REDUCING STICKINESS ON HONEYDEW CONTAMINATED COTTONS USING OVERSPRAY Donald E. Brushwood Clemson, SC

<u>Abstract</u>

Stickiness in textile processing from insect contaminated cottons affects yarn manufacturing and quality. Very sticky cottons may adhere to processing machinery, slow down production, and even cause shut-downs to de-contaminate machinery. The possibility of spraying mild solutions of commonly available compounds to suspect cottons to determine their potential to reduce stickiness was investigated. Water based solutions were applied to slightly sticky, very sticky, and extremely sticky cottons using an atomized overspray system, dried, and tested for their effects on the overall sugar content and fiber stickiness. Reductions in fiber minicard stickiness, particularly in the less sticky cottons, were detected. Fiber SCT stickiness for the extremely sticky cottons was reduced by an average of at least 30 and 25 percent with 5 percent solution pre-treatments with ethyl alcohol and ammonium hydroxide, respectively. Overall fiber sugar contents were also reduced by the ethyl alcohol over-spray treatments as much as 30 percent for very sticky cotton and 24 percent for the extremely sticky cotton. Both treatments were found to successfully reduce levels of the sticky honeydew sugars, trehalulose and melezitose, by at about 50 percent on the very sticky and 40 percent on the extremely sticky cottons.

Introduction

Stickiness on cottons, particularly those harvested in areas conducive to large populations of whiteflies and aphids may be subject to honeydew contamination from these insects. Honeydew, when deposited on open bolls, is extremely sticky and very difficult to remove from the lint. The severity of honeydew contamination prior to harvesting depends upon a number of factors such as insect populations, the type of insect, length of growing season, microbial activity, field weathering, and the intervention steps taken by the grower with the use of insecticides and other products to reduce and control insect populations (Brushwood and Perkins, 1994, Hendrix, Wei, and Leggett, 1992). Honeydew on cottons can cause poor performance in all stages of ginning and yarn manufacturing (Perkins, 1991). Sticky cotton clings to processing rolls, saw blades, spinning machinery, and other processing equipment. Frequent wiping and removing is necessary. In more severe cases, interruptions may prompt the complete shut down of the process to clean up and de-contaminate.

Due to the often sporadic occurrences of honeydew contaminated cottons, from both domestic and foreign sources, the textile industry has adopted various strategies to reduce or minimize the effects of sticky honeydew. The most commonly accepted practice is a technique designed to dilute honeydew stickiness by the selective mixing of contaminated cotton with large volumes of non-sticky cottons in mill lay-downs. This procedure has proved to be reasonably successful at times, but is an inconvenience and is very time consuming for mill operators. A number of other alternative approaches designed to reduce or eliminate the stickiness potential of honeydew contaminated cottons prior to processing have been studied and documented (Balasubramarya, et.al. 1985, Brushwood, 1998, Brushwood, 1998, Hendrix, Blackledge, and Perkins, 1993, Perkins, 1993, Perkins, 1993). Some involve the treatment of module or baled cottons with elevated levels of moisture to promote microbial decomposition the offending sugars in storage (Chun and Brushwood, 1998). However, increased microbial activity may produce conditions that can affect important fiber physical properties such as strength and color. Cotton fiber strength and color can deteriorate to the point that it is no longer meets the necessary criteria for processing. Attempts to reduce stickiness by heating honeydew contaminated fiber (Brushwood, 1998), if not carefully monitored and controlled also could reduce fiber strength and increase yellowness.

This paper describes a study in which honeydew contaminated cottons rated at three stickiness levels by the standard minicard test that were treated with different water based over-spray compounds designed to reduce fiber stickiness potential without adversely affecting fiber physical properties such as strength and color.

