for entrained lint. The grand means in water content across both cultivars were : raw, 7.83%; after cleaning, 7.73%; and trash, 14.81%. Thus, the directly measured grand means difference in water content before and after cleaning was only 0.10%. This compared well with the predicted difference from modeling using the averaged mass fraction of isolated trash of 0.0135 from published work on six cottons. The water content in the trash particles was about twice that of the cleaned fibers; the mass fraction of trash was minute. Clearly, the mass fraction of impurities is the controlling factor in establishing the difference in water content before and after cleaning. Although only a limited number of cultivars were studied, these data suggest a decrease in water content of < 0.5% due to removal of botanical trash in screening studies from the bale to the yarn.

The amount of moisture in cotton is important in all phases of the cotton industry, from harvesting to textile mill processing (Byler, 2006; Lord, 2005). The Southern Regional Research Center has developed a new reference method for water (free and bound) in lint cotton based on Karl Fischer Titration (KFT). The new assay is highly selective for water in lint cotton and has good precision (see Appendix for definition of terms and math symbols). In practice, the sample is placed in a sealed glass container and heated in a small oven for five minutes at 150°C (Cheuk et al., 2011; Montalvo, Von Hoven, and Cheuk, 2011). Moisture released is transported by dry nitrogen carrier gas into the KFT cell where it is titrated with Karl Fischer reagent. The iodine in the reagent reacts stoichiometrically with water and the end point is determined electrometrically with platinum electrodes. The measurement process is fully automated.

Several applications of the KFT method are in progress, with the general emphasis on comparing water content with moisture content measured by the standard oven drying procedure (ASTM D2495, 2007). In the latter method, all weight loss is attributed to moisture. Extensive research has shown that the standard oven drying method does not remove all of the water in cotton. Additionally, there is some oxidation of the sample in the standard oven drying procedure for measuring moisture content that confounds weight loss results (Montalvo et al., 2010).

In this study, two cultivars of different maturity were gin-dried at low or high temperature. Ginned lint from each cultivar was then mechanically cleaned by the Shirley analyzer. After conditioning the samples to moisture equilibrium, the different fiber matrices (raw and cleaned) were analyzed for water content. The mean difference in water content before and after cleaning the raw cottons with the Shirley analyzer was small. The underlying physical mechanism to explain this finding has not been proposed in fundamental or applied studies. Is the water content in raw cotton influenced weakly, moderately, or strongly by the botanical trash that is removed by Shirley cleaning?

Perhaps the small change in water content as a result of mechanical cleaning is due to approximately the same amount of water in both fibers and the trash in the cotton. Still another plausible explanation is that the mass fraction of the trash removed in the cleaning process must be considered. Thus, there is a

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need to: (a) develop models to predict and explain the difference in water content before and after mechanical cleaning, and (b) experimentally test the models. After the models are validated it will be possible to expand the studies to include module to bale and bale to yarn since cotton is mechanically cleaned in these processes. The work reported here differs from previous research in that the KFT method measures water content rather than weight loss in oven drying.

This is a report of the development of two water content models. One of the models is used to explain and predict the water content in raw cotton; the other is concerned with the difference in water content before and after cleaning raw cotton with the Shirley analyzer. Water content is given as a function of three independent variables. These independent variables are combined in simulation trials to produce unique changes in water contents. Then the model is tested with two cultivars. Testing of the models using moisture content by standard oven drying rather than water content by KFT was also attempted, but there were inconsistencies in results. This was expected because of the aforementioned deficiencies in the standard oven drying procedure. This study will provide a deeper understanding of how botanical trash influences water content of ginned lint.

FUNDAMENTALS

Water Content Models. Consider the mass fraction of the two-component mixture of fibers and botanical trash in raw cotton (i.e., ginned lint)

$$F_i + F_c = 1 \tag{1}$$

where F_i is the mass fraction of the aggregate trash particles removed from lint cotton in a specific cleaning operation (Shirley analyzer in this paper, see Appendix). The Shirley analyzer only removes the plant parts and some dust but not the other impurities, such as inorganic salts, waxes and pectins on the surface of the fibers and amorphous material in the lumen. As a consequence, F_i is << 1 (Montalvo and Mangialardi, 1983). The mass fraction of the cleaned cotton is given by F_c.

The mass of water in the cotton before cleaning is

$$M_b = m_{sx}F_i(W_i/100) + m_{sx}F_c(W_c/100) \tag{2}$$

where m_{sx} is the sample weight and W_i and W_c are the concentrations (%) of water in the botanical trash and cleaned fibers, respectively. Substitution of Eq. 1 into Eq. 2 gives:

$$M_b = (m_{sx}/100)(F_iW_i + (1 - F_i)W_c). \tag{3}$$

The concentration (%) of water in the cotton before cleaning is

$$W_b = (M_b/m_{sx})100. \tag{4}$$

Substitution of Eq. 3 into Eq. 4 gives (see Figure 1)

$$W_b = F_iW_i + (1 - F_i)W_c. \tag{5}$$

A simple check of the default conditions of Eq. 5 confirms that if $F_i = 0$, then $W_b = W_c$ and if $W_i = W_c$, then $W_b = W_i = W_c$ and is independent of F_i.

