ANALYSIS OF COTTON MATURITY AND FINENESS BY MULTIPLE NIR HVIS PART I: DATA ANALYSIS

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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 two diode-array HVIs for use on unblended, raw cottons and blended 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 Shirley Developments Limited Micromat Tester (FMT) to reduce short-term drift. This will provide more precise measurements with which to calibrate high speed diode-array HVI instruments. Part II of this series focuses on the improvements to the Micromat Model of the FMT. The diode array HVI also has been improved in multiple ways. It allows for the rotation of cotton samples to increase the measured surface area of the cottons and to provide varying orientations of the cotton fibers. In addition, the measurement by the diode-array has been delayed until ½ second after the plunger pressing on the sample comes to a rest to allow the cotton to settle. The purpose of this study was to determine the precision of the diode-array HVI on unblended raw cottons and clean cottons in both rotating and stationary modes.

Materials and Methods

The Agricultural Marketing Service (AMS) in Memphis provided cotton samples from 639 bales within the 1996 crop year. Of these, 200 cottons had sample weights of at least 80 g. The 200 cottons were divided into four groups of cottons by selecting every fourth cotton for a group. The first two groups (100 cottons) were analyzed for this study. Eighteen of these cottons were Pima cottons. Each of the 100 cottons was divided into two 40 g samples which were treated and measured independently throughout the study. All 200 raw, unblended samples were measured in a rotating sample mode by a NIRSystems Model 6500 spectrophotometer mounted in an HVI configuration, and two KES Diode-Array spectrophotometer instruments mounted in HVI configurations. Cottons were measured on the KES instruments in both a rotating and a stationary mode. The cottons were cleaned and blended in a Shirley Analyzer and then re-measured on the same three instruments with a rotating sample presentation only. Finally, the cottons were measured on an improved version of a Micromat Tester. The results of the measurements on the NIRSystems Model 6500 are not presented in this paper.

Diode-array HVI

An HVI unit was stripped down and a sample presentation system, a 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 a 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 HVI could be operated in a rotation mode, with the sample on the quartz rotated at 60 rpm, or in a stationary mode. The surface area measured by the diode array detector was 18 square inches. The fast diode-array HVI measured 152 data points spanning the spectral region from 400 nm to 1700 nm. Two detectors each measured half of the 152 data points. The "VIS" detector measures from 400 nm to 950 nm and the "NIR" detector measures the 76 points from 950 nm to 1700 nm. Due to "noisy" data, only data points 21 through 76 were used from the VIS detector while 76 points were used from the NIR detector.

Each raw 40 g specimen was analyzed on the diode-array using simultaneously an internal and an external standard. Six 1-second spectra were taken, with data acquisition on the rotating sample beginning 500 ms after the plunger was lowered onto the sample. The cotton sample was flipped over and another six 1-second spectra were obtained on the rotating sample to yield 12 spectra per sample. The samples were moved to the second diode-array and the process repeated to yield 12 additional spectra. Then spectra on the samples were measured again with the samples in a stationary mode. The specimens were cleaned on a Shirley Analyzer. The cleaned specimens were reanalyzed on the NIRSystems spectrophotometer and both diode-array spectrophotometers in a rotating sample mode only. The samples were then analyzed on the Micromat Tester

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following the procedure provided in Part II of this two paper series to obtain eight replicate measurements per sample.

Diode-array Calibration

The eight readings available from the Micromat on each sample were averaged to obtain an average per sample for PL and PH, and the derived properties of maturity ratio, fineness, micronaire, percent maturity, perimeter, wall thickness and percent thickness. Using the VIS/NIR spectral data of a diode-array instrument, corrected against its internal reference, principle components analysis was independently applied to the 76 data points from the "VIS" detector and to the 76 points from the "NIR" detector to obtain 10 principal components for each set of points. Visual inspection of plots of predicted Micromat values against actual values indicated that a curvilinear model would be appropriate. Therefore, the 20 principal components (10 VIS components and 10 NIR components) were entered as linear components into a forward stepwise regression along with the squares of the first five principal components from each of the VIS and the NIR regions. A probability of .15 was used for inclusion and exclusion in the model to select predictive components on the basis of the stepwise F-test. Independent models were obtained for PL, PH, maturity ratio, fineness, micronaire, percent maturity, perimeter, wall thickness and percent thickness for each diode array for raw rotating cottons, raw stationary cottons, and clean rotating cottons.

