FAST DETERMINATION OF MATURITY AND FINENESS BY NIR WITH A DIODE-ARRAY HVI. PART 1. DATA ANALYSIS

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Abstract

Past research has indicated that a nondestructive VIS/NIR high speed 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 and carded cottons. Matched samples are used to improve the calibration of the diode-array HVI.

Introduction

Past research has indicated that nondestructive VIS/NIR spectroscopy using a fast 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 (micromat model) 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) using headspace resistance standards to reduce short-term drift which should result in improved measurement. Part two of this series focuses on using the Micromat Model of the FMT as a reference method 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 potentially eliminated. In addition, the measurement time for the diode-array has been reduced to 1 second. (A discussion of the sample handling speed of the diode-array HVI is discussed in the third paper of this three part series.) The purpose of this study was to determine the precision of the diode-array HVI using a nonrotating specimen holder for raw cottons, carded cottons, and cottons processed by the Shirley Analyzer.

Reprinted from the *Proceedings of the Beltwide Cotton Conference*
Volume 1:552-554 (1997)
National Cotton Council, Memphis TN

Materials and Methods

Cotton samples (N=36) were grown by William Meredith of the Agricultural Research Service at Stoneville, MS. Each cotton represented a different variety bred in the US cotton belt. For each cotton, two 30 g specimens of the raw cotton were weighed and placed in separate bags. Two bags were analyzed by the diode-array HVI and then an FMT. In addition, a sample was drawn for measurement by the AFIS F&M module. Sufficient quantity of sample was drawn to allow 5 replications of 5000 fibers each.

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. 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 surface area measured by the diode-array detector was 19 square inches. The samples were not rotated during collection of the spectra. The fast diode-array HVI measured 152 data points spanning the spectral region from 400 nm to 1700 nm. Two diode-array HVIs were use for analysis of the cottons.

NIR Systems Spectrophotometer. Using the same sample HVI presentation system and pneumatic arm with a plunger, an NIR Systems 6500 scanning spectrophotometer was installed on the HVI bench. The sample cup was rotated while spectra were obtained to maximize the surface area scanned. The spectrophotometer measured the spectral region from 400 to 2498 nm in 2 nm increments.

Analysis. The two 30 g specimen bags of each raw cotton were analyzed by both diode-arrays using the following process: the specimen was removed from the bag and placed in a sample holder. The sample holder was placed on the first HVI and two 1-second spectra were taken of one side of the specimen. The sample holder was moved to the second HVI and two 1-second spectra were taken of the same side of the specimen. The sample was flipped over and returned to the first HVI and two 1-second spectra were obtained of the second side. The holder was moved to the second HVI and two 1-second spectra were taken of the second side of the specimen. The specimen was re-bagged and the second specimen bag opened and analyzed on both HVIs following the same process. Thus, eight spectra per specimen bag were available for a total of 16 spectra for each of the 36 cottons.

The contents of each specimen bag were opened in an opener-blender and cleaned in a minicard and conditioned overnight. The carded specimens were reanalyzed on each diode-array following the same procedure as the raw cottons for a total of 16 spectra for each of the 36 carded cottons. At a separate time, the contents of each carded specimen bag were repacked into the holder and measured by the NIR Systems 6500 with one spectra per side of the specimen.

The contents of each specimen bag were processed now by a Shirley Analyzer and conditioned overnight. The specimens were analyzed a third time on the diode-array HVI to yield a total of 16 more spectra per cotton. Finally, the contents of each specimen bag were analyzed by the Shirley Developments Limited 089 Micromat Tester following the method outlined in the second paper of this series.

Diode-array Calibration. The readings available from the Micromat on each cotton were averaged to obtain an average per cotton specimen (bag) for PL and PH and the derived properties of wall thickness, perimeter, fineness, and micronaire. The readings available from the AFIS F&M module were averaged to obtain an average per cotton for perimeter and wall thickness.

Using the VIS and NIR spectral data separately from the first diode-array HVI, principal components from the raw cotton specimens (bags) were calculated for each spectral region and combined into one principal components model with the number of retained factors determined on the basis of the F-test. Cross-validation results were obtained by sequentially holding out one of the 72 specimens and predicting it from the remaining samples until all 72 specimens had been predicted in this fashion. The root mean squared deviation, coefficient of variation, and the R^2 between the observed and predicted values were obtained. The analysis process was repeated for the spectra of the carded samples, and then for the spectra of the samples processed by the Shirley Analyzer. A total of three cross-validations were performed on the first diode-array HVI: a raw cotton cross-validation, a carded cotton cross-validation, and a cross-validation on cottons processed by the Shirley Analyzer. The procedure was repeated for the spectra obtained on the second diode-array HVI to obtain three cross-validations on that instrument.

NIR Systems 6500 Calibration. Using the VIS and NIR spectral data separately, principal components from the carded cottons specimens were calculated for each spectral region and combined into one principal components model with the number of retained factors determined on the basis of the F-test. Cross-validation results were obtained by the hold-one-sample-out method. Although the NIR Systems 6500 covers a larger region of the NIR spectra, only the portion of the spectra below 1700 nm was used in order to match the spectral range of the diode-array HVI.

