

COLOR CHANGES VIA TREATMENTS

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Abstract

Cotton remains one of the most important natural fibers. Since cotton is produced in the field rather than at a manufacturing facility, it remains difficult to understand all chemical and physical properties that are affected by production, further processing, and utilization. Cotton color differences are not well understood chemically and the protective exterior complexity of raw cotton further entangles matters. This study will evaluate how cotton identified at the gin as sticky cotton may be affected by heat, acid catalysis, and water rinsing treatments to lower cotton stickiness. Cotton fiber physical deteriorations and discolorations are known to occur under these instances but little is understood in how the protective cuticle layer including pectins, waxes, and sugars affect fiber properties and future processing.

The cotton samples consisted of commercial upland varieties that were selected because of their wide range of stickiness levels. The various cottons consisted of commercial upland varieties from California and Syria, which were harvested, ginned, and baled by commercial methods. Several of these bales had previously been identified as potentially sticky and problematic cotton by the acid-base pH spray indicator test. Since the acid-base pH spray indicator is often not accurate in finding sticky cotton, these bales had been previously tested for stickiness on the minicard. The minicard is currently the best test for cotton stickiness. Sugar and other components of cotton's protective cuticle layer have diverse melting points, varying degrees of hydrolysis, and can cause a multitude of chemical reactions. Fiber samples were sorted and separated for various treatments that have the potential to affect this layer. To evaluate the removal effectiveness and fiber properties of treatments, each sample was prepared for testing that included atomizing samples with 10 % citric acid, rinsing samples with 40 °C deionized water, or preparing a control. To evaluate differences among treated and untreated cotton samples, these cotton samples were either unheated or heated at 133 °C with steam or dry heat for 1, 2, and 4 h. Following these treatments, samples were tested for sugar content and HVI fiber properties. Sugar was measured using the glucose oxidase enzyme method. Cotton quality measurements were performed on the HVI that allows cotton fibers to be tested for length, strength, fineness, color and trash.

This study evaluated steps that could be taken at the cotton gin or textile mill to adequately remove sugar from cotton. Heat treatments, rinsing, and acid catalysis likely all affect the surface components and cotton fibers themselves. The lack of these surface components likely decreases the lubrication and the ease of future processing. Citric acid catalysis, water rinsing, and additional heat (both dry and steam) appear to lower cotton sugar levels. Glucose levels decreased for unheated samples from 24.4 to 6.1 mg/l by heating samples at 133 °C with dry heat for 4 h. Combining all heat treatments and water rinsing lowered glucose levels from 18.5 mg/l to 5.0 mg/l. Fiber quality decreases with steam, dry heat, and acid catalysis. Combining all heat treatments, the control's fiber strength was 27.2 g/tex and samples treated with citric acid had a statistically lower strength of 26.1 g/tex while water rinsing had a statistically higher value of 29.6 g/tex. Water rinsing appears to be the most advantageous process to lower sugar levels while maintaining or improving fiber quality. No reliable relationship appears to currently exist with HVI results and cotton stickiness.

Introduction

Cotton remains one of the most important natural fibers. Since it is produced in the field rather than at a manufacturing facility, it remains complicated to understand all the chemical and physical properties, which are affected by production, further processing, and utilization. Cotton color differences are not well understood chemically. The protective exterior complexity of raw cotton further entangles matters. The low absorbency of raw cotton can be attributed to its waxy exterior, which contains pectic substances (Li and Hardin, 1998). This study will evaluate how cotton identified at the gin as sticky cotton may be affected by heat, acid catalysis, and water rinsing treatments to lower cotton stickiness. Heating is known to lower the endotoxin content of cottons while washing cotton is known to lower respirable dust (Rousselle et al., 1996; Rousselle and Domelsmith, 1993; and Rousselle and Chun, 1995). Cotton fiber physical deteriorations and discolorations are known to occur under these instances (Hessler and Workman, 1959; Sasser, 1980; Brushwood, 1988; Rousselle and Domelsmith, 1993; and Rousselle and Chun, 1995) but little is understood in how the protective cuticle layer including pectins, waxes, and sugars affect fiber properties and future processing. Color and fiber quality results may indicate how the fibers respond following heat, acid, and water treatments and the importance of wax, sugar, or pectin levels.

Cotton color is likely influenced by components (wax, pectin, and sugar) found on the protective coating of all fibers. The United States Department of Agriculture (USDA) categorizes the color of various agricultural products. American Upland cotton has 25 official color grades such as good middling, strict middling, middling, strict low middling, low middling, strict good ordinary, good ordinary, and below grade with 5 subcategories such as white, light spotted, spotted, tinged, and yellow stained (Agricultural Marketing Service, 1999). Blending equal portions of these color grades does not result in an intermediate color so understanding color is of vast importance in every aspect of the textile market. Historically, cotton fiber measurements were first performed by humans specially trained to differentiate fibers based on their length, strength, fineness, color, and trash (Shofner and Shofner, 2000). The USDA classes cotton and the Agricultural Marketing Service (AMS) grades cotton for a small fee (Agricultural Marketing Service, 1999). These cotton fiber measurements have progressed from a subjective human classer to the objective high volume instrument (HVI). A representative cotton sample tested on a properly maintained instrument and performed under similar lighting results in values that are impartial and non-biased.

