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TEXTILE topics

International Textile Center Texas Tech University Lubbock, Texas USA

Sharing current research information and trends in the fiber and textile industries.

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ITC NEWS & EVENTS

Working with the Lubbock Cotton Exchange, the International Textile Center hosted the Texas International Cotton School in October. Participants spent two weeks learning about the textile and cotton industries.

In November the ITC, in cooperation with the Texas Department of Agriculture, presented a "U.S. Fiber Properties and Export School" to a select group of representatives from Vietnam's textile and garment industry. The event was timely because, under a recently ratified trade pact, Vietnam has agreed to open its market to industrial and agricultural goods from the U.S.

COTTON FIBER BOOK SALE

Thanks to everyone who ordered a copy of our book, "Cotton Fiber Development and Processing, an Illustrated Overview." We had a tremendous response and received a lot of positive feedback.

We still have copies of the book available for sale. The price is $15 plus shipping and handling. For more information on purchasing this book, please call our office at 806-747-3790 ext. 513 or visit our website at: www.itc.ttu.edu and click "What's New" from the home page.

NEW EQUIPMENT

The International Textile Center has acquired 4 new instruments. They include:

- Digital Elmendorf Tearing Tester - Digitally displays the average force required to propogate a single-rip, tongue-type tear starting from a cut in paper, cardboard, plastics, non-wovens and woven fabrics.
- Nu-Martindale Abrasion & Pilling Tester - This machine determines the abrasion resistance of textile fabrics by performing and counting the number of rubs needed to breakdown or rupture the individual fibers.
- Plasma System V15GL - Plasma, also called the 4th state of matter, is a medium composed of radicals, unstable molecules, photons, as well as charged particles, ions, and electrons. The high-energy particles generated by an electric field are used to create highly reactive sites on fabric surfaces. This allows the modification of the surfaces in order to achieve features like soil resistance, flame retardance, or permanent press without changing the bulk properties of the fabrics.
- Fourier Transform Infrared Micro-spectroscopy - This new analytical instrument will allow identifying contaminants or finishes on fibers, yarns and fabrics with minimal sample preparation.

ITC TRAVEL

- USDA Cotton Research Review, New Orleans, LA, July 16-17, 2002, Dean Ethridge
- International Textile Committee for Textile Care, Baltimore, MD, September 21-24, Noureddine Abidi
- American Association of Textile Chemists and Colorists International Conference and Exhibition, Charlotte, NC, September 30 - October 4, Noureddine Abidi
- Cotton Genetics Group Meeting, Raleigh, NC, September 20-22, Eric Hequet
- USDA Committee of Cotton Agriculture Quality Measurement Conference, Memphis, TN, October 23-25, Eric Hequet

HIGH-SPEED STICKINESS DETECTOR MEASUREMENT:
Effect Of Temperature Settings And Relative Humidity

Eric Hequet
Noureddine Abidi

Cotton Incorporated and the Texas Food and Fiber Commission funded the research reported here.

INTRODUCTION

In spinning mills cottons contaminated with stickiness are causing serious problems. The honeydew present on the cotton lint contaminates all the mechanical instruments used in the transformation process from fiber to yarn; i.e., opening, carding, drawing, roving and spinning operations. These contaminants are mainly sugar deposits produced either by the cotton plant itself (physiological sugars) or by feeding insects (entomological sugars), the latter being the most common source of contamination.

Tarczynski et al., 1992 used the aphis-stylet technique to obtain pure phloem sap from cotton plants to show that the major sugar translocated is sucrose (>90%). Therefore, the presence of sucrose in the cotton lint reveals that the plant was still growing when, either (a) freezing temperatures occurred, killing the plant so quickly that the sucrose within the fiber lumen was not metabolized into cellulose, or (b) harvest-aids had caused some bolls to open prematurely.

