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Fourier Transform Infrared Analysis of Trehalulose and Sticky Cotton Yarn Defects Using ZnSe–Diamond Universal Attenuated Total Reflectance

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ABSTRACT

The universal attenuated total reflectance Fourier transform infrared (UATR-FTIR) equipped with a ZnSe–diamond composite crystal accessory was used to investigate the implication of trehalulose in the formation of yarn defects during rotor spinning of moderately sticky cotton mixes. One cotton bale contaminated with aphid honeydew and one contaminated with whitefly honeydew were selected. Mixes having moderate levels of stickiness were prepared from these two bales by mixing sticky cotton with non-sticky cotton. These cotton mixes were processed and the yarn defects were collected and analyzed using UATR-FTIR. The results obtained showed a high correlation between the FTIR spectra of the yarn defects from the whitefly honeydew-contaminated mixes and the spectra of artificial reconstituted whitefly honeydew. No significant difference was seen between the spectra of yarn defects from aphid honeydew-contaminated cotton and those of non-contaminated cotton. The FTIR and Raman studies of the hydration process of trehalulose showed a drastic impact of moisture absorption on the intensities of the vibration modes of trehalulose. These results all indicate that trehalulose is the primary concern when forming yarns from cotton with moderate honeydew contamination.

Cotton stickiness caused by excess sugars on the lint, from the plant itself [4] or from insects [9], is a very serious problem that affects all segments of the cotton industry [6, 11]. Stickiness is a worldwide contamination problem with around one-fifth of the world production affected to some degree [10]. The sugar composition of water extracts from honeydew-contaminated cottons analyzed by high-performance liquid chromatography (HPLC) is extremely complex. Among the identifiable sugars, the disaccharide trehalulose and the tri-saccharide melezitose are specific to insects and are not found in the plant. In general, a high percentage of melezitose along with a low percentage of trehalulose reveals the presence of aphid (*Aphis gossypii* Glover) honeydew. Conversely, a dominance of trehalulose indicates contamination by whitefly (*Bemisia argentifolii* Bellows and Perring [= *B. tabaci* (Gennadius) strain B]) honeydew. Hendrix *et al.* [3] analyzed the honeydew from insects and found that the aphid honeydew contained around 38.3% melezitose plus 1.1% trehalulose, while the whitefly honeydew contained 43.8% trehalulose plus 16.8% melezitose. However, these relative percentages will vary depending on the environmental or feeding conditions. In other studies, it has been demonstrated that the severity of stickiness incidents in the textile mill is related to the type of sugars present on the lint [8]

We reported in previous work on the implication of trehalulose in the residue build-up on the textile equipment during processing of moderately sticky cotton [3]. We have demonstrated that because of its high hygroscopicity, low-melting point, and film-like structure (hydrated form), trehalulose was the main source of concern in textile processing. It slowly contaminates the machinery: first sticking to surfaces, then catching dust, silica, etc.

In this work, our objective was to evaluate the usefulness of the universal attenuated total reflectance Fourier transform Infrared (UATR-FTIR) equipped with a ZnSe–diamond composite crystal accessory to investigate the implication of trehalulose in the formation of the yarn defects during processing of moderately sticky cotton. The advantage of ZnSe–diamond sampling accessory is that no sample preparation is required and it is not destructive (the measurements are performed directly on the yarn).

Experimental

MATERIALS

One commercial cotton bale contaminated with aphid honeydew and another contaminated with whitefly honeydew were selected, based on trehalulose and melezitose.

TABLE I. High-performance liquid chromatography results on aphid honeydew- and whitefly honeydew-contaminated bales.

	I	G	F	Treh.	S	M	Total sugars
Aphid honeydew contaminated cotton:							
% of the fiber weight	0.067	0.162	0.209	0.001	0.010	0.016	0.465
% of the total sugars	14.4	34.8	44.9	0.21	2.2	3.4	
Whitefly honeydew contaminated cotton:							
% of the fiber weight	0.068	0.071	0.134	0.244	0	0.109	0.626
% of the total sugars	10.8	11.3	21.4	38.9	0	17.4	

I, Inositol; G, Glucose; F, Fructose; Treh., Trehalulose; S, Sucrose; M, Melezitose.