The HVI provides a rapid color measurement at a low cost using a colorimeter at one set of conditions. A colorimeter measures differences in color by detecting the amount of light that is transmitted through colored filters that mimic human visual sensitivity to light. Measurements of Rd and +b are used to determine the intersection point and thus cotton color grade according to the cotton color chart (Agricultural Marketing Service, 1999). Perceived color cannot be directly measured but one can measure and calculate components that are responsible for producing this color. Today, the first textile measurements of color are performed on ginned cotton fibers sent to USDA AMS classing offices for grading and classification. While the HVI is able to provide vast quantities of valuable and insightful data, problems exist with any measurement technique. HVI and classer grade conflicts have occurred and the official grade has traditionally gone to the human classer (Xu et al., 1998b). Grading and classification aids in marketing cotton, so as processing speeds increase, continued improvements in measuring cotton properties are needed.

New techniques or instruments may be necessary to provide rapid, consistent, quantitative, and additional fiber property results with confirmed reliability. Every year in the United States, millions of cotton bales are classed on the HVI with these fiber properties recorded for marketing. Currently, HVI color measurements only include two portions of color space with the redness-greenness attributes disregarded in color grading (Xu et al., 1998a). Cotton color has been correlated to trash (Thomasson, 1993) and natural weathering (Iyer et al., 1995) with other relationships perhaps existing with recorded HVI fiber properties. Less tangible fiber quality correlations may be found using new techniques or instruments that measure pectin, sugar, wax, and metal concentrations, fiber convolutions, surface properties, cotton varieties, maturity levels, growing conditions, or fiber biodegradation.

Cotton stickiness is due to low molecular weight carbohydrates (sugars) located on the surface of cotton fibers. If present at certain levels, these sugars can cause processing difficulties in cotton gins and produce non-uniform webs, sliver, and yarn in textile mills. Processing problems are due to high quantities of sugar that build up on textile equipment and ultimately stop processing. These sugars are derived from plant and insect (primarily cotton aphids, *Aphis gossypii*, and silver leaf whiteflies, *Bemisia argentifoli*) sugars. Plant sugars do not usually cause problems because the sugars are in low levels evenly distributed on the fibers (Perkins, 1971). Insect (honeydew) sugars are very sticky and randomly distributed on the fiber (Brushwood, 2002).

High performance liquid chromatography, HPLC, is a slow research type instrument used to assess these types of sugar contaminations (Gamble, 2001). Many other tests and instruments have been exclusively developed to rapidly assess the stickiness of cotton fibers (Hector and Hodkinson, 1989). A non-exhaustive list of cotton stickiness tests include the acid-base pH spray indicator (Brushwood and Perkins, 1993); potassium ferricyanide method (Perkins, 1971); Benedict test (Brushwood and Perkins, 1993); thermodetector (Perkins and Brushwood, 1993); minicard (Brushwood and Perkins, 1993); enzyme-based method (Gamble, 2001); fiber contamination tester, FCT (Ethridge and Hequet, 1999); high speed stickiness detector, H2SD (Ethridge and Hequet, 1999); Elsner's heating method (Elsner, 1982a and Milnera et al., 1984), Shenkar tester (Hector and Hodkinson, 1989) and near infrared spectral analysis (Brushwood and Han, 2000). Sticky cotton is difficult to measure with scientists currently unable to arrive at an infallible analysis for detection. Because sugar is difficult to detect, expensive and laborious attempts have been made to reduce cotton stickiness in textile processing through fiber blending, additives, storage, microorganisms, and fiber washing (Hector and Hodkinson, 1989).

The surface of a cotton fiber contains pectic substances that are amorphous carbohydrates with a low degree of polymerization that may be extracted with hot water or mild acids (Fennema, 1985). Processing effects of pectic substances and the response of these substances to heat is unknown. Wax on cotton fibers is well known to lubricate and improve spinning (Varadarajan et al., 1990). Waxes are sparingly soluble in water and at elevated temperatures undergo complex chemical reactions at various melting points (Fennema, 1985). Compared to cellulose, sugars hydrolyze relatively easily depending upon its size, configuration, pH, and temperature that lead to color changes (Fennema, 1985). Furthermore, if thermal degradation and dehydration reactions occur, odors and colors are created by the generation of 2-furaldehyde, 5-

hydroxymethyl-2-furaldehyde (HMF), 2-hydroxyacetyl-furan, isomaltol, levulinic acid, formic acid, acetol, acetoin, diacetyl, lactic acid, pyruvic acid, and acetic acid (Fennema, 1985). These reactions may introduce double bonds into sugar rings forming conjugated double bonds that absorb light and produce color (Fennema, 1985). Other constituents found on cotton fibers may interact in these chemical reactions depending upon variables such as temperature, pH, moisture content, presence or absence of metal ions, and effect of sugar structure. Little is known in how wax, pectic substance, and sugar chemical reactions respond to heat, acid catalysis, and water rinsing nor in how they affect fiber properties. Effectiveness of these treatments on predicting the quantity of sugar on cotton fibers, the fiber property affect of sugar removal treatments, and potential processing improvements of sticky cotton will be evaluated.

Materials and Methods

Sample bales were selected because of their wide range of stickiness levels, with some cotton bales considered typical with no stickiness potential. These bales are not representative of a typical harvest at any one location but rather potential harvests. Bales were all harvested, ginned, and baled by commercial methods. The various cottons consisted of commercial upland varieties from California and Syria. Several of these bales had previously been identified as potentially sticky and problematic cotton by the acid-base pH spray indicator test. Since the acid-base pH spray indicator is often not accurate in finding sticky cotton, these bales had been previously tested for stickiness on the minicard by the procedure outlined by Brushwood and Perkins (1993). Two non-sticky cottons were the control with a negative response to the acid-base pH spray indicator and minicard while 7 cottons were potentially sticky only according to the acid-base pH spray indicator and 4 cottons were sticky according to the acid-base pH spray indicator and minicard.