Cotton stickiness is more often caused by insect honeydew than by physiological sugars. The main honeydew-producing insects that infest cotton plants are the cotton whitefly, *Bemisia tabaci* (Gennadius) and the cotton aphid, *Aphis gossypii* (Glover). Whiteflies and aphids are both plant sap-sucking insects that feed by inserting their mouthparts and the stylet into the leaf tissues. The sap is digested and ejected as a droplet of honeydew. The honeydew attaches itself to the leaves and the fibers of opened bolls. The ginning process scatters the honeydew, making it difficult to detect with the naked eye.

Hendrix (Hendrix et al., 1992), analyzed honeydew from *A. gossypii* and *B. tabaci* found 38.3% of melezitose plus 1.1% of trehalulose in the aphid honeydew and 43.8% of trehalulose plus 16.8% of melezitose in the whitefly honeydew. Other relative percentages may occur depending on the environmental or feeding conditions.

In a previous work, Hequet and Abidi (2002) showed that sugars found in contaminated cotton with honeydew have different thermal and hygroscopic properties. Therefore, it was decided to investigate the effect of the relative humidity of the ambient conditions and of the temperature settings of the hot plate of the High Speed Stickiness Detector on cotton stickiness measurements.

MATERIALS AND METHODS

One hundred and fifty commercial cotton bales representing a wide range of stickiness and types of contamination, i.e. from whitefly, from aphids and from physiological sugars, were selected. Samples came from 3 different growing regions. Fifty bales came from Area 1, known to have important whitefly populations and very few aphids. Fifty bales came from Area 2, where both types of insects coexist. Fifty bales came from Area 3, where large populations of aphids and very few whiteflies exist. The bales were broken and 2 samples per bale taken. The sugars present on the contaminated lint were identified and quantified using High Performance Liquid Chromatography. In addition each sample was tested on the High Speed Stickiness Detector.

High Performance Liquid Chromatography on Fiber Samples

For each sample, 1g of cotton fibers was placed in plastic bag and 20 ml of 18.2 megohm water were added. A sample of the aqueous solution was taken from the bag with a 10 cm^3 syringe on which a 0.2 micron filter (nylon membrane-polypropylene housing) was attached. A 1.5 ml filtered sample was deposited into the 1.5 ml autosampler vial. Sugars were separated on the columns (CarboPac PA1 Anion exchange Guard column and two CarboPac PA1 Anion exchange Analytical Columns) in series with a Gradient Eluent system: Eluent 1: 200 mM NaOH and Eluent 2: 500 mM Sodium Acetate and 200 mM NaOH. Three replications were performed on each sample (2 samples per bale x 3 replications = 6 tests per bale). The results are expressed in percent of the fiber weight and in percent of the total sugars.

High Speed Stickiness Detector

The High Speed Stickiness Detector is derived from the Sticky Cotton Thermodetector, which was approved as a reference test by the International Textile Manufacturers Federation in 1994 (Frydrych et al., 1998). The High Speed Stickiness Detector is an automated version of the Sticky Cotton Thermodetector (Frydrych et al., 1994). First, a sample of cotton weighing between 3.0 and 3.5 g is opened using a rotor type opener. The mass of opened fiber is then shaped into a rectangular, even pad of fibers. This pad is deposited by the system on aluminum foil. Then, the sample passes successively in front of four stations. Hot pressure is applied to the

sample (53°C, 30 seconds). This renders the honeydew sticky. The sticky points in contact with the aluminum are then fixed in place by means of pressure exerted at ambient temperature. The loose cotton fibers are then removed using a vacuum then a cleaning roll. The sticky spots still adhering to the aluminum foil are counted and sized by an image analyzer.

As shown previously (Hequet et al., 1997), the High Speed Stickiness Detector readings within a sample follow a Poisson-like distribution; therefore, a square root transformation is adequate to normalize it prior to statistical analysis. Consequently, all the statistical analyses were performed on square root transformed data. Three replications were performed on each sample (2 samples per bale x 3 replications = 6 tests per bale).

