RELATIONSHIP OF INDIVIDUAL HONEYDEW SUGAR CONCENTRATIONS ON COTTON LINT STICKINESS POTENTIAL AND MEASURED SUGAR CONTENT Donald E. Brushwood USDA, ARS, Cotton Quality Research Station Clemson, SC

Abstract

Cotton fiber stickiness caused by the presence of sugars can not only depend upon the total amount sugars, but which specific ones are present. Studies were conducted to measure the stickiness potential of seven sugars commonly found in honeydew cottons. A single upland cotton was treated with pre-determined amounts of these sugars by misting with an air brush applicator. Resultant treatments were analyzed for sugar content to determine actual sugar retention. Stickiness measurements were conducted on rotary blended and hand blended treatment samples to determine degrees of stickiness at five different sugar concentrations. The characteristic honeydew sugars trehalulose and melezitose as well as the disaccharides turanose and sucrose were found to exhibit higher stickiness potentials than the other tested sugars on the thermodetector, especially at concentrations above 0.5%. Sucrose treated cottons did not get high ratings on the minicard test as observed for the same sugar on the thermodetector.

Introduction

Plant sugars on the surface of cottons are part of the natural growing process. Lint stickiness often experienced during textile processing usually occurs when concentrations of these sugars exceed normal levels. Plant sugars, although evenly distributed on the lint, can accumulate on card crush rolls, spindles, and other machinery that comes in direct contact with the cotton (Perkins, 1991; Perkins, 1993). Sugar extracts from non-insect contaminated cottons usually contain (except in the case of severe weathering and/or microbial damage) at least 10 identifiable different carbohydrates. The four most prevalent individual carbohydrates in plant sugars are the monosaccharides glucose and fructose and two disaccharides sucrose and trehalose. Usual orders of concentration are glucose, fructose, sucrose, and trehalose (Brushwood and Perkins, 1996; Brushwood, 1997). Ratios depend upon a number of factors such as cotton boll maturity, growing and harvesting conditions, area of growth, and variety.

Another source of sugars on harvested cottons is from insect con-tamination such as the sweet potato whitefly (Bemisa tabaci) and cotton aphid (Aphis Gossyii). Contamination is commonly found on cottons grown in areas where factors such as weather and other conditions conducive to these insects exist. Each insect processes large quantities of plant phloem sap during feeding activities. The excess phloem is excreted in the form of highly concentrated honeydew droplets that fall on the open bolls of cotton (Talpay, 1983). Without vigilant monitoring and control of whitefly and aphid populations during cotton growing seasons, the potential for honeydew contamination is high. Honeydew consists of highly concentrated extremely sticky carbohydrates randomly deposited on the surface of cotton lint.

Heavy honeydew contamination on cottons can have very devastating effects in all phases of textile processing. Deposits on rolls, blades and other equipment sometimes make processing virtually impossible. Shut down and cleanup is costly. In addition to the normal plant sugars, honeydew has been found to contain the unique carbohydrates trehalulose and melezitose (Bates et.al., 1990; Byrne et. al., 1990; Brushwood and Perkins, 1994; Brushwood, 1998; Hendrix et. al., 1994; Tarczynski et. al., 1992; Wei et. al., 1996) along with a number of more complex oligosaccharides that may also contribute to stickiness. The major sugars that have been identified and routinely quantitated by high performance liquid chromatography (HPLC) in whitefly contaminated cottons, in order of prominence, are trehalulose, fructose, glucose, melezitose, sucrose, trehalose, and smaller amounts of turanose (Brushwood and Perkins, 1994). These seven sugars generally account for 80 to 85% of total known carbohydrates (Brushwood, 1997). Aphid honeydew, which contains little or no trehalulose, is dominated by melezitose, glucose, fructose, sucrose and smaller amounts of trehalose and turanose totaling about 80% of the known sugars identifiable by routine HPLC analysis. Compositions of insect honeydew can vary depending upon a number of factors including other host vegetation (Hendrix et. al., 1992).