These bales of cotton were divided into 50 g aliquots for testing. Fiber samples were sorted and separated for various treatments that have the potential to affect the protective cuticle layer including pectins, waxes, and sugars. The components of this protective cuticle layer have diverse melting points, varying degrees of hydrolysis, and can cause a multitude of chemical reactions. To evaluate the removal effectiveness and fiber properties of treatments, each sample was prepared for testing which included atomizing samples with 10 % citric acid, rinsing samples with 40 °C deionized water, or preparing a control. An acid catalysis treatment of 10 % citric acid v/v (Aldrich Chemical Company, Milwaukee, WI) plus 0.5 % v/v Triton-X wetting agent (Mallinckrodt Baker, Paris, KY) in water was atomized onto cotton fibers. This process atomized 0.05 g of citric solution per 1 g of fiber. The rinsing treatment involved heating deionized water to 40 °C and mixing this in a 14:1 ratio (w/w) with cotton. To evaluate differences among treated and untreated cotton samples, these cotton samples were either unheated or heated at 133 °C with steam or dry heat for 1, 2, and 4 h. Dry heat treatments were performed by placing cotton fiber samples in a 1500E VWR convection oven (VWR International, West Chester, PA) at 133 °C for 1, 2, and 4 h. Steam heat treatments at elevated pressures were completed by exposing cotton fiber bundles to 133 °C and 3 atm for 1, 2, and 4 h in a HL2020-38-SCS-001 Hotpack Autoclave (SP Industries, Philadelphia, PA). Prior to further testing all cotton samples were dried and conditioned for at least 96 hours at 65 % RH and 21 °C (ASTM, 1997d).

Following these treatments, samples were tested for sugar content and HVI fiber properties. Sugar was measured using the glucose oxidase enzyme method, which is an environmentally friendly test for insect sugar contamination (Gamble, 2001). The enzyme and glucose cotton extract react to produce hydrogen peroxide that is oxidized on a platinum anode producing a current proportional to the substrate level (David Brune, personal communication, 1992). One measurement of glucose is performed on the cotton extract with the second test performed on the same extract subjected to acid hydrolysis, which cleaves oligosaccharides present in the extract (Gamble, 2001). Cotton quality measurements were performed on the HVI (Zellweger Uster, Knoxville, TN) by the Testing Laboratory at CQRS. The HVI allows cotton fibers to be tested for length, strength, fineness, color and trash according to established standards (ASTM, 1993). The data were statistically analyzed with the General Linear Models procedure in SAS using Duncan's New Multiple Range Test ($P < 0.05$) to detect differences between means (SAS Institute Inc., 1985).

Results and Discussion

Many individuals believe that as the color of cotton changes, the processability of the fibers decreases. (Hessler and Workman, 1959) have reported that over-dried cotton has structural changes, which is observed through a decrease in moisture regain. This deterioration could be related to the surface properties of cotton. Mature cotton contains 93 to 96% cellulose on dry weight basis with the noncellulosic portion containing 1.3% protein, 0.9% pectic substances, 1.2% ash, 0.6% wax, 0.8% organic acids, 0.3% sugars and 0.9% other components (Perkins, 1971). Overheating and overdrying both cause discoloration and a reduction in fiber quality (Brushwood, 1990). Temperatures at 170 °C and above for prolonged periods has been shown to produce yellowing in pure cotton (Elsner, 1982b) which is likely in part due to cotton's decomposition temperature of 150 °C (Morton and Hearle, 1997). Heating cotton at 180 °C for 3 hours does not cause complete thermal degradation or significant change in the supramolecular structure of cellulose but this overheated cotton has a decreased fiber strength, length, and processability (Rousselle et al., 1996). A reduction in time (20 min) and temperature (93.3 °C) does not

significantly reduce fiber strength or increase discoloration (Rousselle et al., 1996). This is likely because high heat decreases the degree of polymerization (Hessler and Workman, 1959). All cottons in this study were ginned by commercial methods and exposed to temperatures that allow thermochemical reactions. Cotton surface properties were likely affected when some of these same cottons were again heated in this study. Upon exposure to heat at 133 °C, non-sticky and sugar contaminated cotton in this study appeared to follow these same trends with fiber quality properties decreasing with heat.

Combining removal steps, increasing times of heat exposure from 1 h to 2 h and 4 h resulted in decreasing trends for glucose, hydrolyzed glucose, %? glucose, reflectance, length, strength and uniformity while the yellowness increased (Table 1). There was no observable change in elongation. Fiber qualities were adversely affected by the heat, which may be related to the removal of the surface properties. The surface of cotton contains a sugar, pectin, and wax concoction that has unique properties likely affected by heat. Typical sugars found on cotton have a melting point that can range from 102 °C for pure maltose up to 180 °C for pure sucrose (Hodgman et al., 1961). Some observed cotton color changes are likely due to various sugar thermal degradation and dehydration reactions. Color changes could also be related to wax level differences between cottons. Wax found on cotton is an organic mixture of higher fatty alcohols, fatty acids, esters, and higher hydrocarbons having a melting point from 68 to 80 °C (Varadarajan et al., 1990). Color changes could also be due to the loss of pectin from the surface or additional pectin reactions (Gamble 2001, personal communication). As expected for sugars undergoing thermal degradation and dehydration reactions, the measured glucose levels and reflectance decreased with heating and yellowness increased. These results agree with work performed by Elsner (1982a) who found that cotton reducing sugar levels decreased by heating samples at 130 °C for 3 hours with the color conversely increasing (Elsner, 1982a).