High Speed Stickiness Detector Test at Two Different Relative Humidities

The effect of the relative humidity on the stickiness readings was achieved at the following conditions: 55±2% relative humidity, 23±1°C and 65±2% relative humidity, 21±1°C. United States Department of Agriculture – Agricultural Marketing Service recommends to condition samples for at least 48 hours prior to testing. Thus, the High Speed Stickiness Detector instruments and the samples were conditioned for 96 hours prior to testing to insure that both the instrument and the cotton were in equilibrium with the laboratory conditions. The hot plate temperature of the High Speed Stickiness Detector was set at 53°C (recommended manufacturer setting).

High Speed Stickiness Detector Test at Several Hot Plate Temperature Settings

The recommended manufacturer setting for the High Speed Stickiness Detector hot plate is 53°C. The measurements were done at this temperature. Then, this setting was modified to perform the tests at the following hot plate temperatures: 27°C, 34°C, 40°C, and 67°C. Tests were performed under standard laboratory conditions, i.e., relative humidity of the ambient was 65±2% and temperature was 21±1°C. It should be pointed out that the reason for the choice, by the manufacturer, of the hot plate temperature setting at 53°C is not documented in the literature.

RESULTS AND DISCUSSION

Sucrose is virtually the only sugar in the phloem sap of cotton plant (Hendrix et al., 1992). The insects produce trehalulose and melezitose by isomerization and polymerization of sucrose, neither of these sugars occurs in cotton plant (Hendrix, 1999). Therefore their presence on cotton lint demonstrates honeydew contamination.

The one hundred and fifty bales were selected based on their insect sugar content. **Table 1** shows the High Performance Liquid Chromatography results obtained, expressed as a percent of the fiber weight. The examination of this table reveals that all cottons were contaminated with insect honeydew to some degree. When expressed as a percent of the total sugars (**Table 2**) the average trehalulose content for Area 1 is 67% higher than melezitose content, revealing whitefly honeydew contamination. For Area 2, the average trehalulose content is 28% lower than melezitose content, revealing a probable contamination by whitefly and aphid honeydew. For Area 3, the average trehalulose content represents less than two percent of melezitose content revealing aphid honeydew contamination.

Hequet and Abidi (2002) have shown that, the individual sugars present on sticky cotton have different hygroscopic properties. Among the sugars tested, trehalulose and fructose have the highest hygroscopicity. After equilibrium is reached, the amount of adsorbed water at 65% relative humidity and 21°C corresponds to 3 molecules of H_2O adsorbed per molecule of trehalulose or fructose. This suggests a relationship between water content of the raw material and stickiness. It confirms the findings from previous work reporting that stickiness caused by honeydew depends on the relative humidity (Gutknecht et al., 1986 ; Frydrych et al., 1993). Consequently, it was decided to test the samples at two different relative humidities. The lower level (55±2%) was selected to represent common ring spinning conditions. The higher level (65±2%) was selected to represent the standard textile laboratory atmosphere according to American Society for Testing and Materials standard practice D 1776. **Figure 1** shows linear relationship between the High Speed Stickiness Detector readings (square root transformed) performed at 55±2% relative humidity, 23±1°C and 65±2% relative humidity, 21±1°C with the manufacturer recommended hot plate temperature of the instrument set at 53°C. As shown in

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Table 1: Sugar contents, measured by high-performance liquid chromatography for the three growing areas, expressed as a percentage of the fiber weight.

		Sugar contents†								
Area		In	Treh	Gluc	Fruc	Trehal	Suc	Mel	Mal	Total
		% of fiber weight								
1	Average	0.035	0.018	0.043	0.059	0.103	0.000	0.052	0.018	0.328
	Minimum	0.011	0.006	0.010	0.009	0.003	0.000	0.000	0.000	0.064
	Maximum	0.068	0.046	0.107	0.146	0.358	0.003	0.160	0.074	0.940
2	Average	0.061	0.003	0.073	0.107	0.073	0.011	0.088	0.003	0.419
	Minimum	0.042	0.000	0.034	0.010	0.001	0.000	0.017	0.001	0.114
	Maximum	0.074	0.010	0.110	0.187	0.236	0.037	0.201	0.009	0.794
3	Average	0.035	0.011	0.055	0.067	0.000	0.024	0.026	0.006	0.225
	Minimum	0.018	0.002	0.020	0.010	0.000	0.001	0.002	0.001	0.074
	Maximum	0.053	0.029	0.147	0.215	0.002	0.088	0.079	0.017	0.462

† In, inositol; Treh, trehalose; Gluc, glucose; Fruc, fructose; Trehal, trehalulose; Suc, sucrose; Mel, melezitose; Mal, maltose; Total, total sugars.