Equation 5 can be rearranged to give the difference (% units) in the concentration of water in the cotton before and after cleaning

$$W_b - W_c = F_i(W_i - W_c) = F_iD \tag{6}$$

where $D = W_i - W_c$.

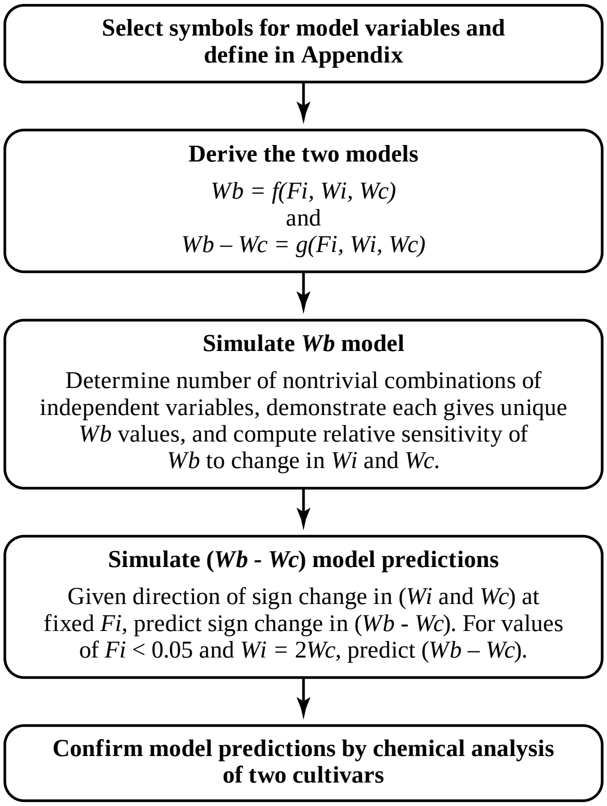


Figure 1. Flow chart of methodology to develop the models for water content in raw cotton before cleaning (W_b) and the difference between before and after cleaning (W_b - W_c). The mass fraction of impurities removed by cleaning (F_i) may vary from 0 to < 1.

MATERIALS AND METHODS

Cottons and Gin-Drying Treatments. The two cultivars selected for this study (ST 4554B2RF and PHY 485WRF) represent extremes in defoliation date and gin-drying temperature. Cultivar ST 4427B2RF had been subjected to early defoliation and a low (32.2°C) gin-drying temperature. In contrast, cultivar PHY 485WRF was defoliated late in the season and ginned at a high (82.2°C) gin-drying temperature. The cottons had been grown in 2009 in Stoneville, MS and ginned at the Stoneville ARS research facility. Standard gin processing was used.

Shirley Analyzer Cleaning and Isolation of Botanical Trash Particles. The two cottons (100 g each) were mechanically cleaned using the Shirley analyzer (ASTM D2812, 2007). Two passes were made through the analyzer. Three 0.1g samples of trash or impurities from each cultivar were separated from the entrained lint in the Shirley analyzer waste with the aid of forceps. (These particles were analyzed for water as described below.)

Moisture and Water Contents at Moisture Equilibrium

Conditioning Systems. Following standard textile testing conditions, a conditioning room set to 21.1° ± 1°C and 65 % ± 2 % relative humidity was used. Cotton samples were conditioned to moisture equilibrium for at least 24 h before measuring moisture content and water content. A glove box was used within the conditioned lab to improve humidity control in acclimating the samples for measuring water content by KFT. Humidity in the glove box was held constant by the use of a saturated aqueous salt solution, which prevented the cyclical nature of humidity control by the standard HVAC system. (Heating, ventilation and air conditioning systems use air ducts to supply conditioned air; a noticeable cyclic trend in the regulated temperature and humidity is inherent in the design. The sensors in the current HVAC system allowed for a 2 % “window” in relative humidity; the variability in temperature was about ± 0.3°C)

A small portable fan (2.1 watts) was placed inside the glove box and fitted with a timer to circulate the air intermittently (15 min per 2 h period). An analytical balance was also placed in the box. A saturated solution of reagent grade sodium nitrite in distilled water was used to produce 65% relative humidity at 21.1°C (Wink and Sears, 1950).

The relative mass of a specimen for moisture content compared to water content determination was 10 to 1. Lack of space in the glove box prevented conditioning of the oven moisture samples.

Moisture Content by Standard Oven Drying. Following a 24 h standard conditioning period to moisture equilibrium in the conditioned room, moisture content was determined for the raw and mechanically cleaned cottons by standard oven drying (ASTM D2495, 2007) with specific changes in the procedure as noted below. Oven drying was carried out using the Yamato DKN 600 mechanical convection oven placed in a general-purpose laboratory.

Approximately 1.5 g samples (five replicates/cotton) were weighed into glass weighing bottles using gloved hands. Glass caps and weighing bottles were also allowed to condition and were weighed. Following the oven heating at 105°C oven for 24 h, the bottles were immediately capped while in the oven, removed and placed in a desiccator, allowed to cool, and then reweighed. Mean moisture content (%) and standard deviation were calculated from the weight loss data.