This work was conducted to determine if the above sugars commonly found on insect and non-insect contaminated cottons were different in their individual stickiness potential when applied to cotton lint. Stickiness was measured by the standard GRAF/IRCT thermodetector (TD) and minicard tests at different concentrations. Stickiness ratings were correlated with numerical modified Perkins sugar test (Brushwood, 1998) results. Identification of the sticky sugars could aid in research currently underway to focus on eliminating or minimizing the effect specific sugars have on processing stickiness.

Materials and Methods

Five different concentrations of the sugars glucose, fructose, sucrose, turanose, trehalose, trehalulose, and melezitose sugars were applied to a non-sticky, non-insect contaminated 1997 Upland cotton. Each individual sugar or mixture was

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sprayed on the surface of a blended (5 passes through a Syncromatic Blending hopper blender, Fiber Controls, Gastonia, NC.) cotton in 10 gram lots spread over a surface area of 500 cm² on a top loading balance. Applications were made using a Model 1500 Craft air brush (20 pounds regulated pressure). The sprayer provides a very fine mist, which aided in successfully distributing the sugars evenly on the exposed cotton surfaces.

Preliminary high performance liquid chromatography analysis (Dionex anionic DX-300 Spectrophotometer using a PA-1 column and isocratic mode) of sugar extracts from the untreated cotton revealed concentrations of the sugars glucose (0.05%), fructose (0.05%), trehalose (0.03%), and sucrose (0.02%). These sugars totaled about 68% of the identified sugars. Lint samples were determined to be non-sticky by minicard and thermodetector fiber stickiness tests.

Stock sugar solutions (1.0% on the weight of the fiber) were made up at 0.1g/ml (10%) in distilled water and uniformly sprayed on the lint at the rate of 1.0 g of solution to 10 g. fiber. Thus, a total of about 10% moisture was added to the lint as a result of the spraying. After 13 treatments (total of 130 grams), the sprayer was rinsed out thoroughly with distilled water and dried for the next application. Subsequent sugar treatments were carried out using the same procedure. Therefore, the approximate concentrations of the five treatments were 1.0, 0.50, 0.25., 0.125, and 0.07 %. A complete series of samples were sprayed for one individual sugar before fresh solutions of another sugar were applied. In a similar manner, a 100g control lint sample was prepared by spraying distilled water (1.0g/10g) on the lint surface.

Two mixtures of sugars were also sprayed on the lint at concentrations of 1.0, 0.5, and 0.25 %, respectively, The first (# 1) contained 25% glucose, 30% fructose, 30% trehalulose, and 15% melezitose. The second (#2) was composed of 25% glucose, 25% fructose, 10% sucrose, and 40% melezitose. The #1 mixture was purposely made up the to have twice as much trehalulose than melezitose and the #2 solution to have no trehalulose and a high level of melezitose.

After each sugar application, the treated and untreated lint samples were allowed to air dry and condition in a standard ASTM laboratory environment $(21 \pm 1^{\circ}C \text{ and } 65 \pm 2\%$ relative humidity) for a minimum of 48 hours before handblending and dividing into two separate portions. To determine the effect of fiber blending on stickiness as determined by thermodetector and minicard, one half was blended on a laboratory (Cutler-Hammer) rotary blender and the other remained as hand blended. Average oven moisture contents for these rotary and hand blended lint samples were determined to be between 7.0 and 7.2%. All lint samples were subjected to the modified Perkins sugar test (5 replications each) to determine sugars present. Standard thermodetector (5 replicates) and minicard (3 replicates) stickiness tests on all samples were conducted at a controlled relative humidity between 55 and 65% at room temperature.

With the exception of trehalulose, all sugars used in these experiments were obtained through Sigma Chemical Company, St. Louis, Mo. Trehalulose was obtained from Wako Chemicals USA, Richmond, Va. (white crystalline form with a minimum purity of 99%).

All data were analyzed using analysis of variance methodology to determine the significance of the interactions and relationships between dependent variables, measured sugar content, thermodetector (TD) and minicard measurements stickiness ratings.