Experimental

Fiber Chemical and physical property tests

The following tests (a minimum of triplicate measurements) were conducted on each control to establish baseline values, 1. Moisture contents were determined according to a standard test (ASTM Method D2495-01) oven drying test. The standard error for the test at the 6 to 8 percent moisture level is ± 0.1 percent, 2. Fiber sugar concentrations, both a simple glucose and total hydrolyzed sugar concentrations, were determined by the YSI 2700 glucose test (Gamble, 2001); 3. Fiber ethyl alcohol extractions, which are generally considered to be a very good indicator of the levels of noncellulosic materials on the cotton, were conducted using a microwave assisted procedure which is much more efficient and reliable than the older Soxhlet extraction method. The standard error of determination for the microwave alcohol extraction test is \pm 3 percent, 4. Standard minicard stickiness and Sticky Cotton Thermodetector (SCT) tests (Brushwood and Perkins, 1993) were conducted. The SCT test method provides a numerical "sticky spot" count and the minicard rating scale ranges from 0 for a non-sticky to a 3+ for extremely sticky cottons; 5.Anionic High Performance Liquid Chromatography (HPLC) scans were also conducted on water extracts of the control and treated samples to characterize each for their content of the five most abundant carbohydrates glucose, fructose, sucrose, and the two honeydew specific sugars, trehalulose and melezitose (Brushwood and Perkins, 1994). The standard error of determination for the SCT and HPLC tests are ± 25 percent due to the random distribution of the honeydew on the contaminated fiber and the fact that only 2.5 and 1.0 grams of sample are used for each test, respectively; and 6. HVI physical property measurements of these fibers for micronaire, strength, and color Rd (grayness) and +b (yellowness) were also determined by the USDA Fiber Testing Laboratory at Clemson. Subsequent values for the controls and over-spray treated samples are the average of at least 3 measurements per cotton.

Control Cottons

Two bales of upland cotton originating from the same growing season with two different pre-determined degrees of honeydew contamination were used in this study. Both were repeatedly tested for stickiness by the standard minicard test. The first was rated as "slightly sticky" (an average of 0.5) and the other "extremely sticky" (3 +) on the minicard scale. Each bale was also subjected to triplicate SCT testing to obtain numerical "sticky spot" value. The slightly sticky bale averaged 12 ± 4 sticky spots and the extremely sticky bale averaged 51 ± 15 sticky spots. A third sample s prepared by blending (at least 15 passes through a Syncromatic Blending System) in a fiber hopper 0.5 pounds (227 grams) of the "extremely sticky" (3 + minicard rating and 51 SCT sticky spots) and 4.5 pounds (2043 grams) of the "slightly sticky" (0.5 minicard rating and 12 SCT sticky spots) sample to obtain a 1/9 mixture ratio. The goal was to create another sample with a stickiness level somewhere between the two original bales. The blended control sample, when subjected to the minicard stickiness test averaged a rating of 3.0 (very sticky) and the average (4 measurements) numerical SCT sticky spot count was 24 ± 6 spots.

After conditioning in our laboratory for at least a week at $65 \pm 2\%$ relative humidity and 21 ± 1 °C (ASTM Method D3374), all three samples were hand blended, stored, and used as reference fibers for the subsequent over-spray treatments.

Overspray Treatments

Using an air brush, which sprays a fine mist on the surface of the cotton, water and water based solutions of the following four commonly available compounds were sprayed onto the fiber surface. The solutions were water, 5 percent solutions (by volume) of water and ammonium hydroxide (concentrated) and absolute ethyl alcohol, and a 0.025 percent solution of Triton X-100 (Union Carbide) surfactant dispersed in water. Each was added to the fiber gravimetrically at the rate of 10 and 25 percent based on the weight on the fiber (OWF). Hence, there were a total of 8 treatments per control cotton.

One hundred grams of the total treated cotton were evenly distributed on sheets of aluminum foil (15 by 25 inches) and placed on a tared top loading balance. One-half of required solution (5 grams for a 10 percent treatment or 12.5 grams for a 25 percent treatment) was sprayed on the cotton. The fiber was then turned over and the remaining one-half of the solution was sprayed on as before. A total of 200 grams of over-spray sample were prepared by repeating the above procedure. Immediately after treatment, each sample was placed in a forced draft oven set at 115 °C, dried for 5 minutes, removed, and allowed to condition overnight in the laboratory at $65 \pm 2\%$ relative humidity and at 21 \pm 1°C. Final fiber moisture contents for the control and over-sprayed cottons were determined after blending on a circular laboratory fiber blender (Cutler-Hammer) and reconditioning as above for at least 24 hours. The chemical and physical tests listed above were eventually conducted on these samples to determine any potential changes caused by the over-spray treatments.

Statistical analyses of the data using SAS version 8 software and calculated coefficients of correlation (R^2 values) were conducted on the collected data and are presented in this paper.

Results and Discussion

Over-spray and untreated fiber moisture contents

Fiber moisture contents ranged from a low of 6.55 percent for a surfactant treated blended control sample to a high of 7.34 percent for a water over-sprayed slightly sticky control sample. Over-spraying with water and the water based mixtures and drying by the prescribed procedure resulted in moisture levels equivalent to the untreated controls. For example, the average moisture content of the slightly sticky control cotton was 7.05 percent (wet basis) with the related over-sprayed samples averaging 6.93 ± 0.19 percent. Moisture content for the blended control (very sticky) averaged 6.92 percent and the subsequent samples that were over-sprayed with the various solutions averaged 6.93 ± 0.20 percent. The extremely sticky control averaged 6.84 percent with the average for the over-sprayed sample was 6.90 ± 0.17 percent. No significant differences in moisture contents were seen between the 10 or 25 percent solution over-spray treatments. Therefore, there were no significant differences seen between the moisture contents for either control fiber and their respective over-sprayed samples.