Water Content by Karl Fischer Titration. Following a 24 h standard conditioning period to moisture equilibrium in the conditioned glove box, water content was determined by Karl Fischer Titration on the raw, mechanically cleaned and isolated trash particles (Cheuk et al., 2011; Montalvo, Von Hoven, and Cheuk, 2011). (The Karl Fischer apparatus consists of a fully automated Metrohm 774 oven sample processor held at 150°C, with a 35 glass vial carousel, an 800 Dosino with an electronic burette, an 801 stirrer, an 803 Ti stand for the titration cell with platinum electrode, and the Tiamo 1.2 titration software.)

Note that the Karl Fischer samples were conditioned, weighed, placed in vials and capped in the glove box. Using gloved hands, 0.1 g samples (six replicates/cotton of raw and cleaned fibers; three replicates/cotton of isolated trash) were weighed to four decimal places, placed in KFT glass vials and immediately crimped with septa caps. To maintain the conditioned environment, the sealed vials were placed in acclimated Mason jars. The samples remained in the jars until just prior to testing. Mean water content (%) and standard deviation were calculated from the amount of reagent consumed after correction for the blank.

Prior to analyzing the isolated trash samples by KFT, one 0.1g sample of the isolated trash from each cotton was sealed in a KFT glass vial and pre-dried at 80°C for one hour using dry nitrogen (at a flow rate of 110 mL/min). The pre-dried trash samples were analyzed by KFT to check for non-aqueous reactive components that interfere with the titration by observing the volume of titrant consumed (Montalvo, Von Hoven, and Cheuk, 2011). Next, the two remaining 0.1 g replicates of trash from each cultivar was analyzed by KFT using the cotton fiber procedure. The amount of interfering material found in the trash, expressed as equivalent water content, was too small to alter the calculated water content in these samples.

***In Vitro* Near Infrared Reflectance (NIR).** To ensure samples (raw and cleaned cottons and trash) were free of moisture following KFT analysis, randomly selected sealed samples were immediately removed from KFT and analyzed on a Bruker MPA using OPUS 5.0 software. The vial was placed on the instrument sampling port and the spectra were taken 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 (1.93 microns) (Montalvo et al., 2010).

Developing the Models, Computer Simulation and Data Analysis. Excel 2003 was used to computer simulate discrete and continuous values of W_b as a function of one independent variable while holding the other two independent variables constant. Small increments were used in the continuous simulations within the desired range of values. The data was used to setup regression equations; the slopes and intercepts were recorded and the R^2 was 1.

RESULTS AND DISCUSSION

Model Assumptions and Assignments. The two models developed are: (a) the concentration of water in the cotton before cleaning (W_b) and (b) the difference in water content before and after mechanical cleaning (W_b - W_c). The assumption in development of the models is that the work done on raw cotton by mechanical cleaning with the Shirley analyzer does not alter water content of the two-component mixture of fibers and impurities. To minimize the effect of cleaning on water content, both the cleaned fibers and recovered botanical trash were conditioned to moisture equilibrium before water analysis.

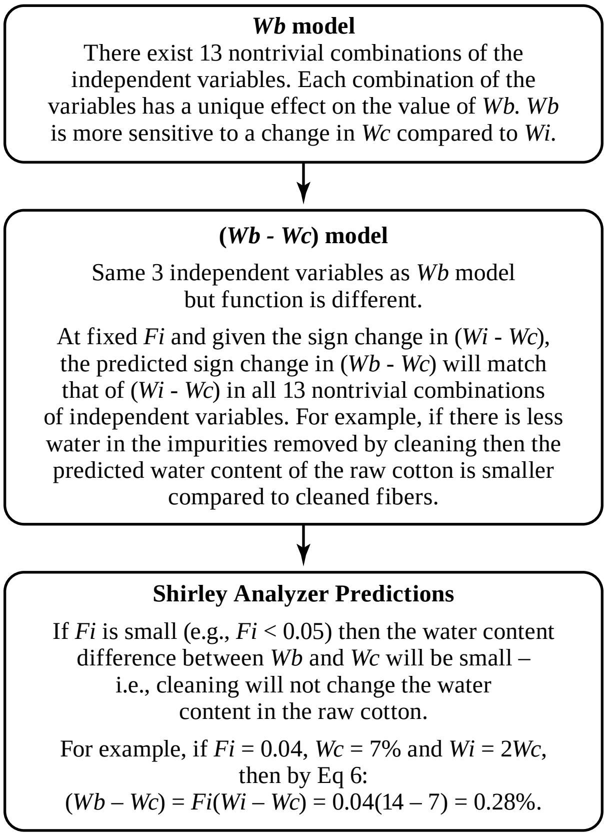
The assignment of the default values used in the tables (see below) is somewhat arbitrary. The 7% default values for W_b and W_c are close to the mean value found in six cottons (Montalvo, Von Hoven, Cheuk, and Byler, 2011). To simplify the results of Table 1 (see also Figure 1), the 7% default value was also selected for W_i. As to the F_i = 0.15 assignment, this is the value needed in Eq. [5] to produce W_b = 7 % given W_i = W_c = 7 %.

