# STATUS OF RESEARCH ON THE MEASUREMENT OF STICKINESS IN COTTON FIBERS

M. Dean Ethridge, Ph.D., Director Eric Hequet, Assistant Director This report was presented at the Engineered Fiber Selection System Conference, June 8-10, 1998, in Memphis, Tennessee.

## Introduction

Since early 1997, the International Textile Center (ITC) has been engaged in a collaborative research effort aimed at developing reliable measurements for stickiness of cotton fibers, in order to enable efficient management of this contamination problem. Critical partners in this project are Cotton Incorporated, the Cotton Program of the Centre de Cooperation Internationale en Recherche Agronomique pour le Developpement (CIRAD) in Montpellier, France, and the Cotton Division of the Agricultural Marketing Service, U.S. Department of Agriculture. Contributions and logistical support have come from several other sources; including participation by the Arizona Cotton Research and Protection Council (ACRPC), in Tempe, Arizona. This report summarizes some of the major results obtained so far and plans for obtaining further results during 1998.

### Procedures

Efforts during 1997 and early 1998 were focused on the selection, collection, and analysis of 50 bales of Texas cottons that were contaminated by varying levels of aphid honeydew. Each of the bales selected was sampled ten times at ten layers throughout. The samples were subdivided and sent to various locations where they were subjected to measurements by all available instruments for measuring stickiness. Instruments used and the locations are as follows:

 STICKY COTTON THERMODETECTOR (SCT) - This is the original, manually operated thermodetector developed by CIRAD. Instruments used were located at CIRAD and at the ACRPC.  HIGH SPEED STICKINESS DETECTOR (H2SD) - This is the prototype, automated thermodetector developed by CIRAD. Only two of these instruments are in existence, located at CIRAD and Cotton Incorporated.

- LINTRONICS FIBER CONTAMINATION TESTER (FCT) - This instrument was developed by the Lintronics Co. in Israel. In 1997, these were pre-commercial versions that were located at the Agricultural Marketing Service of USDA and at the ITC.
- HIGH PERFORMANCE LIQUID CHRO-MATOGRAPHY (HPLC) - This instrument is useful only as a research tool, but it is indispensable for identifying the sources of stickiness contamination (plant sugars vs. insect honeydew and the type of insect involved) and it is helpful in assessing the degree of contamination. HPLC measurement and analysis were done at the ITC.

The experimental design and procedures in this project made it possible to collect accurate data revealing stickiness **levels** and **variations** both **within bales** and **between bales**. Eleven of the bales (numbers 32 to 42) came from a single module; this was done to examine the levels and variations within a module. An obvious hypothesis is that, within the U.S. production system, a given number of measurements on a module will produce more reliable data than the same number of measurements on individual bales.

Replicated measurements were taken on all instruments, providing data on the variability of stickiness measurements. Results presented generally include the **maximum** values and **minimum** values, as well as the **average** values.

TEXTILE TOPICS	Published quarterly Texas Tech University
a research bulle tin on fiber and textile industries.	International Textile Center P.O. Box5888
Summer 1998 - Issue 3	Lubbock, TX 79408-5888

### Results

#### High Speed Stickiness Detector (H2SD)

Measurement results obtained with the H2SD by Cotton Incorporated are shown in Exhibit 1. These tests were run in a straightforward fashion, with the machine operator following procedures but not pausing to evaluate whether the results appear stable. Therefore, these results are indicative of measurements likely to be obtained in a commercial testing laboratory. The ranges on a few of the measurements are too wide; e.g., bale numbers 4, 7, 21, 41 and 43. Nevertheless, the average for each bale appears useful; in particular the 11 bales in the module are clearly identified. Measurement results obtained with the H2SD by CIRAD are shown in Exhibit 2. Note that the pattern of the average values is quite similar to the pattern obtained at Cotton Incorporated; however, the ranges of the measurements are much less. In fact, these ranges are quite small, suggesting that great care was taken in executing these measurements.

A chart of the **differences** between **average values** obtained at CIRAD and Cotton Incorporated verifies that agreement between these two machines was quite good (Exhibit 3). Except for one observation, the differences were quite small and were randomly distributed around the zero axis. On balance, these are encouraging results for the H2SD.

#### Sticky Cotton Thermodetector (SCT)

The manually operated SCT is known to be very operator-sensitive. The operators at CIRAD and at the ACRPC are among the most experienced in the world. Therefore, if repeatability can be obtained anywhere, it should be possible between these two places.

As with the H2SD, CIRAD produced a very precise grouping of measurements with the SCT (Exhibit 4); including a nearly perfect identification of the 11 bales in the cotton module.

Results obtained on the SCT at the ACRPC follow the same general pattern as those from CIRAD (Exhibit 5). The highs and lows are generally wider, but these are still good-looking results for this instrument. Charting the differences between average values at CIRAD and ACRPC (Exhibit 6) verifies that agreement between these instruments was pretty good, but not as good as with the H2SD. One of them was biased on the measurements of the cotton module, since all of these differences are well above the zero axis. On balance, these results from very reliable operators of the SCT reveal how hard it is to make these manual measurements travel from one place to another.