In addition to dry heat, cotton samples were heated in an autoclave with pressure and steam for the same times. Heated cotton fibers are reported to be more friable than unheated fibers in processing (Rousselle and Chun, 1995) but little is known in how autoclaving will affect fibers. Previous work has shown that heating cotton samples with increased moisture resulted in fibers with higher fiber tenacity and elongation (Rousselle and Domelsmith, 1993). Combining removal steps, autoclave heating further decreased glucose, hydrolyzed glucose, %? glucose, reflectance, length, strength and uniformity while the yellowness increased. These fiber property decreases with autoclaving is likely due to the nearly 9-fold internal energy increase from steam and pressure used in autoclaving. The elongation of these fibers was not statistically different between heat treatments. The color results agree with work done on textiles where steam has previously been shown to be an economical wool process that causes hydrothermal yellowing (Schwartz and McKinnon, 2000). Heating cotton in an autoclave for extended periods at 133 °C deteriorates quality by affecting the cotton fiber, its sugar, pectin, and wax surface concoction, color, and likely processing efficiency.

Combining removal steps and heat treatments, and separating the cotton samples into not sticky (control), potentially sticky (acid-base pH spray indicator), and sticky (acid-base spray indicator and minicard) indicates that the glucose levels are indeed higher for sticky cotton with no statistical differences between not sticky and potentially sticky (Table 2). The yellowness is statistically higher for sticky cotton. Sugars interacting with components on cotton fibers have been termed “caramelanoidation” reactions that result in an intense reddish-brown caramel melanoidin color that could be used for a quantitative estimate of the sugar content (Elsner, 1982b). The acid-base pH spray is only an indicator and does not truly differentiate cotton stickiness as seen in these results. The minicard is the backbone of stickiness indicator tests. The cotton samples in this study were thus divided into their 3 respective minicard readings with glucose and HVI properties evaluated (Table 3). Glucose, hydrolyzed glucose, %? glucose, and yellowness increased with the stickiness while reflectance decreased. Glucose and yellowness measurements were statistically different between no stickiness readings and other sticky minicard readings. Gin drying and further heat treatments complicate estimating sugar content from simple HVI color. Measuring glucose levels by the enzyme method resulted in a linear relationship between glucose and hydrolyzed glucose measurements ($R^2=0.52$) indicating that stickiness could be estimated using the glucose enzyme method.

Washing removes and distributes sugars uniformly on cotton fibers but requires additional handling and drying that can cause excessive damage to fibers. Washing and acid catalysis both affected cotton’s protective coating, likely, allowing fibers to swell more under identical relative humidity conditions. This washing and acid catalysis alters the physical and chemical make-up of cotton. During this process the fibers surface properties are likely altered potentially influencing crimp, convolutions, and traditional fiber surfaces. The stickiness removal technique water rinsing was the preferred process over acid catalysis. Acid catalysis did little to affect the glucose levels found on the cotton samples while the length and strength were both statistically lower than the untreated cotton (Table 4). Rinsing cotton increased the length and strength of cotton and statistically removed the largest portion of glucose from the cotton samples. Thermogravimetric analysis (TGA) has shown that washed cotton contain 50% less residue than unwashed cottons (Ward et al., 1985). USDA AMS does not report elongation of cotton fibers and no differences were found in the elongation of these fibers with various heat or surface removal treatments. Cotton contains magnesium up 0.1% and potassium up 0.7%, which are both removed in washing (Ward et al., 1985). Removal of these compounds along with pectin, wax, and sugar affects processing with Rousselle et al. (1996) indicating that ring-spun washed cotton is more difficult than processing unwashed cotton.

There appears to be little effect of citric acid catalysis on the color and glucose levels of cotton samples tested (Tables 5 and 6). Citric acid catalysis does appear to decrease the strength and length and more so as the temperature increases. This catalysis adversely affects the fiber qualities and does little to lower sugar levels. More drastic sugar removal could be possible with increased levels of acid applied in processing but this would likely further decrease the strength of cotton fiber to lower levels. Other acids may provide more desirable sugar removal results than citric acid. While costly and time consuming, rinsing cotton with water would appear to be the preferred method over acid catalysis to remove sugar from contaminated cottons and actually improve HVI fiber properties. Water rinsing produced fibers that had smaller fiber quality differences between no heat and long-term heat exposures. For color results this likely indicates that the sugar, pectin, and wax on the surface of cotton was successfully removed with less compounds available to cause color changes. It appears that additional research is required in understanding the affects and interactions between fiber properties, processing, and fiber surface compounds including sugar, pectin, and wax. Successful sugar removal processes with various heat treatments appear to decrease glucose levels from unheated cotton samples. Additional heating could reduce stickiness but water rinsing would again be the preferred method because of the fiber quality reduction. Heating cotton under dry and steam conditions both appear to affect the fibers negatively with increases in yellowness and decreases in reflectance, length, uniformity and strength.

Conclusions

Heat treatments, rinsing, and acid catalysis likely all affect the surface components of cotton fibers. The lack of these surface components likely decreases the lubrication and the ease of future processing. This study evaluated steps that could be taken at the cotton gin or textile mill to adequately remove sugar from cotton. Citric acid catalysis, water rinsing, and additional heat (both dry and steam) appear to lower cotton sugar levels. Fiber quality decreases with steam, dry heat, and acid catalysis. Water rinsing appears to be the most advantageous process to lower sugar levels while maintaining or improving fiber quality. No reliable relationship appears to currently exist with HVI results and cotton stickiness. Further studies are required to determine how cellulose, pectin, wax, and sugar substances cause color changes, and how moisture interacts with these components. The acid-base pH spray is only an indicator and does not truly differentiate cotton stickiness as seen in these results. Other studies may identify substances or reactions that occur on the cotton surface and their affect on fiber quality properties.