Table 2: Sugar contents, measured by high-performance liquid chromatography for the three growing areas, expressed as a percentage of the total sugars.

		Sugar contents†							
Area		In	Treh	Gluc	Fruc	Trehal	Suc	Mel	Mal
		% of total sugar							
1	Average	13.7	6.3	16.9	20.6	23.7	0.3	14.2	4.2
	Minimum	6.4	3.1	8.2	12.7	2.8	0.0	0.0	0.3
	Maximum	21.0	14.7	33.6	35.1	41.7	1.5	36.7	10.7
2	Average	19.1	1.1	20.1	24.4	13.5	2.3	18.7	0.7
	Minimum	7.9	0.2	12.1	8.6	0.7	0.1	7.0	0.3
	Maximum	36.9	5.1	36.5	36.5	29.4	5.9	25.6	1.4
3	Average	17.6	5.8	25.4	28.2	0.2	8.9	11.4	2.6
	Minimum	8.4	0.9	13.8	13.1	0.0	1.4	2.4	0.3
	Maximum	33.3	16.3	37.3	46.5	1.7	23.8	28.3	6.9

† In, inositol; Treh, trehalose; Gluc, glucose; Fruc, fructose; Trehal, trehalulose; Suc, sucrose; Mel, melezitose; Mal, maltose.

Table 3, the square root transformed High Speed Stickiness Detector readings at 55±2% relative humidity are in average 23.2% lower. No significant interaction between the Area and the relative humidity is noticed. This suggests that the moisture absorption equilibrium of the sugars involved in the stickiness phenomenon is lower at 55% relative humidity than at 65%. Consequently, all the stickiness readings are lower but it does not modify the relative ranking of the 3 areas.

Hequet and Abidi (2002) show that sugars present on honeydew-contaminated lint have different thermal properties. **Figure 2** shows the melting point of inositol, trehalose, glucose, fructose, trehalulose, sucrose and melezitose. Trehalulose has the lowest melting point (48°C). Therefore, when testing cotton for stickiness at 53°C (recommended manufacturer setting for the High Speed Stickiness Detector) trehalulose, which is mainly present in whitefly honeydew, should melt while the other types of sugars should remain unchanged. As shown on the Differential Scanning Calorimetry profile **(Figure 3)**, trehalulose begins to melt around 25°C. Therefore, we can hypothesize that the honeydew droplets having a high percentage of trehalulose would be sticky at any temperature above 25°C, and that the lower the trehalulose percentage in those droplets, the lower the "sticky potential" exists. To confirm this hypothesis, the High Speed Stickiness Detector hot plate temperature was modified to perform the stickiness measurement at different temperature settings. The following hot plate temperatures were chosen: 27°C, 34°C, 40°C, 53°C, and 67°C. All the tests were performed in standard laboratory conditions at 65±2% relative humidity and 21±1°C.

Figure 4 shows an increase of the High Speed Stickiness Detector readings with increasing hot plate temperature for the 3 areas tested. A significant interaction between these two parameters is apparent.

To elucidate this interaction it was decided to plot the High Speed Stickiness Detector reading at each temperature against the readings at the manufacturer recommended temperature of 53°C.

Fig. 1: Relationship between square-root-transformed readings from the H2SD at 65% ± 2% relative humidity (RH), 21°C ± 1°C and at 55% ± 2% RH, 23°C ± 1°C.