#### Lintronics Fiber Contamination Tester (FCT)

The Lintronics Fiber Contamination Tester was operated by the Agricultural Marketing Service personnel in Memphis. The results summarized in Exhibit 7 show that the instrument performed well under their operation. The ranges in the replicated measurements are quite acceptable and the average values appear to agree very well with results from the H2SD. Unfortunately, the same cannot be said for the FCT results obtained at the ITC (Exhibit 8). Neither the ranges nor the average values are acceptable. The apparent cause for this was a problem with residual contamination in the instrument.

The chart of differences between average values at the ITC versus the USDA only verifies that there was no agreement between the two FCT instruments (Exhibit 9). The ITC measurements were consistently higher than the USDA measurements on everything except for those bales in the module, where the ITC measurements were consistently lower.

### Statistical Relationships among Instruments

The correlations and linear relationships

between different measurements are important for settling issues of calibration and repeatability. Exhibit 10 shows results of regressing Cotton Incorporated's average values from the H2SD on CIRAD's average values for the same instrument. What we would like to see is a R<sup>2</sup> value very close to one, a constant term very near zero, and a slope very near one. Based on these criteria, the results are pretty good. If calibration cottons were available, a calibration procedure should enable making the slope and intercept terms approximate one and zero, respectively. The  $R^2$  value of 0.76 is not bad, but it is to be hoped that this value could be raised to 0.85 or higher.

It is a pleasant surprise that the R<sup>2</sup> value obtained by regressing Cotton Incorporated's measurements with the H2SD on the USDA's measurements on the FCT reaches 0.80 (Exhibit 11). The slope and intercept terms are not close to one and zero, but this was expected since the two machines employ different techniques in measurement. A sufficient condition for the number of sticky spots to be larger on the FCT is the fact that this instrument utilizes a much larger surface of cotton fibers than does the H2SD. Nevertheless, given high correlation levels between the two instruments it is possible, with the use of appropriate calibration cottons and procedures, to develop a functional correspondence between them.

Even more surprising is the fact that the  $R^2$ value obtained by regressing CIRAD's measurements with the H2SD on the USDA's measurements with the FCT reaches 0.90 (Exhibit 12). This indicates that these two instruments were in quite close agreement. If such agreement could be consistently repeated, calibrated measurements could eventually be developed and used with confidence.

### High Performance Liquid Chromatography (HPLC)

The analysis using the HPLC proved to be quite interesting. Using the HPLC, we detected in significant amounts the sugars named inositol, trehalose, glucose, fructose, sucrose, trehalulose, and melezitose. Contamination with aphid honeydew is revealed by high percentages of melezitose. Contamination with white fly honeydew is indicated by elevated levels of trehalulose. The melezitose levels, expressed as a percentage of total sample weight, revealed some aphid honeydew contamination in all bales, with serious contamination in about half of them (Exhibit 13). The module of cotton had significant melezitose contamination, which was expected. What did surprise, however, was that this module-which was obtained outside the Texas High Plains-also had trehalulose levels that indicated white fly contamination (Exhibit 14). Putting the two data series together, and expressing the levels as a percentage of total sugars (Exhibit 15), strongly suggests that both insects contaminated this module while only aphids contaminated the rest of the bales.

The discovery of different insect sugars raises a question about whether the different sources of contamination, in combination with machine settings and operational techniques, contributed to some of the measurement differences among instruments that were apparent for the module of cotton. Recall that there were level shifts apparent between the Sticky Cotton Thermodetectors and the FCTs when the module was involved. There was no such level shift for the H2SDs. Perhaps this was due to the fact that the H2SD is the only one of the three instruments that electronically controls the pressure applied to the samples. It is quite possible that the different insect sugars react differently to differences in pressure.

It is also noteworthy that the percentages of the total sugars accounted for by melezitose generally stayed below 20%, while that for trehalulose generally stayed below 10%. Analysis by Hendrix, et. al., (1992) of aphid and white fly honeydew indicated around 40% of total sugars present was melezitose in the aphid honeydew, while the white fly honeydew exhibited about 40% trehalulose **plus** about 17% melezitose. The fact that lower percentages of these sugars were found in these bale samples suggests that these cottons may have also had a high content of physiological sugars.

# Conclusions

The results of work done so far give encouragement that sufficiently fast and repeatable measurements of stickiness will be possible in the months ahead. This process will be repeated this year, using a hundred-bale sample from the Western U.S., and efforts will benefit from what has already been learned. Also, the first commercially available High Speed Stickiness Detector from SDL Ltd. will be utilized for testing—assuming that at least one is available soon enough to include in the project. It is clear that, for both the FCT and the H2SD, painstaking maintenance of the systems is critical for achieving either short-term or long-term stability of measurement results. Nevertheless, those of us who work constantly with the available instruments are pretty sure that we can obtain the information necessary to detect and manage stickiness. Some textile mills that have invested the time and resources necessary to reach a threshold level of confidence can say the same thing.

It should be emphasized, however, that it is a long journey from successfully using in-house stickiness measurements to reach the capability of providing the marketplace with brief, reliable data that can be used directly in the purchasing and utilization of cotton. Before this can even be contemplated, calibration procedures and calibration cottons must be institutionalized.

# References

Hendrix, D. L., Y. Wei and J. E. Leggett. 1992. Homopteran honeydew composition is determined by both the insect and plant species. *Comparative Biochemistry Physiology*, 101B, 1/2, pp. 23-27.