Table 1. Possible nontrivial combination of W_i, W_c, D and W_b with fixed F_i = 0.15[z].

Case	W_i (%)	W_c (%)	$D = (W_i - W_c)$ (%)	W_b (%)
	7	7	0	7
Ij	7	7	0	7
----	----7----	----7----	----0----	----7----
Ik	7	7	0	7
	7	7	0	7
	9	7	2	7.30
IIj	↑8	7	1	7.15
----	----7----	----7----	----0----	----7----
IIk	↓6	7	-1	6.85
	5	7	-2	6.70
	7	9	-2	8.70
IIIj	7	↑8	-1	7.85
----	----7----	----7----	----0----	----7----
IIIk	7	↓6	1	6.15
	7	5	2	5.30
	8	6 8	-2	7.70
IVj	↑7.5	↓6.5 ↑7.5 IVk	-1	7.35
----	7	7 7	----0----	----7----
IVk	↓6.5	↑7.5 ↓6.5 IVj	1	6.65
	6	8 6	2	6.60
	9	9	0	9
Vj	↑8	↑8	0	8
----	----7----	----7----	----0----	----7----
Vk	↓6	↓6	0	6
	5	5	0	5
	11	9	2	9.30
VIj	↑↑9	↑8	1	8.15
----	----7----	----7----	----0----	----7----
VIk	↓↓5	↓6	-1	5.85
	3	5	-2	4.70
	9	11	-2	10.70
VIIj	↑8	↑↑9	-1	8.85
----	----7----	----7----	----0----	----7----
VIIk	↓6	↓↓5	1	5.15
	5	3	2	3.30

[z] **Vertical arrows represent an increase or decrease in water values. Double arrows show W_i and W_c with twice the magnitude or rate of change compared to the corresponding water value.**

***Nontrivial Combinations of W_b* Model Variables.** The concentration of water in the cotton before cleaning (W_b) is a function of three independent variables: F_i, W_i, and W_c (see Eq. 5). Thus, we must consider the nontrivial or unique combinations of the independent variables in order to understand their combined effect on W_b (Figure 2). For example, do the unique combinations produce different, continuous W_b values within each combination and between combinations? By definition, a nontrivial combination of independent variables is a set of values for the variables that differs from the assigned default values and from each other set of values.



***Wb* model**

There exist 13 nontrivial combinations of the independent variables. Each combination of the variables has a unique effect on the value of *Wb*. *Wb* is more sensitive to a change in *Wc* compared to *Wi*.

(*Wb* - *Wc*) model

Same 3 independent variables as *Wb* model but function is different.

At fixed *Fi* and given the sign change in (*Wi* - *Wc*), the predicted sign change in (*Wb* - *Wc*) will match that of (*Wi* - *Wc*) in all 13 nontrivial combinations of independent variables. For example, if there is less water in the impurities removed by cleaning then the predicted water content of the raw cotton is smaller compared to cleaned fibers.

Shirley Analyzer Predictions

If *Fi* is small (e.g., $Fi < 0.05$) then the water content difference between *Wb* and *Wc* will be small – i.e., cleaning will not change the water content in the raw cotton.

For example, if $Fi = 0.04$, $Wc = 7\%$ and $Wi = 2Wc$, then by Eq 6:
$(Wb – Wc) = Fi(Wi – Wc) = 0.04(14 – 7) = 0.28\%$.

Figure 2. Major outcomes of simulation exercises. Note that W_b is the water content before cleaning and ($W_b – W_c$) the difference in water content before and after cleaning; F_i is the mass fraction of nonlint material removed by cleaning after correction for entrained lint.

The logic to probe for and confirm nontrivial combinations in this study is similar to two applications reported: a comparative study of NIR reflectance of cottons grouped according to cross-sectional dimensions (Montalvo, 1991; Montalvo et al., 1991) and cotton Arealometer instrument readings (Montalvo and Vinyard, 1993). In the NIR study, there were seven nontrivial combinations of paired fiber dimensional values. The relative predicted NIR reflectance at a single wavelength of light for each nontrivial combination was made possible by a rotating vector. The existence of each nontrivial combination was confirmed by experiment, but this was a long and costly project. As to the Arealometer study, there were 13 nontrivial combinations of paired instrument readings. Random generation of the data was used to confirm the existence of all of the nontrivial combinations.

To probe for all possible nontrivial combinations (Table 1), the discrete variations in W_i and W_c about the designated default values ($W_i = W_c = W_b = 7$ % at fixed $F_i = 0.15$) are: (I) both W_i and W_c display no variation (default), (II) W_i varies and W_c is fixed, (III) W_i is fixed and W_c varies, (IV) W_i and W_c move in opposite directions, (V) W_i and W_c move in the same direction at the same rate of change such that their difference $D = W_i – W_c$, is always constant, (VI) W_i and W_c move in the same direction but W_i changes at a more rapid rate than W_c, and (VII) W_i and W_c move in the same direction but W_c changes at a more rapid rate. Since two directions of change are possible (i.e., increasing or decreasing values), each principal combination (I to VII) is subdivided into two related combinations designated by the suffix j or k after the Roman numeral (e.g., IIj and IIk).