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Table 1. The influence of heat treatments on fiber qualities and sugar levels.

Heat ¹	Glucose ²	Hydrolyzed Glucose ³	%? Glucose ⁴	Reflectance ⁵	Yellowness	Length	Strength	Uniform.
	(mg/l)	(mg/l)	(%)	(Rd)	(+b)	(mm)	(g/tex)	(%)
0	24.4 a*	75.5 a	0.10 a	73 a	11.8 a	28.4 a	31.5 a	83.0 a
1	12.7 b	58.5 b	0.09 a	71 b	13.2 b	28.4 a,b	30.2 b	82.6 a,b
2	12.2 b	62.1 a,b	0.10 a	68 c	14.6 c	28.2 a,b	29.6 b	82.6 a,b
3	6.1 c	55.6 b	0.10 a	65 d	15.3 d	28.2 a,b	28.2 c	82.3 b,c
4	14.2 b	66.0 a,b	0.10 a	54 e	15.7 d	27.7 c	25.7 d	82.2 c,d
5	16.0 b	61.7 a,b	0.09 a	52 f	16.4 e	27.7 c	24.7 e	81.9 d
6	14.6 b	62.7 a,b	0.10 a	51 g	16.7 e	27.4 c	23.4 f	81.4 e

* Values followed by different letters within columns are significantly different, P<0.05, according to Duncan's New Multiple Range Test.

¹ Heat treatments include: 0-no heat; 1-oven at 133 °C for 1 h; 2-oven at 133 °C for 2 h; 3-oven at 133 °C for 4 h; 4-autoclave at 133 °C for 1 h; 5-autoclave at 133 °C for 2 h; and 6-autoclave at 133 °C for 4 h.

² Glucose value is the concentration of extract from 1 g of cotton and 20 ml of deionized water.

³ Hydrolyzed glucose value is the concentration of extract from 1 g of cotton and 20 ml of deionized water hydrolyzed with HCl at 95 °C for 2 h.

⁴ The percentage difference of glucose and hydrolyzed glucose found on cotton fibers is defined as %? Glucose (Gamble, 2001).

⁵ Fiber quality HVI measurements performed according to ASTM standards (1993).

Table 2. Stickiness and color response of non-sticky, potentially sticky, and sticky cotton.

Stickiness ¹	Glucose ²	Hydrolyzed Glucose ³	%? Glucose ⁴	Reflectance ⁵	Yellowness
	(mg/l)	(mg/l)	(%)	(Rd)	(+b)
Not sticky	12.7 a*	68.2 a	0.11 a	63 a	14.5 a
Potentially sticky	11.6 a	52.3 b	0.08 b	62 b	14.6 a
Sticky	19.8 b	79.6 c	0.12 a	61 b	15.4 b

* Values followed by different letters within columns are significantly different, P<0.05, according to Duncan's New Multiple Range Test.

¹ Stickiness levels include a.) a control cotton with no known stickiness problems as evaluated using the acid-base pH spray and minicard tests; b.) a potentially sticky cotton according to the acid-base pH spray; and c.) a sticky cotton with a positive response to the acid-base pH spray indicator and minicard test.

² Glucose value is the concentration of extract from 1 g of cotton and 20 ml of deionized water.

³ Hydrolyzed glucose value is the concentration of extract from 1 g of cotton and 20 ml of deionized water hydrolyzed with HCl at 95 °C for 2 h.

⁴ The percentage difference of glucose and hydrolyzed glucose found on cotton fibers is defined as %? Glucose.

⁵ Fiber quality HVI measurements performed according to ASTM standards (1993).

Table 3. The impact of 3 levels of sticky cotton on sugar and color measurements.

Minicard reading	Glucose ²	Hydrolyzed Glucose ³	%? Glucose ⁴	Reflectance ⁵	Yellowness
	(mg/l)	(mg/l)	(%)	(Rd)	(+b)
0	11.8 a*	55.8 a	0.09 a	62 a	14.6 a
1	17.5 b	77.6 b	0.12 b	61 a	15.3 b
2	20.5 b	80.2 b	0.12 b	61 a	15.4 b

* Values followed by different letters within columns are significantly different, P<0.05, according to Duncan's New Multiple Range Test.

¹ Minicard readings include: (0) no stickiness; (1) light stickiness; (2) moderate stickiness according to Brushwood and Perkins (1993).

² Glucose value is the concentration of extract from 1 g of cotton and 20 ml of deionized water.

³ Hydrolyzed glucose value is the concentration of extract from 1 g of cotton and 20 ml of deionized water hydrolyzed with HCl at 95 °C for 2 h.

⁴ The percentage difference of glucose and hydrolyzed glucose found on cotton fibers is defined as %? Glucose.

⁵ Fiber quality HVI measurements performed according to ASTM standards (1993).

Table 4. Stickiness and fiber quality response of treated cotton.

Removal step ¹	Glucose ² (mg/l)	Hydrolyzed Glucose ³ (mg/l)	%? Glucose ⁴ (%)	Reflectance ⁵ (Rd)	Yellowness (+b)	Length (mm)	Strength (g/tex)	Uniform. (%)
Control	18.5 a*	75.6 a	0.11 a	61 b	15.4 a	27.9 a	27.2 a	82.3 a,b
Acid	19.4 a	73.3 a	0.11 a	60 b	15.7 a	27.7 b	26.1 b	82.1 b
Rinse	5.0 b	40.4 b	0.07 b	65 a	13.3 b	28.4 c	29.6 c	82.4 a

* Values followed by different letters within columns are significantly different, P<0.05, according to Duncan's New Multiple Range Test.