Results and Discussion

The cotton properties as measured on the Micromat are summarized in Table 1 for the 200 samples.

An analysis of the pooled standard deviation of the diode-array spectra was performed to determine if all six spectra per cotton should be used for analysis. Figures 1 and 2 indicate that the variation among the last three spectra (averaged over sides of a sample) is significantly less than the variation among all six spectra and less than the variation among the first three spectra on diode-array HVI #1. The variation of spectra of clean cottons is less than that of raw cottons. Similar patterns are observed for diode-array HVI #2. Consequently, only the last three spectra from each cotton were used from the diode-array HVIs to measure cotton maturity and fineness.

The results of the stepwise regression models for diode-array HVI #1 using the last three spectra are presented in Tables 2 and 3. To obtain unbiased goodness of fit measures, the root mean squared deviations (RMSD) and R^2 results are obtained from a hold-one-sample-out cross-validation procedure. The cross-validation results indicate that rotating the raw cotton samples on the diode-array HVI vastly improves the precision of fiber property measurements as compared to measuring the same samples in a stationary mode. Cleaning the cottons prior to measuring them in a rotation mode enhances the precision further. A similar pattern of results is found for diode-array HVI #2 in Tables 4 and 5, although this instrument was not as precise as the first diode-array HVI.

To obtain the precision of the fiber properties as measured by the Micromat, a variance component model was fit to determine the variance due to cottons, samples within cottons, days within samples, and replicates within days. The last two variance components were added together to find the variance of a single Micromat measurement due to replication and day variability. The square roots of these results (standard deviations) are shown in Table 6. To obtain the precision of fiber properties as measured by the diode-array HVI, the pooled standard deviation between sides of the samples measured on the HVI were calculated and appear in Table 6. Comparison of these results indicates that the diode-array HVI measures maturity ratio, fineness, percent maturity, perimeter, and percent thickness on raw cottons better than the Micromat does on clean cottons. In addition, the diode-array HVI more precisely mreasures all of the above properties plus wall thickness on clean samples than does the Micromat.

The results from the high speed NIR diode-array HVI demonstrate that it can be used on raw, unblended cottons with good precision. This precision was obtained, in part, because the Micromat 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.

Acknowledgments

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References

Buco, S. M., J.G. Montalvo, Jr., S. E. Faught, R. Grimball, E. Stark, and K. Luchter. 1995. Determination of maturity/fineness by FMT and diode-array HVI. Part 2: Data Analysis and results, p. 1279. Proceedings of the Beltwide Cotton Conference. 1279-1281.

Table 1. Micromat properties of cotton samples.

Property	Mean	Minimum	Maximum
PL	192.9	140.1	384.98
PH	136.8	89.9	343.63
Maturity ratio	0.972	0.635	1.179
Fineness	170.9	124.8	230.1
Micronaire	4.334	2.60	5.32
% Maturity	85.1	55.06	98.8
Perimeter	50.22	42.4	61.8
Wall thickness	2.70	1.77	3.23
% Thickness	33.8	20.4	43.4

Table 2. Measurement of raw cotton maturity and fineness by diode-array HVI #1.

	Stationary Sample		Rotating Sample	
Property	RMSD (cross-valid.)	R^2	RMSD (cross-valid.)	R^2
PL	10.04	0.941	6.98	0.971
PH	10.23	0.934	7.13	0.970
Maturity ratio	0.026	0.912	0.019	0.961
Fineness	4.736	0.940	3.945	0.957
Micronaire	0.124	0.953	0.077	0.981
% Maturity	2.033	0.926	1.447	0.963
Perimeter	0.975	0.887	0.883	0.911
Wall thickness	0.066	0.949	0.040	0.980
% Thickness	1.169	0.912	0.826	0.958

Table3. Measurement of clean cotton maturity and fineness by diode-array HVI #1.

