DETERMINATION OF WALL THICKNESS AND PERIMETER BY FMT AND DIODE ARRAY HVI. PART 1. DATA ANALYSIS AND RESULTS

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Abstract

Past research has indicated that a nondestructive VIS/NIR diode-array HVI measures the fundamental fiber properties of wall thickness and perimeter on blended cottons with precision equal to the primary methods used to calibrate the diode-array HVI. In this study, an improved Micromat model of the FMT is used as the reference to calibrate the diode-array HVI for use on unblended, raw cottons.

Introduction

Past research has indicated that nondestructive VIS/NIR spectroscopy using a diode-array HVI measures the fundamental fiber properties of wall thickness and perimeter on blended cottons with precision equal to the laboratory error of the FMT used to calibrate the diode-array HVI (Buco, Montalvo, Faught, Grimball, Stark, & Luchter, 1995). Recent work has been done to improve the precision of the Shireley Developments Limited Micromat Tester (FMT) to reduce short-term drift which should result in improved measurement. Part 2 of this series focuses on using the Micromat Model of the FMT to calibrate the diode-array HVI. The diode array HVI also has been improved in multiple ways. It now views a larger surface area of cotton and thus the need for sample rotation to increase the viewed area is eliminated. In addition, the measurement time for the diode-array has been reduced to 1 second which, with the associated specimen handling time, will allow up to 250 specimens to be measured per hour. The purpose of this study was to determine the precision of the diode-array HVI on unblended, raw cottons.

Materials and Methods

Cotton samples (N=80) were from the 1993 National Cotton Variety Test (NCVT). These cottons represented four varieties (Acala, DP-50, DP-90, and Paymaster) from 16 growth areas in the U.S.

Micromat Tester

All cotton samples were analyzed by the Shireley Developments Limited Micromat Tester (FMT). The FMT was calibrated with the AMS H3 standard cotton using the new headspace resistance manifold described in part 2 of this two paper series. The PL and PH air flow rates through the cotton were adjusted to give the required 122 and 80 mm water pressure drop for PL and PH, respectively. Using these same flow rates, the calibration was transferred to the headspace resistance standards manifold and checked by running the resistance standards for PL and PH as regular samples. The operational cycle was as follows. The flow rates were adjusted to get the correct pressure drop through the resistance standards for PL and PH. Four cotton specimens for each of three randomly selected NCVT samples were measured. The flow rates were readjusted, if necessary, and the cycle was repeated until all 80 cottons were analyzed four times. Then the complete process was repeated two more times to yield 12 replications per NCVT cotton. The H3 cotton and another quality control cotton were periodically run and results charted to ensure the FMT did not drift during the measurement period.

Diode-array HVI

An HVI unit was stripped down and a sample presentation system, pneumatic arm with plunger, and a VIS/NIR spectrophotometer were installed on the HVI bench. A 5” diameter cell over a quartz bottom held a cotton sample and the fiber mass was pressed against the quartz with a plunger with 40 lbs of force. The pneumatic arm with plunger consisted of a 0.25" metal rod fitted through a bushing and mounted on the horizontal, triangular plate of a pneumatic arm. A flat 4.8" diameter sample plunger was attached to the lower end of the rod. The diode-array instrument (KES Analysis, Inc., N.Y., N.Y.) was mounted under the quartz. This spectrophotometer has separate light source and detector modules. Each was placed off-center and several inches from the sample plane. The service area measured by the diode array detector was 13 square inches. The fast diode-array HVI measured 152 data points spanning the spectral region from 400 nm to 1650 nm.

Sixty g of each cotton was partitioned into two thirty g specimens. Each raw 30 g specimen was analyzed on the diode-array using simultaneously an internal and an external standard. Two 1-second spectra were taken, the cotton flipped over and another two 1-second spectra were obtained to yield four spectra per specimen or eight spectra per sample. The specimens were cleaned on a Shirley Analyzer and conditioned overnight. The cleaned specimens were reanalyzed on the diode-array to give eight 1-second spectra per sample.

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Diode-array Calibration

The twelve readings available from the Micromat on each cotton were averaged to obtain an average per cotton for PL and PH, and the derived properties of wall thickness, perimeter, and micronaire. These data were used to separate the 80 cottons into a calibration set, consisting of 60 samples, and a validation set, consisting of the remaining 20 samples. Calibration and validation cottons were selected so that the range of properties on PL and PH were approximately equivalent for both sets.

Using the VIS/NIR spectral data corrected against the diode-array internal reference from the raw and cleaned cottons, partial least squares were applied to the data with the number of retained factors determined on the basis of the F-test. Optimal models were obtained independently for wall thickness, perimeter and micronaire. The calibration models were applied to the raw, unblended cottons in the validation set to obtain a goodness of fit using the standard error of prediction, not biased corrected.

Results and Discussion

The properties as measured on the Micromat are summarized in Table 1 for the 60 calibration and the 20 validation cottons.

Buco et al. (1995) showed that use of both clean and raw samples for the calibration data set results in reduced fit to the calibration data, yet provided better results on the validation set of raw cottons. In this study, use of 60 clean samples and 60 raw samples resulted in calibrations with R^2's of 0.89, 0.84 and 0.92 for wall thickness, perimeter, and micronaire, respectively, on the combined raw plus clean samples. The associated root mean square deviations (RMSD) were 0.50 for wall thickness, 0.856 for perimeter, and 0.088 for micronaire.

Inspection of the partial least squares residuals for the three properties in the validation results indicated that one cotton was an outlier for perimeter only. Thus, only 19 samples were used in the validation set for perimeter while all 20 samples were used in the validation sets for wall thickness and micronaire. The validation results in table 2 indicate that good precision is obtainable by a high speed diode-array HVI if the FMT is improved to reduce short-term drift.

Since four cotton varieties from 16 growth regions were used in this study, an attempt was made to develop four calibration models, each model using three of the varieties to predict the remaining variety. The results yielded significant decreases in precision for the remaining variety. Thus, all four varieties were required for calibration.

The results from the high speed NIR diode-array HVI demonstrate that it can be used on raw, unblended cottons with high precision. High precision was obtained, in part, because the FMT used to calibrate the diode-array HVI was modified to reduce short-term drift. The results justify industry evaluation of the high speed diode-array HVI on a larger set of unblended, raw cottons which should include a cross section of varieties and growth areas from the US.

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Table 1. Micromat properties of calibration and validation samples

Calibration	Samples (N=60)			Validation Samples (N=20)		
Property	Minimum	Maximum	Mean	Minimum	Maximum	Mean
PL	142.3	257.2	189.6	142.3	257.2	188.9
PH	91.8	182.7	128.9	91.8	182.9	127.9
Mic	3.46	5.26	4.34	3.46	5.26	4.38
Perimeter	42.6	50.5	47.3	42.7	50.4	47.0
Wall Thick	2.35	3.19	2.76	2.35	3.19	2.79

Table 2. Diode-Array HVI validation on raw cottons

Property	R^2	RMSD	%CV	Bias
Wall Thickness	0.962	0.052	1.94	0.025
Perimeter	0.901	0.986	1.92	-0.613
Micronaire	0.982	0.069	1.60	0.022