¹ Removal treatments include a.) preparing a control; b.) atomizing samples with 10 % citric acid; or c.) rinsing samples with 40 °C deionized water.

² Glucose value is the concentration of extract from 1 g of cotton and 20 ml of deionized water.

³ Hydrolyzed glucose value is the concentration of extract from 1 g of cotton and 20 ml of deionized water hydrolyzed with HCl at 95 °C for 2 h.

⁴ The percentage difference of glucose and hydrolyzed glucose found on cotton fibers is defined as %? Glucose.

⁵ Fiber quality HVI measurements performed according to ASTM standards (1993).

Table 5. The relationships of HVI properties to surface removal and heat treatments.

Heat ¹	Treatment ²	Reflectance ³ (Rd)	Yellowness (+b)	length (mm)	Uniformity (%)	Strength (g/tex)
Control						
0	None	71 bcdefg	12.3 pqrstu	27.2 ijklmnop	80.3 opqrs	27.0 ijklmnopq
1	None	73 abcde	12.1 rstu	26.4 lmnopqr	81.2 ijklmnopqr	25.1 lmnopqrst
2	None	69 defghi	15.1 ghijkl	26.7 jklmnopqr	81.0 jklmnopqrs	25.6 klmnopqr
3	None	61 lmno	16.5 abcdefg	26.9 jklmnopq	80.5 lmnopqrs	24.4 opqrst
4	None	55 pq	15.9 bcdefghij	26.4 lmnopqr	79.7 rst	22.9 rstuv
5	None	51 qr	16.8 abcdefg	25.5 r	79.7 rst	21.0 vwxy
6	None	50 r	17.6 ab	26.4 lmnopqr	79.8 qrst	21.0 vwxy
0	Citric acid	77 a	11.1 u	26.8 jklmnopq	80.8 klmnopqrs	26.7 ijklmnopq
1	Citric acid	72 abcdefg	13.9 klmnopqr	26.5 lmnopqr	81.8 efghijklmno	25.3 klmnopqrst
2	Citric acid	70 cdefghi	15.3 fghijkl	26.9 jklmnopq	81.2 ijklmnopqr	25.1 mnopqrst
3	Citric acid	64 jklm	15.6 cdefghijk	26.0 pqr	80.9 klmnopqrs	24.8 nopqrst
4	Citric acid	57 nop	14.0 jklmnopq	26.3 mnopqr	80.8 klmnopqrs	24.6 opqrst
5	Citric acid	50 qr	17.7 ab	25.5 r	80.0 pqrs	19.7 wxy
6	Citric acid	48 r	17.8 ab	25.5 r	78.4 t	19.5 xy
0	Water rinse	70 cdefgh	12.2 pqrstu	27.3 ijklmno	81.5 hijklmnopq	28.1 efghijklmno
1	Water rinse	73 abcde	11.9 tu	26.4 lmnopqr	80.8 klmnopqrs	26.4 ijklmnopq
2	Water rinse	72 abcdef	12.2 pqrstu	26.2 opqr	81.3 ijklmnopqr	25.9 jklmnopqr
3	Water rinse	69 defghi	13.0 nopqrstu	26.4 lmnopqr	80.2 opqrs	25.8 jklmnopqr
4	Water rinse	57 op	15.2 fghijkl	26.3 mnopqr	80.7 lmnopqrs	23.6 qrstuv
5	Water rinse	59 mnop	14.6 hijklmn	26.3 mnopqr	80.4 nopqrs	24.2 pqrstu
6	Water rinse	61 lmno	14.2 jklmno	26.7 jklmnopqr	80.8 klmnopqrs	24.7 nopqrst
Potentially sticky						
0	None	75 ab	11.1 u	29.2 ab	83.8 ab	33.4 ab
1	None	72 abcdef	13.0 nopqrstu	29.0 abcd	83.4 abcd	32.5 abc
2	None	68 efghij	15.2 fghijkl	29.1 abc	83.8 abc	31.1 abcde
3	None	68 efghij	14.9 ghijklm	29.1 abcd	83.9 ab	30.8 bcdefg
4	None	51 qr	16.6 abcdefg	28.6 abcdefgh	83.2 abcdefg	25.5 klmnopqrs