	Rotating Sample		
Property	RMSD (Cross-valid.)	R^2	Coeff. Var. (%)
PL	5.88	0.981	3.05
PH	6.13	0.978	4.48
Maturity ratio	0.017	0.970	1.72
Fineness	3.724	0.963	2.18
Micronaire	0.061	0.988	1.41
% Maturity	1.269	0.971	1.49
Perimeter	0.878	0.910	1.75
Wall thickness	0.030	0.989	1.17
% Thickness	0.732	0.967	2.16

Table 4. Measurement of raw cotton maturity and fineness by diode-array HVI #2.

	Stationary Sample		Rotating Sample	
Property	RMSD (cross-valid.)	R^2	RMSD (cross-valid.)	R^2
PL	10.23	0.941	6.66	0.975
PH	10.14	0.937	7.07	0.970
Maturity ratio	0.024	0.912	0.017	0.969
Fineness	4.658	0.939	3.437	0.971
Micronaire	0.118	0.956	0.069	0.986
% Maturity	1.828	0.942	1.271	0.972
Perimeter	1.020	0.867	0.735	0.939
Wall thickness	0.061	0.957	0.036	0.984
% Thickness	0.061	0.932	0.726	0.969

Table 5. Measurement of clean cotton maturity and fineness by diode-array HVI #2.

	Rotating Sample		
Property	RMSD (cross-valid.)	R^2	Coeff. Var. (%)
PL	6.05	0.979	3.13
PH	6.13	0.975	4.48
Maturity ratio	0.018	0.962	1.87
Fineness	4.024	0.957	2.36
Micronaire	0.061	0.989	1.42
% Maturity	1.371	0.967	1.61
Perimeter	0.905	0.902	1.80
Wall thickness	0.030	0.989	1.14
% Thickness	0.800	0.960	2.36

Table 6. Pooled within cotton standard deviation of Micromat and Diode-Array HVI #1.

	Pooled Standard Deviation of Micromat	Pooled Standard Deviation of Diode-Array	
Property	Clean Cotton	Raw Cotton, Rotating Sample	Clean Cotton, Rotating Sample
PL	3.83	otto6	4.08
PH	3.65	5.09	3.63
Maturity ratio	0.019	0.012	0.010
Fineness	3.47	2.81	2.58
Micronaire	0.054	0.077	0.058
% Maturity	1.43	0.83	0.79
Perimeter	0.922	0.436	0.504
Wall thickness	0.031	0.037	0.027
% Thickness	0.851	0.496	0.430

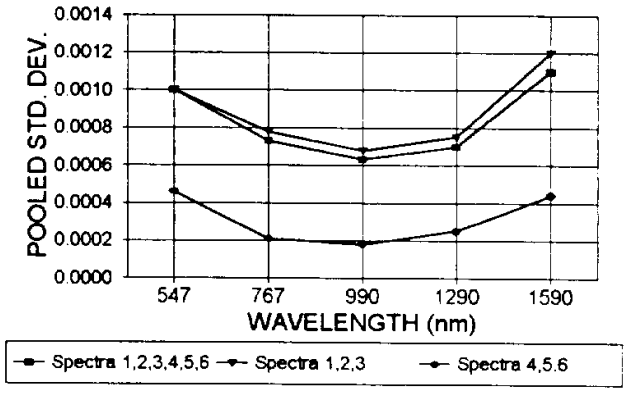

Figure 1. Variation among spectra within samplesw of raw, rotating cottons.

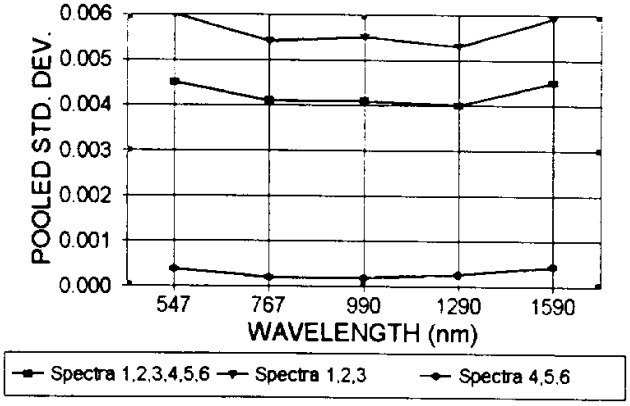

Figure 2. Variation among spectra within samples of clean, rotating cottons.