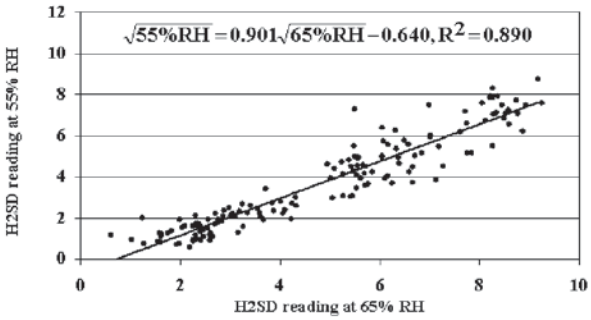


Table 3: Variance analysis on the square root transformed data: effect of growing area and relative humidity on H2SD readings.

		df†	*F*†	Probability	H2SD‡
	Intercept	1	1093.26	0.0001	
	Area	2	6.20	0.0023	
	1				4.336 a§
	2				3.671 b
	3				4.774 a
	RH	1	18.85	0.0001	
	55%				3.701 b
	65%				4.820 a
Area	RH	2	0.11	0.8990	
1	55%				3.752
	65%				4.919
2	55%				3.193
	65%				4.148
3	55%				4.157
	65%				5.392
	Error	294			

† df, degrees of freedom; *F*, variance ratio.
‡ Square root transformed.
§ Readings not followed by the same letter are significantly different with a = 5% (according to the Newman-Keuls test).

Figures 5 to **8** show the relationship between the High Speed Stickiness Detector readings (square root transformed) performed at 53°C and the readings performed at 67°C, 40°C, 34°C, and 27°C for Area 1, Area 2, and Area 3.

Hot plate temperature setting at 67°C:

The three Areas seem to follow the same trend; the relationships were not linear, revealing a probable saturation phenomenon (**Figure 5**). The image analysis software does not seem to be able to separate 2 merging sticky spots. When the number of sticky spots on the aluminum foil is large, the probability for 2 sticky spots to merge is high, making the image analysis software unable to count accurately above 60 sticky spots. Of course this number needs to be adjusted depending on the size of the sticky spots. The statistical analysis (**Table 4**) did not show a significant interaction between temperature setting and Area. At 67°C the readings averaged 31.8% higher than at 53°C on the square root transformed data.

Fig. 2: Melting point of selected sugars.

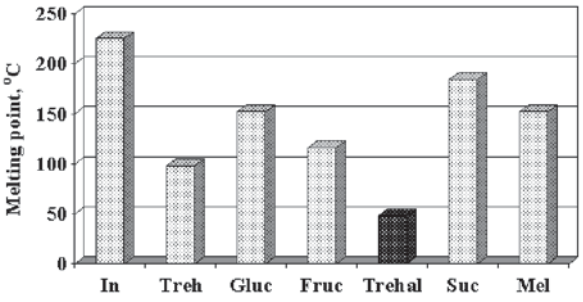
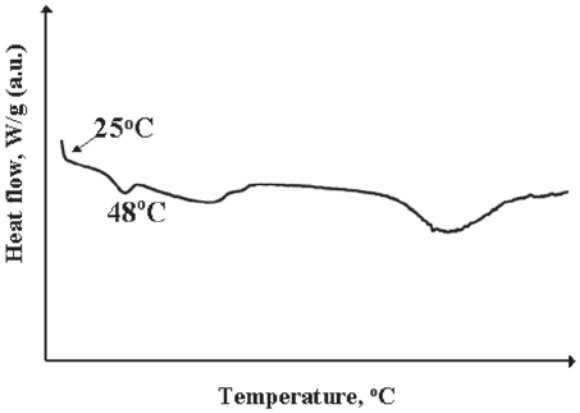


Fig. 3: Differential scanning calorimetry profile of trehalulose (heat rate 5°C min $^{-1}$ between 25°C and 250°C)



Hot plate temperature setting at 40°C:

For the three Areas, the relationship between the readings at 40°C and 53°C is linear (**Figure 6**). The statistical analysis (**Table 5**) did not show a significant interaction between temperature setting and Area. The High Speed Stickiness Detector readings at 40°C averaged 29% lower than the readings obtained at 53°C.