Results and Discussion

Sugar Surface Distribution

Routine modified Perkins sugar analysis of three unblended raw whitefly honeydew contaminated cottons from previous work was determined to have 0.50, 1.00, and 1.50% sugar (0.5 gram samples - 5 replicates) had standard deviations of determination of \pm 14%, \pm 19%, and \pm 27%, respectively. Average modified Perkins sugar content results for both hand and rotary blended cottons (a test sample sprayed with a mixture of 30% glucose, 30% fructose, 25% trehalulose, and 15% melezitose) were not different. Hence, the rotary blending process did not cause any significant lose of sugar. Standard deviations of sugar content determinations for hand blended samples were $\pm 26\%$, $\pm 37\%$, and $\pm 41\%$ at 0.5, 1.0, and 1.5%, respectively, compared to $\pm 8\%$, $\pm 9\%$, and $\pm 9\%$ for the rotary blended samples. Therefore, the variability of sugar determination for the artificially prepared samples were greater for the hand blended and smaller for the rotary blended cottons when compared to actual honeydew contaminated cottons.

Sugar Analysis

Average modified Perkins sugar test results (based on fiber mass) for each rotary blended treated and untreated lint samples are shown in Table 2. The untreated and water treated (blank) cotton samples (10 determinations each) averaged $0.36 \pm 0.04\%$. There was no statistical difference between the control and water treated lint. All other sugar analyses were determined by averaging the results of 5 replicates. Trehalose (a non-reducing sugar) is not measured by the modified Perkins test. Therefore, target spray concentrations were used for this sugar. The actual sugar contents at the target 1.0% level for the remaining six sprayed-on sugars (after correcting for the untreated control cotton) were determined to be 0.93, 1.02, 0.89, 0.85, 0.95, and 1.30% for glucose, fructose, trehalulose, sucrose, turanose and melezitose, respectively. Sugar test standard deviations at this determination level were 20%, very similar

to the actual whitefly honeydew contaminated cotton. Total melezitose sugar at each treatment level was higher than targeted, however, the actual sugar concentrations levels as determined by the Perkins test were used in correlating to stickiness ratings.

Modified Perkins sugar test results for the rotary blended cotton containing mixed sugar solutions are listed in Table 3. Actual sugars determined to be on the surface of the fiber for the #1 mixture were 0.81, 0.38 and 0.21 at the 1.00, 0.50, and 0.25% application concentrations, respectively. Likewise, actual sugar concentrations for the #2 mixture were 0.76, 0.38 and 0.21% for the same application concentrations. Standard deviations for the sugar content results of mixtures 1 and 2 were slightly less than the standard deviation of results of the single sugar samples.

Stickiness Tests

Where applicable, stickiness ratings were extrapolated for each single sugar and mixture of sugars were determined for 1.0, 0.5, 0.25, 0.13 and 0.07% sugar concentrations by plotting TD sticky counts against actual Perkins test sugar results.

The standard thermodetector stickiness scale (0 to 4 sticky spots - non-sticky; 5 to 14 - slightly sticky; 15 to 24 - moderately sticky, and above 24 as extremely sticky) was used to rate the relative stickiness for all, treated and untreated samples. The size of TD sticky spots on the aluminum foil sheets were larger and easier to count when testing the hand blended lint. Sticky spots for rotary blended samples were somewhat smaller. Hence, a higher degrees of difficulty in achieving a reliable count with the blended samples. The unblended samples produced a higher number of spots, therefore, higher stickiness rating.

Individually, the rotary blended sugar samples sucrose, trehalulose, turanose and melezitose were rated by the thermodetector as most sticky at sugar levels exceeding 0.25% (Figure 1), followed in ascending order by the sugars fructose, trehalose, and glucose. Hand blended TD measurements (Figure 2) indicated that sucrose and trehalulose were more sticky than the other sugars at concentrations above 0.25%. At the 1.0% sugar level, these sugars were rated at the upper end of the moderately sticky range. Turanose at the same concentration level (1%) was rated slightly less sticky (middle of the moderately sticky range) followed by melezitose, fructose, trehalose, and glucose (slightly sticky). The mixed sugar samples (Figure 3) were both rated at moderately sticky at sugar concentrations above 0.40%. Sample #1 was slightly more sticky than Sample #2 at sugar concentrations below 1.0%. This is possibly due to observed differences in stickiness potential between trehalulose and melezitose (Figures 1 and 2). Mix # 1, by design, contained 30% trehalulose and mix #2 had 40% melezitose in the mixture. There was no real difference in TD stickiness between rotary blended and hand blended samples treated with the #2 mixture.