Next, Eq. 5 was used to compute W_b for each pair of W_i and W_c values in Table 1. The final step was to transform the Table 1 data to directions and relative rates of change represented by single or double arrows and also to inequalities (> or = or <) about the default values (Table 2). An examination of Tables 1 and 2 reveals thirteen nontrivial combinations in W_i and W_c values (Figure 2). Statistical regression techniques produced, where possible, a linear equation for W_b as a function of F_i, W_i or W_c; correlation coefficients were 1. None of the lines in the regression plots overlap, which means that nontrivial combination of continuous values for the independent variables produced different, continuous W_b values within each combination (i.e., j and k) and between combinations. All lines pass through $W_b = 7.0\%$ (the default value) with F_i fixed at 0.15 in going from the j to the k subcombinations within a case as expected.

Thirteen different line plots were also generated with F_i as the independent variable. All correlation coefficients produced were 1.0. (The reader can plot the results in Table 1 at fixed F_i. Plot W_b as a function of W_c, and W_b as a function of D. Observe for each plot twelve lines with different slopes and intercepts and one point in the plot space – Case I. Also, plot W_b as a function of F_i and get 13 lines with different slopes and intercepts.)

Table 2. Demonstrated nontrivial combinations of discrete water content values^z.

Case	W_i (%)	W_c (%)	$D = (W_i - W_c)$(%)	Inequalities	
				D	W_b
Ij	-	-	-		
				$(D_j = D_k)_{Wi,Wc}$	$(W_{b,j} = W_{b,k})_{Wi,Wc}$
Ik	-	-	-		
IIj	↑	-	↑		
				$(D_j > D_k)_{Wc}$	$(W_{b,j} > W_{b,k})_{Wc}$
IIk	↓	-	↓		
IIIj	-	↑	↓		
				$(D_j < D_k)_{Wi}$	$(W_{b,j} > W_{b,k})_{Wi}$
IIIk	-	↓	↑		
IVj	↑	↑ **IVk**			
				$\downarrow D_k < \uparrow D_j$	$W_{b,j} < W_{b,k}$
IVk	↓	↓ **IVj**			
Vj	↑	↑	-		
				$D_j = D_k$	$(W_{b,j} > W_{b,k})D$
Vk	↓	↓	-		
VIj	↑↑	↑	↑		
				$D_j > D_k$	$W_{b,j} > W_{b,k}$
VIk	↓↓	↓	↓		
VIIj	↑	↑↑	↓		
				$D_j < D_k$	$W_{b,j} > W_{b,k}$
VIIk	↓	↓↓	↑		
Total = 13 nontrivial combinations					

^{z} Vertical arrows represent an increase or decrease in water values. Double arrows show W_i and W_c with twice the magnitude or rate of change compared to the corresponding water value.

Table 3. Effect on W_b by reducing one independent variable in Eq. 5^z by one half.

Independent variables			Calculated values		
F_i	W_i (%)	W_c (%)	$D = W_i - W_c$	W_b (%)	ΔW_b (%)
0.15	**7**	**7**	**0**	**7 (default)**	**-**
0.15	**3.5**	**7**	**- 3.5**	**6.475**	**- 0.525**
0.15	**7**	**3.5**	**3.5**	**4.025**	**- 2.975**
0.075	**7**	**7**	**0**	**7**	**0**

^{z} Eq. 5: $W_b = F_iW_i + (1 - F_i)W_c$.

Relative Sensitivity of W_b to the Independent Variables. In raw cottons, the expected mass fraction of botanical trash removed by the Shirley analyzer is $F_i << 1$ (Montalvo and Mangialardi, 1983). Thus, examination of the two terms to the right of Eq. 5 reveals that if $F_i < 1$, then $(1 - F_i)W_c > F_iW_i$. This implies that the water content in the cotton before cleaning (W_b) is more sensitive to changes in the water content of the cleaned fibers (W_c) compared to the water content of the impurities (W_i) (Figure 2).

Table 3 shows the effect on W_b by reducing one of the independent variables in Eq. 5 by one-half, while holding the other two constant. By reducing W_i from 7% to 3.5% while keeping F_i and W_c at the default values of 0.15 and 7%, respectively, W_b decreases from 7 to 6.475% or $\Delta W_b = W_b$ (new value) – W_b (default value) = - 0.525 %. Upon reducing W_c from 7% to 3.5% while keeping F_i and W_i at the default values of 0.15 and 7%, respectively, W_b decreases from 7 to 4.025% or ΔW_b = - 2.975%. By reducing F_i from 0.15 to 0.075 while keeping W_i and W_c at the default values of 7%, there is no change in W_b ($\Delta W_b = 0$). Thus, W_b is more sensitive to a change in W_c compared to a change in W_i, and if the water content in the impurities and cleaned cotton is the same, is independent of the mass fraction of impurities in the cotton. The three examples presented here are representative of nontrivial combinations IIk, IIIk, and I.