tose contents as determined by HPLC (Table I). Their stickiness levels were evaluated with the high speed stickiness detector (H2SD). The bale contaminated with aphid honeydew had an H2SD reading of 21, while the bale contaminated with whitefly honeydew had an H2SD reading of 76. In addition, two bales of non-contaminated cotton were selected for mixing with the contaminated cotton, in order to obtain mixes that exhibited moderate stickiness levels. The mix referenced as "mix aphid" was obtained by mixing 150 pounds of aphid honeydew-contaminated cotton with 150 pounds of non-sticky cotton. The mix referenced as "mix whitefly" was obtained by mixing 257 pounds of non-sticky cotton with 43 pounds of whitefly honeydew-contaminated cotton. Each sample was tested with the high volume instrument (HVI 900A; Uster, Knoxville, TN) and the H2SD. Table II shows the HVI and H2SD readings of the two mixes.

HIGH SPEED STICKINESS DETECTOR

The high-speed stickiness detector is an automated instrument derived from the sticky cotton thermodecator (CIRAD, Montpellier, France), which was approved as a reference test by the International Textile Manufacturers Federation in 1994 [2]. Frydrych *et al.* first described the H2SD in 1994 [1]. The general procedure is as follows: after conditioning the fiber samples for at least 12 hours, a 3.5-g specimen of lint is fed into the instrument to be mechanically opened. It forms a pad of 130 mm by 170 mm, which is then automatically transferred onto a strip of aluminum foil originating from a roll. The aluminum is rolled along a conveyor belt, which delivers sample sequentially to each station. The aluminum foil is rolled up at the other end of the machine. The H2SD has four distinct stations. The first is a hot press, where a fixed

amount of pressure is applied for 25 seconds with the heating element (53°C) in contact with the cotton. The second station is a cool press, where the same fixed pressure is applied for 25 seconds at ambient temperature. This fixes the sticky points to the aluminum foil. Next follows the cleaning station, where the non-sticky material is removed by a combination of a cleaning roll and suction. The fourth station is an image analysis chamber, where the sticky points are automatically counted and reported.

HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

Each fiber sample was placed into a plastic bag and 20 mL of 18.2-megohm water was added. A sample of the aqueous solution was taken from the bag with a 10-cm⁻³ syringe (Becton Dickinson and Company, Franklin Lakes, NJ), to which a 0.2-μm filter (nylon membrane polypropylene housing) (National Scientific, Scottsdale, AZ) was attached. A 1.5-mL filtered sample was deposited into 1.5-mL autosampler vial (C4013-15A; National Scientific). Sugars were separated on the columns (CarboPac PA1 anion-exchange analytical columns; Dionex Corporation) in series with a gradient eluent system. Eluent 1 was 200 mM NaOH and eluent 2 was 500 mM sodium acetate (C₃H₃NaO₂, 3H₂O) and 200 mM NaOH. Three replications were performed on each sample. The results were expressed in percentage of the fiber weight.

UATR-FTIR AND RAMAN SPECTROSCOPIES

FTIR Spectrum-One equipped with a universal attenuated total reflectance (UATR) accessory was used in this research (Perkin Elmer, USA). The UATR is an internal reflection used to simplify the analysis of solids,

TABLE II. HVI and H2SD readings of the four mixes.

Mix ID	Mike	UMHL (mm)	Unif. (%)	Strength (cN/tex)	Elon. (%)	Leaf	Rd (%)	+b	H2SD readings
Mix aphid	4.5	24.9	78.6	23.9	6.8	1.0	75.6	11.0	12.7
Mix whitefly	4.8	25.4	79.3	23.4	6.7	1.0	76.1	10.8	15.3

powders, gels, and liquids. It is a rapid and flexible FTIR sampling technique, which consists of placing a sample on top of the ZnSe–diamond composite crystal with a high refractive index. The infrared radiation from the instrument is passed into the accessory and up into the crystal. It is then reflected internally in the crystal, and then back toward the detector which is located in the spectrometer. When the beam is reflected within the crystal, it penetrates into the sample by approximately 2 μm . Figure 1 illustrates this process. The mid-infrared detector is a deuterated tri-glycine sulphate (DTGS). Sugars and yarn defects were placed on top of the ZnSe–diamond crystal. Pressure was applied on the sample to ensure a good contact between the sample and the incident IR beam and to prevent loss of the IR beam. The FTIR spectra were collected at a spectrum resolution of 4 cm^{-1} , with 32 scans, over the range of 650–4000 cm^{-1} . A background scan of clean ZnSe–diamond crystal was acquired before acquiring the spectra of the sample. The Perkin-Elmer software was used to perform the necessary spectra correlation and peak integration.