5	None	50 qr	17.3 abcd	28.7 abcdefg	82.6 abcdefghij	25.2 lmnopqrst
6	None	50 r	17.4 abc	28.6 abcdefgh	83.0 abcdefgh	25.6 klmnopqr
0	Citric acid	74 abcd	11.2 u	29.1 abc	84.2 a	33.1 ab
1	Citric acid	72 abcdef	13.1 mnopqrst	29.1 abc	83.8 ab	31.7 abcd
2	Citric acid	66 ghij	15.8 bcdefghij	28.8 abcdefg	83.3 abcdef	30.6 bcdefgh
3	Citric acid	62 klmn	16.4 abcdefgh	28.9 abcde	83.5 abcd	28.6 defghijk
4	Citric acid	51 qr	17.0 abcdef	28.3 bcdefghi	83.2 abcdefg	25.2 klmnopqrst
5	Citric acid	49 r	17.7 ab	28.4 bcdefghi	82.7 abcdefghi	24.4 opqrst
6	Citric acid	47 r	17.9 a	27.3 ijklmno	81.4 hijklmnopq	20.9 vwxy
0	Water rinse	70 bcdefghi	12.3 opqrstu	29.7 a	84.1 a	34.3 a
1	Water rinse	70 bcdefgh	12.0 stu	29.4 ab	83.4 abcde	33.2 ab
2	Water rinse	70 bcdefghi	12.2 pqrstu	29.5 ab	83.4 abcde	33.0 ab
3	Water rinse	66 ghijkl	13.5 mnopqrst	29.0 abcd	83.2 abcdefg	30.8 bcdefg
4	Water rinse	61 lmno	13.1 mnopqrst	29.0 abcd	83.6 abcd	31.1 abcdef
5	Water rinse	58 nop	14.0 jklmnop	29.0 abcd	83.3 abcde	30.4 bcdefgh
6	Water rinse	56 op	14.4 ijklmn	28.9 abcdef	83.1 abcdefg	29.1 defghij
Sticky						
0	None	75 abc	12.1 qrstu	27.7 efghijkl	82.0 defghijklmno	29.5 cdefghi
1	None	69 cdefghi	15.3 fghijkl	27.7 efghijkl	81.6 fghijklmnop	28.5 defghijkl
2	None	68 efghij	15.5 defghijk	27.4 hijklmno	82.4 bcdefghijk	28.2 efghijklm
3	None	59 mnop	17.2 abcde	27.6 ghijkl	81.7 fghijklmno	26.1 ijklmnopqr
4	None	50 r	17.6 ab	26.2 nopqr	81.0 ijklmnopqrs	22.0 tuvwx
5	None	49 r	17.6 ab	27.0 jklmnopq	80.9 klmnopqrs	22.2 stuvw
6	None	47 r	18.0 a	27.0 jklmnopq	80.4 mnopqrs	20.9 vwxy
0	Citric acid	75 ab	12.1 rstu	27.6 fghijkl	82.2 cdefghijkl	29.3 cdefghi
1	Citric acid	70 cdefghi	15.2 fghijkl	27.6 fghijkl	82.1 defghijklmno	27.8 fghijklmno
2	Citric acid	65 ijkl	16.8 abcdefg	27.4 hijklmno	81.7 fghijklmnop	27.5 ghijklmnop
3	Citric acid	65 hijkl	16.2 abcdefghi	27.2 ijklmnop	80.8 klmnopqrs	26.6 ijklmnopq
4	Citric acid	49 r	17.7 ab	26.6 klmnopqr	80.2 opqrs	21.2 uvwxy
5	Citric acid	48 r	17.9 a	26.6 klmnopqr	80.6 lmnopqrs	20.4 vwxy
6	Citric acid	46 r	18.0 a	25.9 qr	79.4 st	18.0 y
0	Water rinse	70 bcdefghi	12.8 nopqrstu	27.9 cdefghij	82.1 defghijklm	31.3 abcde
1	Water rinse	71 bcdefg	12.3 opqrstu	27.9 defghijk	81.4 hijklmnopq	29.0 defghij
2	Water rinse	71 bcdefg	12.8 nopqrstu	27.6 ghijklm	81.7 fghijklmnop	28.4 defghijklm
3	Water rinse	67 fghijk	14.5 hijklmn	27.5 hijklmn	81.1 ijklmnopqr	27.8 ghijklmno
4	Water rinse	60 mnop	13.9 klmnopqrs	27.7 efghijkl	81.5 hijklmnop	27.4 hijklmnop
5	Water rinse	57 nop	14.4 ijklmn	27.5 hijklmn	81.6 ghijklmnop	25.9 jklmnopq
6	Water rinse	55 pq	15.4 efghijk	27.4 hijklmno	80.8 klmnopqrs	25.1 mnopqrst