Water Content ($W_b - W_c$) Model Predictions. It is possible to use Eq. 6 to predict the relative difference in water content in the cotton before and after cleaning with the Shirley analyzer. If the water content in the aggregate trash particles recovered from the Shirley analyzer is greater than that in the cleaned cotton (i.e., $W_i > W_c$), the model predicts that the water content in the cotton before cleaning is greater than that of the cleaned cotton (i.e., $W_b - W_c > 0$) (Table 4). If the water content is the same in both the Shirley analyzer impurities and the cleaned fibers ($W_i = W_c$), the model predicts that the water contents are the same in the cotton before and after cleaning ($W_b - W_c = 0$). Conversely, if there is a lower concentration of water in the impurities compared to that in the cleaned cotton ($W_i < W_c$), the model predicts that the water content in the cotton before cleaning is less than that in the cleaned cotton ($W_b - W_c < 0$).

The thirteen nontrivial combinations in Tables 1 and 2 were examined for the corresponding inequality or equality between ($W_i - W_c$) *and* ($W_b - W_c$) and matched with all cases (Table 4 and figure 2). Five cases were found where the two related inequalities, ($W_i - W_c$) *and* ($W_b - W_c$), were > 0, three cases for $W_i = W_c$ *and* $W_b = W_c$, and five cases for $W_i < W_c$ *and* ($W_b - W_c$) < 0, for a total of thirteen cases. In no case was there a mixing of results, demonstrating that Eq. 6 is reliable prediction model.

Experimental Test of ($W_b - W_c$) Model Predictions on Two Cultivars. The actual determination of the mass fraction of the impurities (F_i) in the Shirley analyzer visible waste was not possible in this study. Equipment required to separate the nonlint from entangled fibers (Montalvo and Mangialardi, 1983) was not available. However, to allow for NIR and KFT measurements of water content in these materials, a sufficient amount of impurities in the Shirley analyzer visible waste was separated from the entrained lint with the aid of forceps.

NIR qualitative studies on the two cultivars indicated a greater water content in the isolated nonlint material or impurities from the Shirley analyzer visible waste compared to the cleaned cotton (i.e., $W_i > W_c$), see Figures 3 and 4. Measurement of the NIR peak heights associated with the water band at 1.93 µm gave essentially identical values for the trash from each cultivar. The average peak height of the water band for the cleaned cotton from the two cultivars was 86.2 mm, which was close to the individual cottons. The NIR results suggest about the same amount of water in each cultivar, even though the STV variety was defoliated early and gin-dried at low heat compared to the Phytogen variety which was defoliated late and gin-dried at high heat.

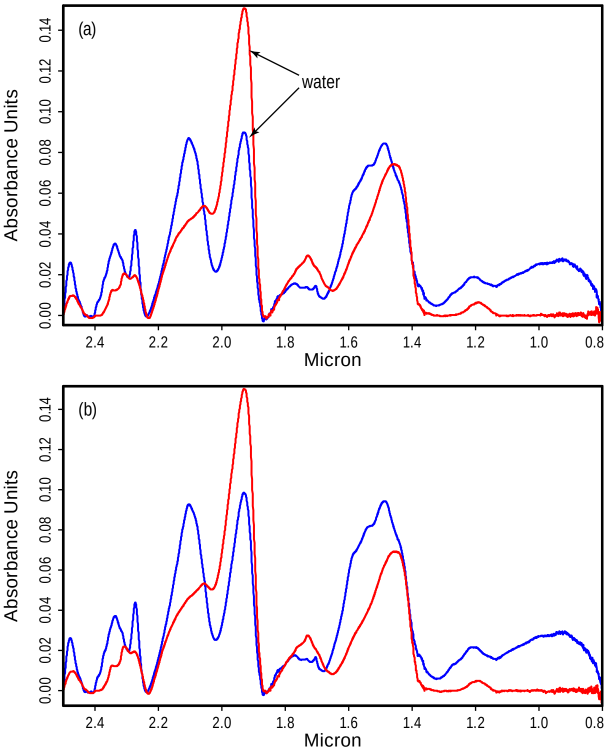


Figure 3. Baseline NIR corrected absorption spectra of Shirley analyzer cleaned cotton and isolated trash (water peak at 1.93 microns; water peak in isolated trash is stronger): (a) cultivar STV4554B2RF, and (b) cultivar PHYTO585.

Table 4. Water content predictions by Eq. 6.