Fiber physical properties

Average HVI micronaire for the three controls were 4.30 for the slightly sticky, 4.31 for the very sticky, and 4.62 for the extremely sticky cottons, respectively. HVI strengths averaged 28.8, 29.3, and 32.7g/tex for the same cottons. The corresponding average over-sprayed sample strengths were 28.1, 29.1, and 32.4g/tex, respectively. No significant differences were seen between untreated and their respective over-sprayed samples.

HVI color Rd (grayness) for all three controls ranged from 76.6 to 77.3. Over-sprayed sample Rd averages ranged from a high of 77.5 to a low of 75.2. No statistical differences were seen between values for the untreated and over-sprayed samples. Fiber yellowness (+b) ranged from 10.9 to 11.3 for the control cottons with an average of 11.1 ± 0.2 for all 24 of the over-sprayed cottons. As with the Rd measurement, no significant differences in the +b measurement were seen between the untreated and over-sprayed samples. Therefore, over-spraying with water, ethyl alcohol, surfactant, and ammonium hydroxide at the concentrations used in this study followed by immediate drying appeared to have no significant effect on overall fiber strength and color.

Treatments and fiber hydrolyzed sugar content

Hydrolyzed sugar contents for the controls averaged 0.32 percent for the slightly sticky, 0.52 percent for the very sticky, and 1.42 percent for the extremely sticky controls. With the single exception of the 25 percent water over-spray sample for the slightly and very sticky cottons, sugar concentrations were reduced for all three stickiness levels when overspray was applied. The average differences in hydrolyzed sugar content between average 10 and 25 percent solution treatments with water, ethyl alcohol, ammonium hydroxide, and surfactant treatments varied from 3 to 6 percent of the total sugars. Since these differences represented such a small percentage of the overall total sugars, the 10 and 25 percent over-spray sugar contents for water, ethyl alcohol/water, surfactant/water, and ammonium hydroxide/water solutions were averaged.

Results (figure 1) show the decreases in fiber hydrolyzed sugar content by the water, ethyl alcohol, surfactant, and ammonium hydroxide over-spray treatments. No reduction in sugar level was seen as a result of water over-spray for the slightly sticky cotton and an average of 6 percent for the more sticky samples. Additional measurable reductions in sugar content were seen with the other three over-spray solutions at all three levels of stickiness. For example, the slightly and very sticky cottons averaged an overall decrease in sugar content of 27, 34, and 30 percent for the ethyl alcohol, surfactant, and ammonium hydroxide treatments, respectively. The average reductions for the extremely sticky cotton were 11, 24, and 11 percent for the same over-spray treatments, respectively.



Stickiness testing

All untreated and over-sprayed samples in this series (27) were subjected to at least triplicate standard minicard and SCT stickiness tests. The average (triplicate measurements) minicard stickiness rating for the slightly sticky cotton for the 4 over-sprayed treatments was reduced from the original 0.5 to 0. Ratings for the very sticky (blend sample) over-spray treated cottons remained unchanged (3.0), except in the case of the ethyl alcohol and ammonium hydroxide treated samples that were reduced to a 2.5 rating. No significant change in minicard rating was seen with any over-spray treatment for the extremely sticky cotton. The results of the average numerical sticky spot ratings for the untreated controls and the combined 10 and 25 percent over-spray treatments are shown in figure 2. Overspraying with the water and water based solutions reduced the number of sticky spots detected for the slightly sticky control. The number of sticky spots were reduced an average of 33 percent for both the water and 5% ethyl alcohol treatments, about 50% with the surfactant solution, and 42 percent with the ammonium hydroxide solution treatments. Sticky spot counts for the very sticky and extremely sticky cottons were also reduced by the water treatments by 38 and 20 percent, respectively. The ethyl alcohol over-spray treatment resulted in the highest overall reduction in net sticky spots on the very sticky and extremely sticky cottons. These reductions were 42 and 29 percent. The ammonium hydroxide overspray solution successfully reduced stickiness for these samples by about 30 percent each. SCT sticky spot counts for the surfactant treated very and extremely stick cottons were mixed and highly variable. It was thought that perhaps residual surfactant on the surface of the over-sprayed fibers may have influenced the type, formation, and size of the SCT sticky spots that were counted.