* Values followed by different letters within columns are significantly different, P<0.05, according to Duncan's New Multiple Range Test.

¹ Heat treatments include: 0-no heat; 1-oven at 133 °C for 1 h; 2-oven at 133 °C for 2 h; 3-oven at 133 °C for 4 h; 4-autoclave at 133 °C for 1 h; 5-autoclave at 133 °C for 2 h; and 6-autoclave at 133 °C for 4 h.

² Removal treatments include preparing a control, atomizing samples with 10 % citric acid, or rinsing samples with 40 °C deionized water.

³ Fiber quality HVI measurements performed according to ASTM standards (1993).

Table 6. The relationships of sugar measurements to surface removal and heat treatments.

Heat ¹	Treatment ²	Glucose ³ (mg/l)	Hydrolyzed Glucose ⁴ (mg/l)	% ? Glucose ⁵ (g glucose /g cotton)
Control				
0	None	40.20 abc	114.83 abc	0.14 abcdef
1	None	14.15 ghijklmno	78.23 bcdefghij	0.12 abcdefghi
2	None	17.68 ghijklmno	75.98 bcdefghijk	0.11 abcdefghi
3	None	3.78 klmno	48.98 defghijk	0.09 abcdefghi
4	None	18.05 ghijklmno	62.93 cdefghijk	0.08 bcdefghi
5	None	16.60 ghijklmno	70.87 bcdefghijk	0.10 abcdefghi
6	None	10.00 ghijklmno	69.83 bcdefghijk	0.11 abcdefghi
0	Citric acid	30.20 abcdefgh	92.33 abcdefg	0.12 abcdefghi
1	Citric acid	23.00 cdefghijkl	98.85 abcdef	0.15 abcdef
2	Citric acid	21.68 cdefghijklm	68.25 bcdefghijk	0.09 abcdefghi
3	Citric acid	2.15 lmno	54.90 cdefghijk	0.10 abcdefghi
4	Citric acid	21.63 cdefghiklm	106.88 abcde	0.17 abcd
5	Citric acid	25.35 cdefghij	82.88 bcdefghi	0.11 abcdefghi
6	Citric acid	7.05 ijklmno	63.23 cdefghijk	0.11 abcdefghi
0	Water rinse	4.83 jklmno	72.23 bcdefghijk	0.13 abcdefgh
1	Water rinse	0.00 o	91.13 abcdefghi	0.18 abc
2	Water rinse	1.45 mno	32.40 ghijk	0.06 defghi
3	Water rinse	0.16 on	11.73 k	0.02 I
4	Water rinse	1.45 mno	38.03 fghijk	0.07 cdefghi
5	Water rinse	1.40 mno	60.13 cdefghijk	0.11 abcdefghi
6	Water rinse	5.44 jklmno	38.18 fghijk	0.06 defghi
Potentially sticky				
0	None	26.96 bcdefghi	69.15 bcdefghijk	0.08 bcdefghi
1	None	13.61 ghijklmno	58.29 cdefghijk	0.08 bcdefghi
2	None	3.87 klmno	57.49 cdefghijk	0.010 abcdefghi
3	None	3.48 klmno	60.49 cdefghijk	0.11 abcdefghi
4	None	15.50 ghijklmno	64.01 cdefghijk	0.09 abcdefghi
5	None	14.21 ghijklmno	47.74 efg hijk	0.06 defghi
6	None	16.33 ghijklmno	59.72 cedfghijk	0.08 bcdefghi
0	Citric acid	27.39 bcdefghi	66.73 bcdefghijk	0.07 bcdefghi
1	Citric acid	13.00 ghijklmno	59.19 cdefghijk	0.09 abcdefghi
2	Citric acid	14.40 ghijklmno	47.85 efg hijk	0.06 defghi
3	Citric acid	4.51 jklmno	52.82 cdefghijk	0.09 abcdefghi
4	Citric acid	17.96 ghijklmno	75.43 bcdefghijk	0.11 abcdefghi
5	Citric acid	11.92 ghijklmno	53.22 cdefghijk	0.08 bcdefghi
6	Citric acid	11.12 ghijklmno	48.36 efg hijk	0.07 bcdefghi
0	Water rinse	4.35 klmno	27.93 hijk	0.04 fghi
1	Water rinse	1.82 mno	13.69 jk	0.02 hi
2	Water rinse	10.85 abcdefghijklmno	47.8 efg hijk	0.07 bcdefghi
3	Water rinse	1.82 mno	37.37 fghijk	0.07 cdefghi
4	Water rinse	10.22 ghijklmno	59.49 cdefghijk	0.09 abcdefghi
5	Water rinse	9.45 ijklmno	38.49 fghijk	0.05 efg hi
6	Water rinse	11.00 ghijklmno	52.90 cdefghijk	0.08 bcdefghi
Sticky				
0	None	48.40 a	148.58 a	0.20 a
1	None	30.60 abcdef	109.69 abcd	0.15 abcdef
2	None	18.42 ghijklmno	110.66 abcd	0.18 ab
3	None	21.37 cdefghijklm	106.08 abcde	0.16 abcde
4	None	21.00 cdefghijklmno	57.60 cdefghijk	0.07 cdefghi
5	None	32.98 abcd	94.84 abcdefg	0.12 abcdefghi
6	None	23.17 cdefghijk	90.79 abcdefghi	0.13 abcdefg
0	Citric acid	46.00 ab	130.69 ab	0.16 abcde
1	Citric acid	21.17 cdefghijklmno	59.40 defghijk	0.07 bcdefghi
2	Citric acid	27.58 bcdefghi	113.18 abcd	0.17 abcd
3	Citric acid	14.46 ghijklmno	85.35 bcdefghi	0.14 abcdefg
4	Citric acid	21.70 cdefghijklm	76.80 bcdefghij	0.11 abcdefghi
5	Citric acid	37.50 abcd	102.26 abcdef	0.12 abcdefghi
6	Citric acid	31.68 abcdef	110.93 abcd	0.15 abcdef
0	Water rinse	3.58 klmno	30.60 ghijk	0.05 fghi
1	Water rinse	3.70 klmno	37.18 fghijk	0.06 defghi
2	Water rinse	1.42 mno	25.63 ijk	0.04 ghi
3	Water rinse	0.00 o	16.69 jk	0.03 ghi
4	Water rinse	0.19 on	55.35 cdefghijk	0.11 abcdefghi
5	Water rinse	1.27 mno	53.46 cdefghijk	0.10 abcdefghi
6	Water rinse	9.21 ijklmno	42.71 efg hijk	0.06 defghi

* Values followed by different letters within columns are significantly different, P<0.05, according to Duncan's New Multiple Range Test.

¹ Heat treatments include: 0-no heat; 1-oven at 133 °C for 1 h; 2-oven at 133 °C for 2 h; 3-oven at 133 °C for 4 h; 4-autoclave at 133 °C for 1 h; 5-autoclave at 133 °C for 2 h; and 6-autoclave at 133 °C for 4 h.

² Treatments included atomizing samples with 10 % citric acid, rinsing samples with 40 °C deionized water, or preparing a control.

³ Glucose is the concentration of extract from 1 g of cotton and 20 ml of deionized water.

⁴ Hydrolyzed glucose is the concentration of extract from 1 g of cotton and 20 ml of deionized water hydrolyzed with HCl at 95 °C for 2 h.

⁵ The difference between glucose and hydrolyzed glucose is defined as %? Glucose.