Hot plate temperature setting at 34°C:

The relationship between the readings at 53°C and 34°C is also linear (**Figure 7**), with the Area 3 reacting differently as reflected by a highly significant interaction between temperature and area (**Table 6**). The High Speed Stickiness Detector readings at 34°C, on average, are lower than the readings obtained at 53°C: 35.8% lower for Area 1 (mainly contaminated by whiteflies), 40.9% for Area 2 (mix whiteflies and aphids contaminations), and 60.7% for Area 3 (mainly contaminated by aphid).

Hot plate temperature setting at 27°C:

The relationship is also linear (**Figure 8**) with the Area 3 reacting differently as reflected by a highly significant interaction between temperature and area (**Table 7**). The

Fig. 4: Effect of hot plate temperature of H2SD on square-root transformed readings.

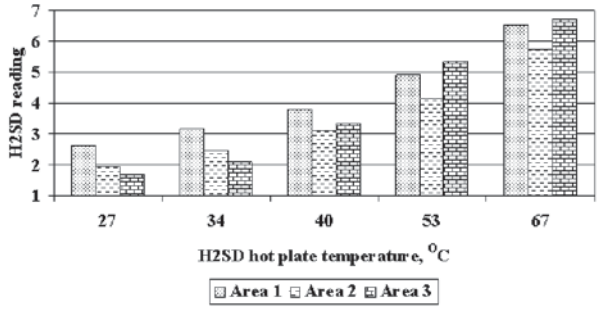


Fig. 5: Square-root-transformed readings from the H2SD at 67 vs. 53°C.

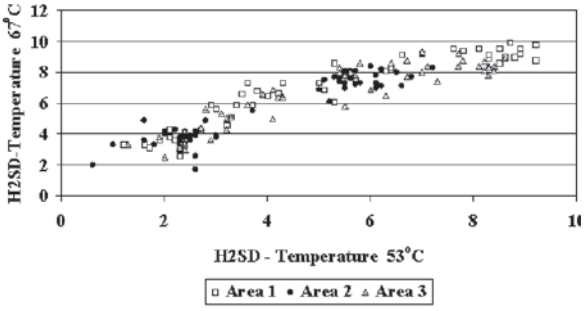
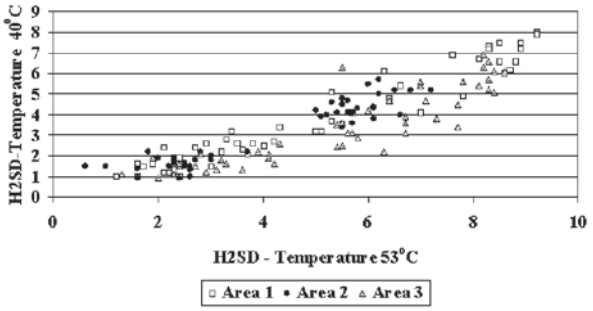


Fig. 6: Square-root-transformed readings from the H2SD at 40 vs. 53°C.



High Speed Stickiness Detector readings at 27°C, in average, are lower than the readings obtained at 53°C: 46.4% for the Area 1, 54% for the Area, and 68.7% for the Area 3. This supports our hypothesis that trehalulose is sticky even at low temperatures while melezitose is not.

Figure 9 summarizes the High Speed Stickiness Detector readings at 27°C and 53°C for the three Areas. This figure shows that for Area 1, all the cottons that tested sticky at 53°C are also sticky at 27°C. For Area 2, most of the cottons that tested sticky at 53°C are slightly sticky at 27°C. However, for Area 3 nearly all the cottons that tested sticky at 53°C are not sticky at 27°C. These results demonstrate clearly an effect of the hot plate temperature of the High Speed Stickiness Detector on the stickiness readings. They also confirm, as hypothesized earlier, that different behaviors of the honeydew deposits depend upon the origin of the contamination (whitefly vs. aphid).

