PROSPECTS FOR RAPID MEASUREMENT OF STICKINESS IN COTTON M. Dean Ethridge and Eric F. Hequet International Textile Center, Texas Tech University Lubbock, TX

Abstract

For several years the International Textile Center (ITC) has been deeply involved in a collaborative research effort aimed at developing reliable measurements for stickiness of cotton fibers. Results of work done in 1998 are very encouraging; they indicate that fast and repeatable measurements of stickiness are feasible. Among the remaining issues to be resolved are the very important ones of instrument calibration and long-term stability of the measurements.

Introduction

Efforts to develop reliable measurements for stickiness of cotton fibers have been building for more than a decade. These efforts became quite collaborative in the mid-1990s. The results reported here were made possible by a partnership with Cotton Incorporated (CI) in the USA and with the Cotton Program of CIRAD (*Centre de Cooperation International en Recherche Agronomique pour le Developpement*) in France.

In 1996, fifty bales were selected from Texas using procedures for getting a wide range of stickiness due to aphid (Aphis gossypii) infestation. Eleven of these bales were from one module. Analysis of this cotton was completed in 1997-98; procedures and results from this study have been reported (Hequet, Ethridge & Wyatt, March 1998; Ethridge & Hequet, June 1998; Hequet, Ethridge & Wyatt, September 1998). Information gained from it was used in planning and conducting the study reported here.

The 1998 study involved the selection of a hundred more bales; fifty bales from Arizona and fifty from California. The selection process ensured getting a wide range of stickiness due to contamination from the white fly (Bemisia tabaci). Within the California bales, 23 of them came from one module.

Procedures

The bales were broken and layered. Ten samples were taken from each bale. Each of these samples was then divided into sub-samples, which were sent to each of the cooperators providing instrument measurements of stickiness.

> Reprinted from the Proceedings of the Beltwide Cotton Conference Volume 1:56-60 (1999) National Cotton Council, Memphis TN

The sampling and testing procedures were the same as with the earlier study on Texas cottons. They made it possible to determine the following:

- Relationships among the measurements obtained from the various instruments and between the two states;
- Variability of stickiness measurements within samples;
- Variability of stickiness measurements within bales;
- Variability of stickiness measurements within modules.

It should be emphasized that the fiber samples were selected in a manner to get a wide range of stickiness. They are not "representative" of the stickiness of the cotton in Arizona and California, just as the earlier samples from Texas were not representative of the state. Getting "statistically representative samples" would require a completely different sampling procedure.

As with the earlier study, all analyses on data from the two available types of instruments were done using a square root transformation. This is due to the Poisson-like distribution of the stickiness measurements from these instruments.

It should be noted that, preceding the formal efforts in the second phase of this work and in collaboration with the company Lintronics Ltd., a substantial effort was devoted to verifying and dealing with a problem of residual contamination in the Lintronics instrument, the Fiber Contamination Tester (FCT). In the process, it was found necessary to make alterations on the crush rolls and on the cleaning system of the ITC's machine. After these alterations, comparative tests between the FCT and the Card machine confirmed that the adjustments were effective in achieving agreement between them.

Results

As of this report, stickiness data have been provided by the following instruments and cooperators:

- Carding machine ITC
- FCT (Fiber Contamination Tester) ITC
- H2SD (High Speed Stickiness Detector), prototype CIRAD and CI
- H2SD (High Speed Stickiness Detector), commercial release ITC
- HPLC (High Performance Liquid Chromatography) ITC

The data provided by CI from its prototype H2SD is incomplete as of this report, because we did not receive all of it in time for inclusion. CI has just received the new, commercial release of the H2SD (just like the one owned by the ITC) within its laboratory; measurements from it will be provided within the next few months. Also, Calcot Ltd. will produce measurements from another FCT within a few months. All of these measurement results will eventually be included in the analysis.

Insights from HPLC Analysis

The HPLC 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. Its use enabled detection of significant amounts of 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 percentage of total sugars among the three states studied is shown in Figure 1. The level is lowest in Texas and highest in California. But the composition of the sugars in each of these states is quite different. In Texas the plant sugars glucose and fructose are dominant (Figure 2). In Arizona the insect sugars Trehalulose and Melezitose are dominant (Figure 3). In California both the plant and insect sugars are high (Figure 4). Yet the sticky performance, as indicated by the ITC's card test, is almost the same among the regional cottons (Figure 5).

These results impress upon us the need to understand the synergies among the various sugars contributing to the sticky performance of cotton in textile processing. The calibration of high-volume measurements of sticky performance—indeed, the interpretation of meanings of such measurements—will require the data provided by the HPLC and by other laboratory techniques.

Relationships Among Instruments and Between States

Regressing measurements from the ITC's FCT on measurements from the ITC's H2SD results in a coefficient of correlation between the two measurements of 0.97 (Figure 6). This is an excellent result. The slope coefficient for the regression equation is much above one, which implies that the FCT is counting more sticky spots than the H2SD. This is to be expected, because the FCT utilizes a much larger surface of cotton fibers than does the H2SD. But the high correlation between the two instruments assures that a workable correspondence between the measurements could be developed.

Regressing measurements from the ITC's FCT on measurements from the CIRAD's H2SD prototype reveals non-linear results, which lowers the coefficient of correlation between the two measurements to 0.89 (Figure 7). The same non-linearity phenomenon was evident when measurements from the ITC's H2SD are regressed on CIRAD's H2SD prototype (Figure 8). It was quickly observed that the non-linearity was associated with the two states. Thus, by splitting the bale samples into two subsamples from Arizona and from California, the relationships become linear and have very high correlation coefficients (Figures 9 and 10). However, they are *different* linear relationships, as revealed by the divergent slope and offset coefficients.