FIGURE 1. Principle of the UATR-FTIR.

Raman spectra of trehalulose were collected using a Raman System 2001 equipped with a miniature fiber optic with a 785 nm laser (Ocean Optics, USA).

HYDRATION PROCESS OF TREHALULOSE

Trehalulose crystal was dehydrated at room temperature under vacuum for 48 hours. Then it was deposited on the UATR–ZnSe crystal and a pressure was applied to ensure a good contact between the sample and the IR beam. The corresponding FTIR spectrum was recorded immediately between 650 and 4000 cm^{-1} using 32 scans with a spectrum resolution of 4 cm^{-1} . This spectrum constitutes the FTIR spectrum at $t = 0$. Then, an automatic acquisition was performed to collect the FTIR

spectra at different hydration times (from 0 to 300 hours with 5-min increments) at $65 \pm 2\%$ relative humidity and $21 \pm 1^\circ\text{C}$. The spectra were analyzed and the peaks that show a significant increase with the hydration time (located at 3280 and 1018 cm^{-1}) were integrated to get the peak intensities. All measurements were performed without changing the amount of trehalulose or the pressure on the top of the ZnSe crystal. Thus, any change observed in the FTIR spectra of trehalulose during the hydration process is attributed to the effect of the humidity.

SPINNING EXPERIMENTS

The mechanical process used to make the yarn is described in Figure 2 [6]. The opening, carding, drawing, roving, and rotor spinning machines used were all industrial machines. The yarn manufactured was $26.84 \times 10^{-6} \text{ kg m}^{-1}$ (26.84-tex or 22 English number) on a Rieter R20 rotor spinning frame. Ten spinning positions were used and each mix was run for 20 hours. After each end-down, the two yarn ends were collected for further microscopic and infrared evaluation.

FIGURE 2. Outline of the mechanical process for rotor spinning tests.

ARTIFICIAL HONEYDEW MIXES

The selected sugars used to prepare artificial honeydew were inositol, fructose, glucose, sucrose, trehalulose, and melezitose. Trehalulose was obtained from Cornell University; the other sugars were purchased from Sigma Chemical Company (St. Louis, MO). The mix referenced as "mix 1" was composed of 1% trehalulose, 15% sucrose, 15% glucose, 19% fructose, 45% melezitose, and 5% inositol. The mix referenced as "mix 45" was composed of 45% trehalulose, 15% sucrose, 10% glucose, 10% fructose, 15% melezitose, and 5% inositol. In preparing these two mixes, the dehydrated sugars were weighed and mixed with a spatula, then the open containers containing mixed sugar samples were stored in controlled atmosphere, $65 \pm 2\%$ relative humidity and $21 \pm 1^\circ\text{C}$ for more than 1 month (720 hours) [5]. The FTIR spectra of mix 1 and mix 45 were acquired and stored in the FTIR software library.

SCANNING ELECTRON MICROSCOPE

The yarn defects causing ends-down were identified and collected. The samples were mounted in the stub and coated with a layer of gold by means of thermal evaporation in a vacuum coating unit. They were examined in the scanning electron microscope (Model S500; Hitachi, Tokyo, Japan) using an accelerating voltage of 20 kV.

Results and Discussion

UATR-FTIR AND RAMAN STUDY OF TREHALULOSE HYDRATION

Sugars belong to the carbohydrate class and are hydrophilic because of several hydroxyl groups that establish hydrogen bonding with water molecules. In previous work [5], we evaluated the hygroscopic properties of sugars found on sticky cotton (inositol, glucose, fructose,

trehalulose, sucrose, and melezitose). Among the sugars tested, trehalulose has the highest hygroscopicity and very fast hydration kinetic. After the equilibrium was reached, at $65 \pm 2\%$ relative humidity and $21 \pm 1^\circ\text{C}$, the amount of absorbed water corresponded to 3 molecules of H_2O per molecule of trehalulose. Furthermore, the differential scanning calorimetry study of thermal properties of these sugars showed that trehalulose has a very low melting point [5]. These characteristics explain why we expected trehalulose to accumulate on the spinning equipment. In fact, the HPLC tests performed on the residues collected from the textile equipment after processing sticky cottons showed very high concentrations of trehalulose [5], thereby confirming the hypothesis of selected accumulation of trehalulose on the textile equipment. It follows that trehalulose could also become a precursor for yarn defects during processing of contaminated cotton with whitefly honeydew.