Given $W_i - W_c$	Prediction $W_b - W_c$	Cases	No. cases
> 0	> 0	IIj, IIIk, IVj, VIj, VIIk	5
$W_i = W_c$	$W_b = W_c$	Ij,k, Vj, Vk	3
$W_i < W_c$	< 0	IIk, IIIj, IVk, VIk, VIIj	5
	TOTAL		13 cases

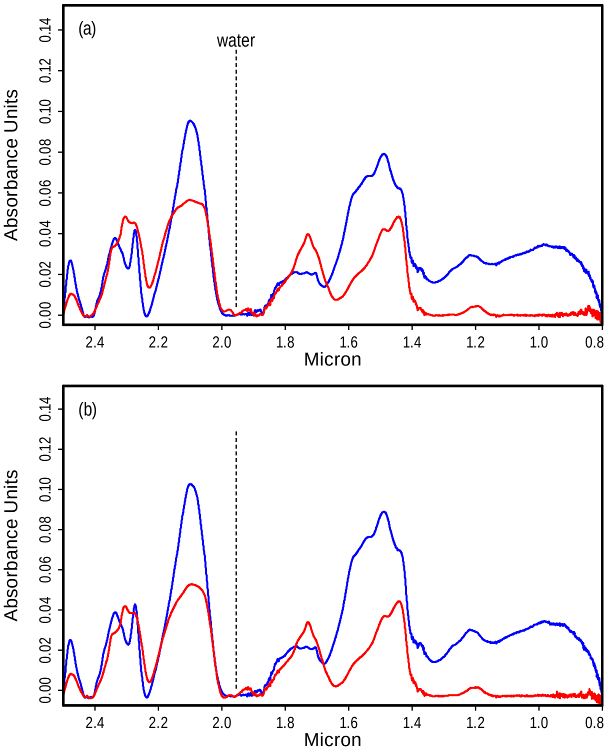


Figure 4. Baseline NIR corrected absorption spectra of Shirley analyzer cleaned cotton and isolated trash after drying for 1 hr at 80°C in nitrogen (water peak at 1.93 microns is absent): (a) cultivar STV4554B2RF, and (b) cultivar PHYTO585.

The actual amount of water in all of the materials was measured using Karl Fischer Titration (Table 5). The water content grand means and across both cultivars was : W_b = 7.83%, W_c = 7.73%, and W_i = 14.81%. Since $W_i – W_c$ = 14.81 – 7.73 = 7.08% (i.e., > 0), the model predicted (Table 4) that $W_b – W_c > 0$. Across both cottons, the actual difference in $W_b – W_c$ was positive and small: ST 4427B2RF, 0.11%; PHY485WRF, 0.09%; and the grand means, 0.10%. These results represent experimental confirmation of nontrivial combination, specifically case VIj; the results are greater than the default values of 7% for W_i, W_c, and W_b. Additionally, $W_i > W_c$.

Examination of the experimental results in Table 5 and the prediction model (Eq. 6) suggests that if $W_b – W_c$ = 0.10 % and $W_i – W_c > 7\%$, then F_i must be < 0.015 for Shirley analyzer cleaned cottons. A literature search revealed one study in which F_i had been determined for Shirley analyzer cleaned cottons (Montalvo and Mangialardi, 1983). Two hundred gram samples of six raw cottons, representing the full range of Shirley analyzer trash content, were cleaned in a Shirley analyzer (ASTM D2812, 2007) followed by a detailed protocol to isolate and measure the true nonlint material from the entrained lint in the visible waste. The corrected nonlint contents by this method ranged from 0.07% to 3.30%. The corresponding mass fraction of impurities (F_i) removed in cleaning was calculated by dividing the corrected nonlint results by 100 to give the sample set averaged and maximum values of only 0.0135 and 0.033, respectively.

A plot of the prediction model (Eq. 6) outcome using the KFT $W_i – W_c$ grand mean difference in Table 5 to give the slope of the line (14.81 – 7.73 = 7.08%) is shown in Figure 5. The hypothetical independent F_i values used to generate the plot (diamond shaped points) ranged from 0.01 to 0.15 and resulted in $W_b – W_c$ predicted values ranging from 0.07% to 1.06%.

Table 5. NIR peak heights and water content by KFT on two cultivars.

Cultivar	NIR peak height (mm)		Water content (%)		
	Cleaned cotton	Trash	Raw cotton^z (W_b)	Cleaned^z (W_c)	Trash^y (W_i)
STV4427B2RF	82.8	129.8	7.84 ± 0.093	7.73 ± 0.045	14.58 ± 0.24
PHYTO485	89.5	128.8	7.82 ± 0.051	7.73 ± 0.028	15.04 ± 0.14
		Grand means	7.83	7.73	14.81
			$W_b – W_c$ = 0.10 %		
			Moisture Content (%) by Standard Oven Drying^x		
STV4427B2RF			8.02 ± 0.13	7.60 ± 0.03	-
PHYTO485			7.48 ± 0.11	7.34 ± 0.09	-
		Grand means	7.75	7.47	
			$W_b – W_c$ = 0.28 %		

^{z} Means based on six 0.1g replicates.

^{y} Means based on two 0.1g replicates.

^{x} Means based on five 1.0g replicates.