Fig. 7: Square-root-transformed readings from the H2SD at 34 vs. 53°C.

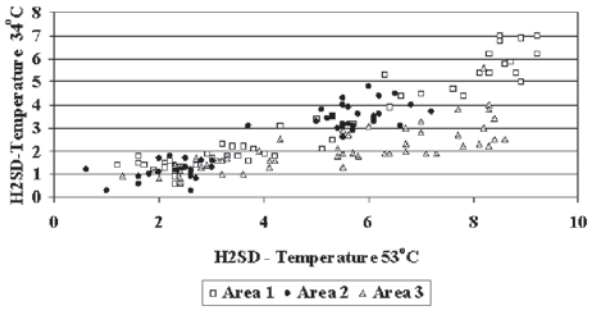


Fig. 8: Square-root-transformed readings from the H2SD at 27 vs. 53°C.

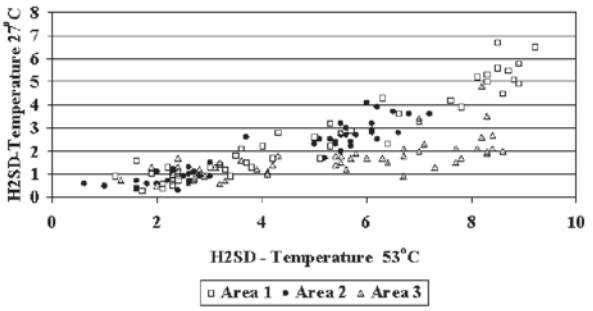


Figure 10 shows the High Speed Stickiness Detector readings for two types of cotton contaminated with whiteflies and aphids based on the high performance liquid chromatography tests. The two cottons had nearly the same number of sticky spots at 53°C (respectively 72.8 and 71.7 spots). However, by lowering the hot plate temperature from 53°C to 27°C, the two cottons reacted differently. The cotton contaminated with whiteflies honeydew remained sticky even at low temperature, while the cotton contaminated with aphids honeydew was not sticky at that temperature.

CONCLUSION

The results obtained demonstrate significant effects of the relative humidity on stickiness measurement. By testing at a lower relative humidity (55±2% instead of 65±2%), the High Speed Stickiness Detector readings were significantly lower, 23.2%.

The results also demonstrate that by testing at high temperature (67°C), the High Speed Stickiness Detector readings on the contaminated cottons are higher than at 53°C with no significant interaction Area x Temperature. However, at 27°C significant interactions Area x Temperature was noticed. The High Speed Stickiness Detector readings at this temperature were lower: 46.4% for the Area 1, 54% for the Area 2, and 68.7% for the Area 3. This suggests that the origin of the contamination (whiteflies vs. aphids) may have an effect on the High Speed Stickiness Detector readings. Therefore, the

Table 4: Variance analysis on the square root transformed data: effect of hot plate temperature setting (67°C) on H2SD readings.

		df†	*F*†	Probability	H2SD‡
	Intercept	1	1898.67	0.0001	
	Area	2	6.33	0.0020	
	1				5.735 a§
	2				4.950 b
	3				6.031 a
	Temperature	1	35.86	0.0001	
	53°C				4.814 b
	67°C				6.345 a
Area	Temperature	2	0.09	0.9161	
1	53°C				4.923
	67°C				6.547
2	53°C				4.155
	67°C				5.745
3	53°C				5.340
	67°C				6.723
	Error	294			

† df, degrees of freedom; *F*, variance ratio.
‡ Square root transformed.
§ Readings not followed by the same letter are significantly different with a = 5% (according to the Newman-Keuls test).

Table 5: Variance analysis on the square root transformed data: effect of hot plate temperature setting (40°C) on H2SD readings.

		df†	*F*†	Probability	H2SD‡
	Intercept	1	1161.11	0.0001	
	Area	2	3.98	0.0196	
	1				4.363 a§
	2				3.627 b
	3				4.342 a
	Temperature	1	33.19	0.0001	
	40°C				3.417 b
	53°C				4.814 a
Area	Temperature	2	1.59	0.2052	
1	53°C				4.923
	40°C				3.803
2	53°C				4.155
	40°C				3.099
3	53°C				5.340
	40°C				3.345
	Error	294			

† df, degrees of freedom; *F*, variance ratio.
‡ Square root transformed.
§ Readings not followed by the same letter are significantly different with a = 5% (according to the Newman-Keuls test).