Hydrated trehalulose and artificial whitefly honeydew mix were analyzed by scanning electron microscopy. The micrograph of hydrated trehalulose shows an amorphous film-like structure appearance (Figure 3). The artificial honeydew mix has a similar appearance, although it contains only 45% of trehalulose. The high hygroscopicity of trehalulose leads to the formation of a polymeric film that readily adheres to the textile equipment. It also causes cotton fibers to stick together initiating the formation of yarn defects.

Figure 4 shows the UATR-FTIR spectrum of trehalulose in its dehydrated state. All vibration bands are very weak (absorption < 0.009). This could mean that infrared vibrations of trehalulose are not active in this state. However, the exposure of trehalulose to humidity resulted in an increase of the intensity of all vibration bands with increasing hydration time, particularly for the 3280 and 1018 cm^{-1} bands (Figure 5). After 77 hours of exposure to 65% relative humidity at 21°C , the intensity

FIGURE 3. Scanning electron microscopy of: (a) hydrated trehalulose, (b) artificial whitefly honeydew mix.

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of the FTIR spectra of hydrated trehalulose is approximately 60 times higher than the intensity of the spectra of trehalulose in dehydrated state for the peak at 3280 cm^{-1} .

FIGURE 4. FTIR spectra of dehydrated trehalulose.

FIGURE 5. FTIR spectra of trehalulose at different hydration time: (a) $t = 7$ hours, (b) $t = 21$ hours, (c) $t = 31$ hours, (d) $t = 57$ hours, (e) $t = 70$ hours, and (f) $t = 77$ hours.

Since infrared and Raman spectroscopies complement each other, we investigated the effect of humidity on the Raman vibrations of trehalulose. Thus, the very weak infrared vibrations obtained for trehalulose in the dehydrated state could mean that the vibration modes of trehalulose are not active in infrared but are active in Raman. Figure 6 shows a comparison between the Raman spectra of dehydrated trehalulose and hydrated trehalulose after only 10 minutes exposure to at $62 \pm 2\%$

relative humidity and $23 \pm 1^\circ\text{C}$. In contrast to the FTIR spectra of hydrated trehalulose, the intensity of the Raman vibration bands decreased when trehalulose was exposed to humidity.

FIGURE 6. Raman spectra of trehalulose: (a) dehydrated, and (b) after 10 minutes at $62 \pm 2\%$ relative humidity and $23 \pm 1^\circ\text{C}$.

The absorption of water on trehalulose sugar was measured by observing the FTIR peaks at 3280 and 1018 cm^{-1} . The peak located at 3280 cm^{-1} is attributed to the O-H stretching vibration of absorbed water molecules, while the peak located at 1018 cm^{-1} is attributed to C-O stretching vibration in the primary alcohol ($-\text{H}_2\text{C}-\text{OH}$). In order to obtain the integrated intensity at each hydration time, the peak located at 3280 cm^{-1} was integrated from 3000 and 3700 cm^{-1} and the peak at 1018 cm^{-1} was integrated from 1095 and 941 cm^{-1} . The integrated intensities I_{3280} and I_{1018} as a function of hydration time are shown in Figure 7a. The evolution of I_{3280} and I_{1018} may be divided into two regions, from 0 to 50 hours and from 50 to 300 hours. For both integrated intensities, a first equilibrium was reached at 25 hours and a second at 150 hours of hydration. This behavior suggests that two mechanisms are probably involved in the effect of water on trehalulose. As shown in Figure 7b, the weight gain of trehalulose in percent [5] and the integrated intensity I_{3280} of the peak at 3280 cm^{-1} follow the same trend for the hydration time < 50 hours. This could be attributed to a simple absorption mechanism of water molecules on trehalulose (three molecules of water per molecule of trehalulose). The weight gain of trehalulose reached equilibrium and no further increase is noticed (not shown). However, the integrated intensity I_{3280} continued to increase after 50 hours of hydration (Figure 7a), which indicates that another mechanism takes place after the water absorption process.

FIGURE 7. (a) Integrated intensities I_{3280} and I_{1018} versus hydration time; (b) weight gain and integrated intensity I_{3280} versus hydration time.