Relationship between sugar content, SCT stickiness, and fiber alcohol extractions

A calculated correlation coefficient determined between hydrolyzed sugar content and SCT sticky spots for these untreated and over-sprayed cottons (all 27 samples) resulted in an R square value of 0.83 (Figure 3). The same sugar values, when correlated with the fiber alcohol extraction percentages also gave a very positive relationship of $R^2 = 0.92$ (Figure 4). Since all of these fibers were in a narrow micronaire range of between 4.3 and 4.6, differences in fiber micronaire had little or no significant influence on the total alcohol surface extractions. Therefore, it follows that there should be a very strong relationship between fiber hydrolyzed sugar content and the total alcohol extractions. The calculated correlation coefficient ($R^2 = 0.82$) verified that relationship (Figure 5.).







Figure 5. The relationship between alcohol extractables and fiber SCT sticky spots

Changes in fiber surface individual sugar compositions as a result of overspray treatments

Anionic HPLC analysis of water extracts from the controls and over-sprayed samples were conducted to determine concentrations of the five most abundant sugars found on honeydew contaminated cottons. These sugars were the mono-saccharides glucose and fructose, the di-saccharide sucrose, and the two sugars associated with insect contamination, trehalulose and melezitose. Concentrations were determined by comparison to known calibration standards prepared from pure sugars that were run during sample analyses. Sucrose concentrations for all three controls and over-sprayed samples varied from 0.02 to 0.03 percent (about 6 percent of the hydrolyzed sugar content for the slightly and very sticky controls and samples and 2 percent of the extremely sticky controls and samples). No significant changes in sucrose levels were detected between the untreated and over-sprayed samples. Combined concentrations of the very sticky honeydew sugars, trehalulose and melezitose, averaged about 0.04 percent (or about 12 percent of the hydrolyzed sugar content) for the untreated slightly sticky control cotton. The same sugars averaged 0.087 percent (about 17 percent of hydrolyzed sugar content) and 0.342 percent (24 percent) for the very sticky and extremely sticky controls, respectively. Combined levels of the two least sticky of these sugars on these controls, glucose and fructose were found to account for as much as 80 percent of the hydrolyzed sugar content for the extremely sticky control, decreasing to 53 percent for the very sticky control, and only about 24 percent for the extremely sticky control.

Shown in Table 1 are the average calculated concentrations of these five sugars for the control as well as the average over-sprayed samples. The slight increases detected in total glucose and fructose content with the ethyl alcohol treated cottons at all three stickiness levels may be explained by the possibility that the alcohol over-spray treatment may have actually converted some of the sticky di and tri-saccharides, trehalulose and melezitose, on the fiber to the less sticky mono-saccharides, glucose and fructose.

		co	ctons.		
		Sugar Co	ncentration (%)*		
Sample ID	Glucose	Fructose	Sucrose	Trehalulose	Melezitose
Control-S.S.	0.134	0.140	0.023	0.017	0.019
Control-V.S.	0.131	0.145	0.032	0.059	0.028
Control-E.S.	0.132	0.204	0.025	0.213	0.129
Water-S.S.	0.142	0.141	0.023	0.013	0.005
Water -V.S.	0.134	0.155	0.023	0.055	0.016
Water -E.S.	0.127	0.191	0.020	0.138	0.119
TX100-S.S.	0.122	0.139	0.024	0.019	0.007
TX100-V.S.	0.133	0.153	0.029	0.057	0.020
TX100-E.S.	0.129	0.192	0.017	0.135	0.109
Ethanol-S.S.	0.160	0.156	0.032	0.016	0.007
Ethanol-V.S.	0.192	0.213	0.019	0.018	0.014
Ethanol-E.S.	0.138	0.202	0.018	0.105	0.109
A. Hyd-S.S.	0.118	0.130	0.021	0.015	0.004
A. Hyd-V.S.	0.120	0.137	0.017	0.028	0.011
A. Hyd-E.S.	0.143	0.171	0.013	0.097	0.092
	<u> </u>	* Standard error	of determination ±	= 25%	<u> </u>

Table 1. Concentrations of five major HPLC identified sugars in extracts from control and over-sprayed sticky

Decreases in concentrations of the average trehalulose and melezitose in the very sticky and extremely sticky series of cottons were seen with all 4 over-spray treatments. For example, the combined concentrations of these two sugars found in on the extremely sticky cotton were reduced by 25, 29, 37, and 45 percent for the average water, surfactant, ethyl alcohol, and ammonium hydroxide over-sprays, respectively. A graphical summary of the changes in HPLC determined concentrations of these honeydew sugars as related to average over-spraying treatment for all three levels of stickiness can be seen in Figure 6.