Minicard ratings were very mixed. As with the TD measurements, visual ratings were much easier to determine when evaluating hand blended samples. The rotary blending process obviously succeeded in homogenizing the samples. The standard minicard ratings scale is: 0 (non-sticky), 1 (slightly sticky), 2 (moderately sticky), and 3 (heavily sticky). Each rating was determined by the same experienced operator. At the highest sugar concentrations of 1.0% (Figure 4) the rotary blended samples sprayed with trehalulose, turanose, and melezitose were rated in the 1 scale (slightly sticky) followed by the sugars glucose, fructose, trehalose, and sucrose that were rated as 0 (non-sticky). Hand blended minicard ratings (Figure 5) were higher. Ratings of 3.0 (extremely sticky) for trehalulose, and (2-3 range) moderately sticky for melezitose and turanose were determined at the 1.0% sugar level. Trehalose, sucrose, glucose, were rated slightly sticky (1-2 range) and fructose as non-sticky (0-1 range) at the same concentration.

Statistical analysis of variance for the above measurements indicate highly significant relationships between sugar concentrations and minicard and TD stickiness for both blended and unblended treated lint (Table 1).

Summary

Samples of a single non-insect contaminated upland cotton were treated with varying amounts of sugars and sugar mixes. These sugars were applied by spray on the surface of the cotton using an air brush. Modified Perkins test sugars were determined after treatment to measure actual sugar recovery levels. Subsequent thermodetector and minicard stickiness measurements were conducted on rotary blended and hand blended subsamples of each treated lint. Thermodetector ratings determined trehalulose and sucrose as more sticky at all treatment levels followed by turanose, melezitose, glucose, trehalose, and fructose at a lesser degree of stickiness. Minicard tests also rated trehalulose, turanose, and melezitose treated cottons as the most sticky sugars above the level of 0.5% concentration. The only traditional plant sugar in this series to exhibit any significant degree of stickiness was sucrose as measured by the TD. However, sucrose treated lint was not rated sticky on the minicard. The honeydew specific sugars trehalulose and melezitose have been affirmed as more sticky than the common plant sugars in this study. Both minicard and thermodetector tests rate trehalulose as the stickiest of the seven sugars studied in this test. Melezitose was rated slightly less sticky on the minicard and about half as sticky as the trehalulose on the thermodetector. Another sugar, the disaccharide turanose, commonly found in small quantities in honeydew was also rated as potentially sticky.

These results have proved that there are differences in the potential for stickiness of individual sugars commonly associated with plant and insect sugars. These observations demonstrate that raw cottons containing varying amounts of individual sugars can be expected to exhibit widely different stickiness levels.

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Table 1. Analysis of variances for each variable.

Dependant	Source of		
Variable	variation	Mean Square	F Value
Sugar (%)	Sugar Conc.	0.18602	13.27**
	Interaction	5.07096	361.74 **
		0.01453	1.04 **
TD sticky spots	Sugar Conc.	69.9942	21.87**
(rotary blended	Interaction	363.13228	113.48 **
lint)		10.12491	3.16**
TD sticky spots	Sugar Conc.	44.90782	9.18**
(hand blended	Interaction	757.13726	154.77**
lint)		21.49581	4.39**
Minicard rating	Sugar Conc.	0.19465	5.32**
(rotary blended	Interaction	3.30154	90.24**
lint)		0.06565	1.79*
Minicard rating	Sugar Conc.	1.77484	26.98**
(hand blended	Interaction	12.88061	195.79**
lint)		0.57435	8.73**

** Significant at the 99% confidence level

* Significant at the 95% confidence level

Table 2. Modified Perkins test sugar determinations of an Upland cotton treated with individual honeydew sugars.