Table 6: Variance analysis on the square root transformed data: effect of hot plate temperature setting (34°C) on H2SD readings.

		df†	*F*†	Probability	H2SD‡
	Intercept	1	1108.53	0.0001	
	Area	2	3.66	0.0268	
	1				4.042 a§
	2				3.305 b
	2				3.718 ab
	Temperature	1	101.73	0.0001	
	34°C				2.569 b
	53°C				4.814 a
Area	Temperature	2	5.24	0.0058	
1	53°C				4.923 a
	34°C				3.161 c
2	53°C				4.155 b
	34°C				2.456 cd
3	53°C				5.340 a
	34°C				2.096 d
	Error	294			

† df, degrees of freedom; *F*, variance ratio.
‡ Square root transformed.
§ Readings not followed by the same letter are significantly different with a = 5% (according to the Newman-Keuls test).

Table 7: Variance analysis on the square root transformed data: effect of hot plate temperature setting (27°C) on H2SD readings.

		df†	*F*†	Probability	H2SD‡
	Intercept	1	1008.96	0.0001	
	Area	2	3.97	0.0199	
	1				3.780 a§
	2				3.036 b
	3				3.507 ab
	Temperature	1	158.69	0.0001	
	27°C				2.075 b
	53°C				4.814 a
Area	Temperature	2	4.71	0.0097	
1	53°C				4.923 a
	27°C				2.636 c
2	53°C				4.155 b
	27°C				1.918 cd
3	53°C				5.340 a
	27°C				1.675 d
	Error	294			

† df, degrees of freedom; *F*, variance ratio.
‡ Square root transformed.
§ Readings not followed by the same letter are significantly different with a = 5% (according to the Newman-Keuls test).

Fig. 9: Readings from the H2SD for 150 bales compared at 27 and 53°C.

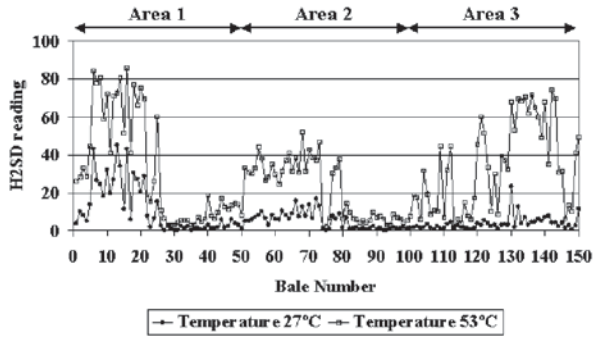
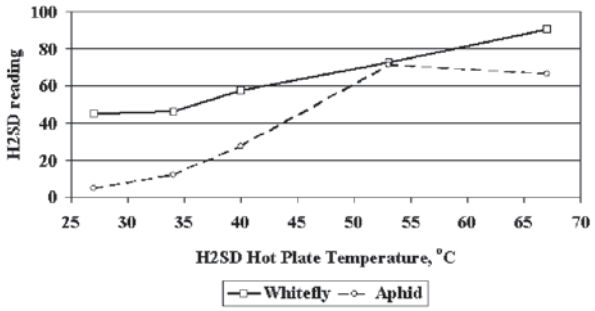


Fig. 10: Effects of the type of honeydew contamination on H2SD readings.



question of the most appropriate hot plate temperature setting for the High Speed Stickiness Detector arises. To answer this question we conducted spinning trials of sticky cottons from different origins representing both whiteflies and aphids honeydew contamination. Then, we related both productivity and yarn quality parameters to the High Speed Stickiness Detector readings performed at different hot plate temperature settings. The results of this work will be published in the near future.

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