UATR-FTIR STUDY OF YARN DEFECTS

During rotor spinning of the cotton mixes contaminated by aphid honeydew and whitefly honeydew, the ends-down were collected and the yarn defects were examined with scanning electron microscopy. Figure 8 shows micrographs of a representative yarn defect caus-

ing ends-down during rotor spinning of the cotton containing aphid honeydew versus the cotton containing whitefly honeydew. The micrographs reveal that the cotton fibers in the yarn defects resulting from the aphid honeydew-contaminated mix are entangled with crystals, while the cotton fibers in the yarn defects resulting from whitefly honeydew-contaminated mix are embedded in amorphous matrix acting like glue.

These yarn defects were analyzed using a ZnSe-diamond UATR as described in the materials and methods section. Figure 9 shows representative FTIR spectra of the yarn defects causing ends-down during processing of the two contaminated cotton mixes. The FTIR spectrum of the cotton yarn is also shown as a control. The spectrum of the yarn defects resulting from aphid honeydew is similar to the spectrum of non-contaminated cotton yarn (control). However, for the yarn defect of the whitefly honeydew-contaminated mix, the vibration bands at 3280 and 1018 cm^{-1} are very strong. Therefore, these two vibrations could be used to distinguish between the two types of contamination. Three groups of spectra are distinguished: the yarn group (which includes the spectra of cotton yarns without any defect), the aphid group (which includes the spectra of the yarn defects from aphid honeydew-contaminated cotton mix), and the whitefly group (which includes the spectra of yarn defects from whitefly honeydew-contaminated cotton mix). To differentiate between these spectra, we analyzed 34 samples for each group and recorded their FTIR spectra. The peaks located at 3280 and 1018 cm^{-1} were integrated respectively from 3700 to 3000 cm^{-1} and from 1095 to 941 cm^{-1} . Analysis of variance was performed, taking the group as a treatment and the integrated intensities I_{3280} and I_{1018} as factors. The results summarized in Table III show significant effect of the type of contamination (aphid versus whitefly) on both integrated intensities I_{3280} and I_{1018} . As shown in Figure 10, there is no

FIGURE 8. Scanning electron microscopy of yarn defects causing ends-down during rotor spinning of: (a) aphid honeydew contaminated mix; and (b) whitefly honeydew contaminated mix ($\times 1000$).

significant difference between the integrated intensities I_{3280} and I_{1018} of the spectra of yarn defects from aphid honeydew-contaminated cotton mix and the spectra of the cotton yarn without defects. However, for yarn defects of the mix contaminated with whitefly honeydew the integrated intensities of both peaks are significantly higher. Therefore, according to these results and for the stickiness level tested, the contamination with aphid honeydew seems to cause no detrimental effects on yarn quality. Indeed, the detailed analysis of yarn quality showed that aphid honeydew contamination did not translate into negative effects on either yarn quality or productivity up to 26 H2SD spots [7]. However, for the whitefly honeydew-contaminated cotton even 12 H2SD spots had drastic negative impact, producing unacceptable yarn quality and productivity loss [7].

FIGURE 9. FTIR spectra of cotton yarn and yarn defects causing ends-down: (a) cotton yarn without defect; (b) yarn defect from aphid honeydew-contaminated cotton; (c) yarn defect from whitefly honeydew-contaminated cotton.

The FTIR spectra of the yarn defects causing ends-down were compared with the spectra of artificial honeydew mix 1 and mix 45, previously stored in the library, to find the closest match. The matching coefficient between the spectra of the yarn defects produced during rotor spinning of whitefly honeydew-contaminated mix exhibit higher correlation (0.84) with the reconstituted whitefly honeydew mix 45. This is in agreement with HPLC results of the cotton fibers used to make this mix (Table I). However, the matching coefficient between the spectra of the yarn defects from aphid honeydew-contaminated mix and reconstituted aphid honeydew mix 1 was only 0.61. These results are in agreement with the previous finding that aphid-contaminated cotton seems to cause no yarn defects for the level of stickiness tested.