		Average	Actual
		Sample	Applied
SAMPLE	*TRT(%)	Sugar (%)	Sugar (%)
Untreated		0.36 ± 0.03	0
Water treated		0.36 ± 0.04	0
Chuassa	1.00	1.20 + 0.20	0.02
Glucose	0.50	1.29 ± 0.30	0.93
	0.30	0.70 ± 0.11	0.40
	0.25	0.01 ± 0.03	0.25
	0.13	0.48 ± 0.10	0.12
	0.07	0.39 ± 0.06	0.03
Fructose	1.00	1.38 ± 0.36	1.02
	0.50	1.02 ± 0.16	0.66
	0.25	0.64 ± 0.04	0.28
	0.13	0.49 ± 0.04	0.13
	0.07	0.41 ± 0.03	0.05
	1.00	1.05 0.04	0.00
Trehalulose	1.00	1.25 ± 0.26	0.89
	0.50	0.81 ± 0.06	0.45
	0.25	0.59 ± 0.06	0.23
	0.13	0.53 ± 0.06	0.17
	0.07	0.42 ± 0.06	0.06
Sucrose	1.00	1.21 ± 0.25	0.85
	0.50	0.81 ± 0.18	0.45
	0.25	0.57 ± 0.04	0.21
	0.13	0.57 ± 0.01 0.47 ± 0.04	0.11
	0.07	0.17 ± 0.01 0.39 ± 0.03	0.03
	0.07	0.59 ± 0.05	0.05
Turanose	1.00	1.31 ± 0.22	0.95
	0.50	0.87 ± 0.11	0.51
	0.25	0.65 ± 0.09	0.29
	0.13	0.59 ± 0.08	0.23
	0.07	0.47 ± 0.03	0.11
Malazitasa	1.00	1.66 ± 0.17	1.20
wielezitose	1.00	1.00 ± 0.17	1.50
	0.50	1.10 ± 0.08	0.74
	0.25	0.89 ± 0.08	0.55
	0.13	$0.6/\pm 0.11$	0.31
	0.07	$0.5/\pm 0.04$	0.21

* Spray on target concentration (based on weight of fiber)

Table 3. Modified Perkins test sugar determination of a Upland cotton treated with mixtures of honeydew sugars.

		Average Sample	Actual Applied
	*TRT (%)	Sugar (%)	Sugar (%)
Mixture #1	1.00	1.17 ± 0.04	0.81
	0.50	0.74 ± 0.06	0.38
	0.25	0.59 ± 0.07	0.21
Mixture #2	1.00	1.12 ± 0.16	0.76
	0.50	0.74 ± 0.11	0.38
	0.25	0.57 ± 0.08	0.21

* Spray target concentration (based on weight of fiber)



Figure 1. Relationship of lint TD sticky spots to measured sugar content - rotary blended samples SUC (sucrose), THU (trehalulose), TUR (turanose), MEL (melezitose), FR (fructose), TREH (trehalose), GLU (glucose).



Figure 2. Relationship of lint TD sticky spots to measured sugar content - hand blended samples SUC (sucrose), THU (trehalulose), TUR (turanose), MEL (melezitose), FR (fructose), TREH (trehalose), GLU (glucose).



4 3.5 3 Minicard Rating ΉU 2.5 ЛEL UR 2 REH 1.5 SUC GLU 1 0.5 FR 0 0 0.2 0.4 0.6 0.8 1 1.2 Sugar (%)

Figure 3. Relationship of the TD sticky spots to measured sugar content - mixed samples #1-B (sample 1 - rotary blended) #2-B (sample 2 - rotary blended) #2-U (sample 2-hand blended).

Figure 5. Relationship of lint minicard stickiness rating to measured sugar content - hand blended samples SUC (sucrose), THU (trehalulose), TUR (turanose), MEL (melezitose), FR (fructose), TREH (trehalose), GLU (glucose).



Figure 4. Relationship of lint minicard stickiness rating to measured sugar content - rotary blended samples SUC (sucrose), THU (trehalulose), TUR (turanose), MEL (melezitose), FR (fructose), TREH (trehalose), GLU (glucose).