Figure 6. Reductions in total trehalulose and melezitose content as a result of overspray treatments (combined 10 and 25% treatments) E.S. untreated = 3 + on minicard V.S.untreated = 3.0 on minicard

S.S. untreated = 0.5 on minicard

SCT stickiness and HPLC determination of entomological sugar content

The calculated correlation of 0.84 (figure 7) between SCT sticky spots and the combined sticky entomological sugar concentrations, trehalulose and melezitose, for the controls and 24 over-sprayed samples, as determined by HPLC, illustrates a very positive relationship between the SCT method of measuring stickiness and the average concentrations of these two honeydew specific sugars from water extracts of the cotton.



Figure 7. The relationship between HPLC determined the combined concentrations of trehalulose and melezitose sugars and average SCT sticky spots

Summary

A preliminary study was conducted to determine the possibility of over-spraying honeydew contaminated cottons prior to processing to reduce stickiness. Relatively low concentration over-spray compounds were water based mixtures of 5 percent ethyl alcohol and ammonium hydroxide, 0.025 percent Triton X-100 surfactant, and water. Upland cottons representing three levels of honeydew contamination were treated with various over-sprays to determine the potential to reduce fiber stickiness. Subsequent results produced significant reductions in determined hydrolyzed sugar content, numerical stickiness ratings by the standard SCT test, and indications of reduced stickiness by the minicard test, and the average fiber content of the sugars trehalulose and melezitose when analyzed by anionic HPLC scans of water extracts from the cottons. All of the above tests may be considered as good methods that are often used to predict fiber stickiness in processing. The minicard test is the preferred test method because it more closely simulates actual performance in processing. In this study, general results show that overspraying with of ethyl alcohol and ammonium hydroxide were the most successful in reducing fiber stickiness potential. This was a small and limited laboratory study using low water based concentrations of commonly available compounds. These promising results should open up the possibilities for a much more extensive and detailed studies using other or more concentrated solutions of the above mixtures on additional cottons of varying degrees of honeydew contamination and levels of stickiness. It would be particularly good to have a range of samples at different minicard stickiness levels to determine the concentrations and levels of over-spray necessary to reduce minicard ratings by one are two levels.

References

Balasubramarya, R.A., S.P. Bhalaudikar, and K.M. Paralickar 1985 A new method of reducing the stickiness of cotton. Textile Res. Journal Vol.55: pp 227-232.

Brushwood, D.E., and Perkins, H.H. Jr. 1993 Cotton sugar and stickiness test methods. Canadian Textile Journal 110:6 pp 54-62.

Brushwood, D. E. and Perkins, H.H. Jr. 1994 Characterization of sugar from honeydew contaminated and normal cottons. Proc. of Beltwide Cotton Conference, National Cotton Council, Memphis, TN. pp 1408-1411.

Brushwood, D.E. 1998 Moderate moisture storage effects on strength, color, stickiness, and carbohydrate content of raw cotton. Proc. of Beltwide Cotton Conference, National Cotton Council, Memphis, TN. pp 696-702.

Brushwood, D.E. 1998 The use of elevated temperatures to reduce the stickiness potential of cottons. Proc. of Beltwide Cotton Conference, National Cotton Council, Memphis, TN. pp 1553-1557.

Chun, D. T.W., and Brushwood, D. E. 1998 Part I. High moisture storage effects on cotton stickiness. Textile Res. Journal 68(9) pp 642-648

Gamble, G.R. 2001 Evaluation of an enzyme-based method for the detection of stickiness potential on cotton lint. Journal of Cotton Science Vol.5 pp 169-173

Hendrix, D.L., Y.A. Wei, and L.E. Leggett, 1992 Homo-pteron honeydew sugar composition is determined by insect and the plant species. Comp. Biochemical. Physiology. 101B: pp 23-27.

Hendrix, D. L., Blackledge, B., and H.H. Perkins, Jr. 1993 Development of methods for detection and elimination of insect honeydew on cotton fiber. Proc. of Beltwide Cotton Conference, National Cotton Council, Memphis, TN. pp 1600-1603.

Perkins, H.H. Jr. 1991 Cotton stickiness – a major problem in textile processing. Proc. of Beltwide Cotton Conference, National Cotton Council, Memphis, TN. pp 523-524.

Perkins, H.H. Jr. 1993 Strategies for processing honeydew contaminated cotton. Proc. of Beltwide Cotton Conference, National Cotton Council, Memphis, TN. pp 1461-1462.

Perkins, H.H. Jr. 1993 Controlling cotton stickiness – summary of progress. Proc. 21st International Cotton Conference, Bremen, Germany pp 219-224.