TABLE III. Variance analysis: Effect of yarn defect type on the integrated intensities I_{3280} and I_{1018} .

	df†	Peak 3280 cm ⁻¹		I_{3280}
		F†	Probability	
Intercept	1	2387.044	0.000001	
Group	2	452.775	0.000001	
Cotton yarn				19.822 b‡
Mix aphid				19.954 b
Mix whitefly				66.104 a
Error	98			

	df†	Peak 1018 cm ⁻¹		I_{1018}
		F†	Probability	
Intercept	1	1523.679	0.000001	
Group	2	198.014	0.000001	
Cotton yarn				8.744 b‡
Mix aphid				10.814 b
Mix whitefly				25.649 a
Error	98			

† df, degrees of freedom; F, variance ratio. ‡ Values not followed by the same letter are significantly different with $\alpha = 5\%$ (according to the Newman-Keuls test).

FIGURE 10. Comparison between integrated intensities (I_{3280} and I_{1018}) for cotton yarn without defect (Cotton yarn); yarn defects from aphid honeydew-contaminated cotton (Mix Aphid); yarn defects from whitefly honeydew-contaminated cotton (Mix whitefly); artificial whitefly honeydew (Mix 45); and trehalulose (Vertical bars denote 0.95 confidence intervals).

Conclusions

The UATR-FTIR was successfully used to study the hydration process of trehalulose and analyze the yarn defects causing ends-down during rotor spinning cotton mixes with moderate honeydew contamination. The exposure of trehalulose to the humidity resulted in a drastic change on the FTIR and Raman spectra. The absorption of water on trehalulose molecules could initiate structural and/or conformational changes. The results also showed very good correlation between the FTIR spectra

of the yarn defects obtained during rotor spinning of the whitefly honeydew-contaminated cotton mix (trehalulose-rich) and the artificial whitefly honeydew mix. This indicated that trehalulose is the dominant sugar in the yarn defects. These results confirmed our previous conclusions that trehalulose is the main concern during processing cotton with moderate honeydew contamination.

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Literature Cited

1. Frydrych, R., Hequet, E., and Cornuejols, G., A High Speed Instrument for stickiness Measurement, in "Proc. 22nd International Cotton Conference," 3–5 March 1994, International Textile Manufacturers Federation, Bremen, Germany, pp. 83–91, 1994.
2. Frydrych, R., and Hequet, E., Standardization Proposal: The Thermodetector and its Methodology, in "Proc. International Comm. Cotton Testing Methods," Zurich, Switzerland, International Textile Manufacturers Federation Bremen, Germany, pp. 97–102, 1998.
3. Hendrix, D. L., Wei, Y.-A., and Leggett, J. E. Homopteran honeydew sugar composition is determined by both the insect and plant species. *Comp. Biochem. and Physiol.* **101B**(1/2), 23–27 (1992).
4. Hendrix, D. L., Steele, T. L., and Perkins Jr., H. H., *Bemisia* Honeydew and Sticky Cotton, in "Bemisia: Taxonomy, Biology, Damage, Control and Management", D. Gerling, and R. T. Mayer, Eds. Intercept, Andover, UK, pp. 189–199, 1995.
5. Hequet, E., and Abidi, N., Processing Sticky Cotton: Implication of Trehalulose in Residue Build-up. *J. Cotton Sci.* **6**(1), 77–90 (2002).
6. Hequet, E., Ethridge, D., Cole, B., and Wyatt, B., How Cotton Stickiness Measurements Relate to Spinning Efficiency, in "Proc. 13th Annual Engineer Fiber Selection System Conference," 17–19 April 2000, Cotton Inc., Cary, NC, pp. 99–121 (2000).
7. Hequet, E., Abidi, N., and Ethridge, D., Processing Sticky Cotton: Effect of Stickiness of Yarn Quality, Submitted to the *Textile Res. J.*, **75**(5), 402–410 (2005).
8. Miller, W. B., Peralta, E., Ellis, D. R., and Perkins, Jr., H. H., Stickiness Potential of Individual Insect Honeydew Carbohydrates on Cotton Lint. *Textile Res. J.* **64**(6), 344–350 (1994).
9. Sisma, S., and Schenek, A. Bremen Honeydew Test – New Method for Testing the Stickiness Tendency of Cotton. *Milliand Textilberichte* **13**, 593–595 (1984).
10. Strolz, H. M., ITMF Cotton Contamination Survey 2001, in "Proc. 26th International Cotton Conference", International Textile Manufacturers Federation, Bremen, 13–16 March, pp. 35–39, 2002.
11. Watson, M. D. An Overview of Cotton Stickiness. In "Proc. International Cotton Conference," 1–4 March 2000, International Textile Manufacturers Federation, Faserinstitut, Bremen, Germany, 2000.