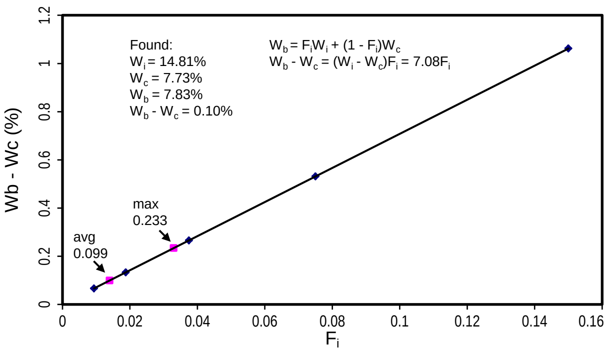


Figure 5. Dependence of the model for the difference in water content before and after Shirley analyzer cleaning (W_b – W_c) on the mass fraction of impurities removed in cleaning F_i. The "found" data represents the grand mean across the two cultivars investigated. The "max" and "avg" points represent the upper bound and grand mean across six cottons (Montalvo and Mangialardi, 1983).

Next, the Shirley analyzer sample set averaged and maximum F_i values of 0.0135 and 0.033 were superimposed on the plot (square points) and thus, the predicted W_b – W_c values for this study are 0.099% (average) and 0.233% (maximum). The grand mean W_b – W_c difference of 0.10% was found for the two cultivars in the present study (Table 5) and agrees with the predicted difference (0.099%) that was computed from the averaged F_i value on the set of six cottons (Montalvo and Mangialardi, 1983).

Again, it is worthwhile noting that these two cultivars represent extremes in defoliation date and gin-drying temperature (with same crop year and area of growth). The ST cultivar was subjected to early defoliation and low gin-drying temperature whereas the PHY cotton was defoliated late and dried at a higher temperature. Nonetheless, there was consistency in the KFT W_b and W_c data. In contrast, the grand mean W_b – W_c difference of 0.28% based on moisture content by standard oven drying (Table 5) is about three times larger. Furthermore, for the individual cultivars, (W_b – W_c) was 0.42% and 0.14%, respectively.

CONCLUSIONS

A model was developed for the concentration of water in cotton before cleaning by the Shirley analyzer. The three independent variables are the mass fraction of impurities removed by the Shirley analyzer, and the concentrations of water in the cleaned cotton and the isolated impurities. Computer simulation demonstrated that thirteen different combinations of the independent variables exist centered about designated default values. No indication of non-uniqueness could be found originating from the difference independent variables. Due to limitations on the upper bound of the mass fraction of impurities removed by the Shirley analyzer, the concentration of water before cleaning is more sensitive to changes in water content of the cleaned cotton than changes in the other two independent variables. The model was rearranged into a prediction model to estimate the relative difference in water content before and after Shirley cleaning. Again, thirteen nontrivial combinations were demonstrated. In no case was there a mixing of results, suggesting a reliable prediction model.

Experimental test of the model to estimate the difference in water content before and after cleaning with the Shirley analyzer gave good agreement with water contents measured by Karl Fischer Titration and published work on the mass fraction of impurities or trash removed by the Shirley analyzer. However, this difference was small, 0.10%. The water content in the trash particles was about twice that of the cleaned fibers; the mass fraction of trash was minute. Clearly, the mass fraction of impurities is the controlling factor in establishing the difference in water content before and after cleaning. In contrast, direct measure of the model predictions based on moisture content by standard oven drying gave results not consistent with KFT data..

One reviewer of the draft manuscript asked the question "Under what circumstances would someone want to use the model?" The models provide a better understanding in elucidating the mechanistic role of botanical trash in water content of ginned lint. The models provide a method for predicting the change in water content as a result of mechanical cleaning. Additional studies focusing on bale to yarn can be easily achieved using the approach described in this work, ultimately leading to an improved understanding of lint water content.

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DISCLAIMER

Mention of a trade name, proprietary product, or specific equipment does not constitute a guarantee or warranty by the USDA and does not imply approval of a product to the exclusion of others that may be suitable.

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APPENDIX: GLOSSARY OF TERMS AND MATH SYMBOLS

Terms

KFT: reference method developed to measure the amount of water (free and bound) in lint cotton based on a chemical reaction involving water, iodine, an alcohol and an organic base; the method is highly selective for water over interfering substances

Shirley analyzer: a mechanical cleaner used to determine non-lint content of cotton by ASTM D2812 (cleaner used in this study)

moisture content: amount of weight loss by standard oven-drying methods including non-aqueous volatile materials and expressed as a percentage of the mass of the specimen

water content: specific measure of all or the total amount of water (free plus bound) in the test specimen and expressed as a percentage of the mass of the specimen taken for analysis (wet basis in this paper)

Math symbols

D (%): difference in water content between the aggregate botanical trash removed from cotton in a specific cleaning operation (w_i) and the cleaned cotton (w_c)

F_c: mass fraction of the cleaned cotton in the two component mixture of fibers and aggregate botanical trash in raw cotton

F_i: mass fraction of the aggregate botanical trash in the two component mixture of fibers and trash in raw cotton

M_b(g): mass of water in a raw cotton sample before mechanical cleaning

m_{sx}(g): sample weight of raw cotton sample before mechanical cleaning

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

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

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