Data from CI's H2SD prototype are about half completed for both states. Using these data, a regression of measurements from CIRAD's H2SD prototype on them reveals a very good linear relationship (r = 0.97) between the two prototype machines (Figure 11). Therefore, it appears that these prototype instruments are measuring the same differences between the states.

Given these results, it is useful to look at the *average* values for stickiness between the two states that are given by the different instruments. It has already been observed that the ITC's card values indicated no significant difference between the states (Figure 5). Likewise, there is no significant difference for the ITC's FCT (Figure 12) or for the ITC's H2SD (Figure 13). However, there are significant state differences for both of the prototype H2SDs (Figure 13). (Remember that the results for CI's H2SD prototype are tentative, because the data are incomplete.)

Taken together, these (incomplete) results suggest that the H2SD prototypes are not tracking results from *either* the new, commercial H2SD *or* from the FCT. First an analysis must be completed using data from CI's H2SD prototype, from CI's new H2SD, and from Calcot's FCT data. Then the basis for any differences between the prototype H2SDs and the new H2SDs will require further investigation.

Variability within Samples

The standard deviations of the measurements within samples are summarized for each state in Table 1. Comparing between states, it is seen that the standard deviations are virtually the same for every instrument except the FCT. The differing levels for the FCT are troublesome, in the same way that the differing slopes were troublesome for the H2SD. The causes and remedies for this situation will require further investigation.

The higher standard deviation values for the FCT are not surprising, because the range of sticky spots counted is greater than for the other measurements. Likewise, the lower values for the card are due to the very constricted range of measurements taken with this machine.

Variability within Bales

It is very encouraging to observe that the standard deviations of the measurements *between* samples within a bale are smaller than the standard deviation *within* samples (Table 2 vs. Table 1). This means that, within the United States, the traditional sampling technique used for HVI classification will probably also be valid for stickiness classification. The within-bale variability is closely related

to production practices (cotton field size, varieties grown, harvesting techniques, ginning techniques, etc.); therefore, it cannot be concluded that USDA-type sampling will work everywhere.

Variability within Modules

The mean values of the stickiness measurements for each of the 23 bales in the module, as measured by the three instruments, are shown in Figure 14. It clearly shows that the variability of the stickiness measurements among bales within a module is quite low for all instruments. This confirms the previous results obtained using Texas cottons. While it will be necessary to confirm this on a large number of modules, these results give reason for optimism that module averaging will be appropriate for large-scale classification of stickiness in cotton.

Operational Efficiency of Instruments

The reliability and repeatability of measurements from the FCT can be assured only by careful cleaning and constant monitoring of the machine. As a result, it cannot be said that the FCT performed as a truly high-volume instrument during the testing done for this project. The time required to adequately operate the H2SD was only about one-fourth of the time spent with the FCT. The two basic reasons for this difference are (1) the design of the H2SD minimizes the problems from residual contamination by previous samples and (2) the H2SD has electronic control mechanisms that regulate heat and pressure variables.

Conclusions

Both the FCT and the commercial version of the H2SD are able to provide reliable measurements of stickiness. Based on limited experience to date, it appears that the H2SD may be able to provide measures that are both more rapid and more repeatable.

Results have consistently indicated that USDA sampling for cotton classing will also be adequate for stickiness measurement in the United States. Furthermore, results so far indicate that module averaging would work quite well for stickiness measurement in the U.S.

Additional research is needed to confirm the adequacy of module averaging and to determine the threshold levels of stickiness that are relevant to providing authoritative guidance in cotton marketing and in the management of textile manufacturing processes. Then, before full-fledged commercialization of stickiness measurements can be feasible, the issues of instrument calibration and long-term stability of the measurements must be settled.

Acknowledgment

The financial support given by Cotton Incorporated and the Texas Food and Fiber Commission to realize this project is gratefully acknowledged. Appreciation is also due to the Plains Cotton Cooperative Association and to Calcot Ltd., for expert assistance and for substantial in-kind contributions toward the successful execution of the project.

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Table 1. Average Standard Deviations within Samples

Instrument	Arizona	California
ITC's Card	0.279	0.202
ITC's FCT	1.521	1.140
ITC's H2SD	0.855	0.882
CIRAD's H2SD	0.790	0.772
CI's H2SD	0.757*	0.759*
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*Based on incomplete data.

Table 2. Average Standard Deviations within Bales	Table 2	. Average	Standard	Deviations	within	Bales
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Instrument	Arizona	California
ITC's Card	0.227	0.356
ITC's FCT	1.012	0.796
ITC's H2SD	0.783	0.911
CIRAD's H2SD	0.553	0.461
CI's H2SD	0.716*	0.746*

*Based on incomplete data.



Figure 1. HPLC Total Sugars



Figure 2. HPLC Profile: Texas Bales





Figure 8. ITC's H2SD vs Cirad's H2SD



California SQRT ITC's H2SD 9 8 7 6 5 4 3 2 1 SD ITC 1.33 H2SD CIRAD + 0.92 r = 0.969

2 5 0 1 3 4 7 SQRT CIRAD's H2SD

Figure 10 ITC's H2SD vs Cirad's H2SD



Figure 11. CIRAD's H2SD vs CI's H2SD





Figure 13. Average Values for H2SDs



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