Results and Discussion

The properties as measured on the Micromat and AFIS F&M Module are summarized in Table 1 and Table 2 for the 36 cottons. Of note is the fact that FMT perimeter and AFIS perimeter correlate 0.95 while FMT wall thickness and AFIS wall thickness correlate 0.93.

The cross-validation results on the diode-array HVI #1 in Table 3 indicate that AFIS perimeter is measured better than FMT perimeter while the opposite is true for wall thickness; FMT wall thickness is measured better than AFIS wall thickness. The same relationships hold for the second diode-array HVI in Table 4. In general, diode-array HVI #2 performed better than diode-array HVI #1. The reasons for the performance difference remain to be investigated.

Comparison of the results from the diode-array HVI with the results in Table 5 for the NIR Systems 6500 spectrophotometer indicates that the slower laboratory grade instrument performed better than the diode-arrays. The improved performance of this instrument may be due in part to the fact that the sample holder was rotated while obtaining the spectra on the NIR Systems 6500 and the sample holder was not rotated for the diode-array HVI spectra. Past work with the NIR Systems instrument indicated improved results with a rotated sample cup.

The results from the high speed NIR diode-array HVI demonstrate that it can be used on raw, unblended cottons. The results justify continued evaluation of the high speed diode-array HVI on a larger set of cottons with a rotating sample cup. In addition, expansion of the use of the diode-array HVI to other properties may be warranted.

Acknowledgments

The authors wish to express their appreciation to Dr. William Meredith, USDA, ARS, Stoneville, MS for providing the cotton samples.

References

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Table 1. Fiber properties of 36 Stoneville cottons.

Property	Mean	Minimum	Maximum
FMT			
PL	193.1	144.9	233.8
PH	136.9	97.5	172.8
Micronaire	4.28	3.70	5.19
Fineness	171.1	141.4	208.3
Perimeter (P)	50.7	45.7	55.7
Wall Thickness	2.66	2.37	3.07
AFIS			
Perimeter (P)	52.0	48.1	55.4
Wall Thickness	2.48	2.25	2.84

Table 2. Correlations among fiber properties of 36 Stoneville cottons.

Property	FMT PH	FMT Mic.	FMT Fine.	FMT P	FMT t	AFIS P	AFIS t
FMT							
PL	.98	-.99	-.92	-.55	-.97	-.58	-.88
PH		-.99	-.85	-.43	-.99	-.47	-.91
Micronaire			.92	.54	.98	.57	.90
Fineness				.83	.83	.82	.73
Perimeter (P)					.38	.95	.29
Wall Thick. (t)						.42	.93
AFIS							
Perimeter (P)							.32

Table 3. Cross-validation Statistics for Diode-Array HVI #1 for raw cottons, carded cottons, and cottons processed by the Shirley Analyzer.

	Raw			Carded			Shirley		
Property	RMSD	%CV	R^2	RMSD	%CV	R^2	RMSD	%CV	R^2
FMT									
PL	5.00	2.58	.947	4.63	2.39	.954	5.32	2.76	.943
PH	5.58	4.08	.913	5.93	4.31	.896	4.49	3.26	.940
Micronaire	.074	1.75	.957	.067	1.57	.962	.076	1.78	.955
Fineness	5.04	2.95	.882	3.92	2.29	.928	4.99	2.91	.883
Perimeter	1.14	2.27	.709	1.44	2.84	.565	1.35	2.66	.610
Wall Thick.	.048	1.80	.918	.055	2.06	.885	.039	1.48	.941
AFIS									
Perimeter	.815	1.57	.805	1.03	1.98	.699	1.17	2.24	.626
Wall Thick.	.063	2.55	.830	.073	2.97	.766	.061	2.47	.839

Table 4. Cross-validation Statistics for Diode-Array HVI #2 for raw cottons, carded cottons, and cottons processed by the Shirley Analyzer.

	Raw			Card			Shirley		
Property	RMSD	%CV	R^2	RMSD	%CV	R^2	RMSD	%CV	R^2
FMT									
PL	4.41	2.28	.959	5.16	2.68	.946	3.81	1.97	.969
PH	4.67	3.41	.939	5.24	3.81	.919	3.54	2.58	.962
Micronaire	.063	1.48	.969	.069	1.63	.959	.055	1.31	.973
Fineness	4.26	2.50	.913	4.25	2.49	.912	4.85	2.84	.881
Perimeter	1.13	2.24	.718	1.37	2.69	.629	1.35	2.67	.629
Wall Thick.	.040	1.52	.943	.047	1.78	.914	.044	1.66	.930
AFIS									
Perimeter	.797	1.53	.814	1.10	2.11	.668	1.13	2.17	.647
Wall Thick.	.063	2.54	.830	.072	2.91	.775	.058	2.33	.857

Table 5. Cross-validation Statistics for NIR Systems 6500 for carded cottons.

Property	RMSD	%CV	R^2
FMT			
PL	2.77	1.42	.983
PH	3.00	2.17	.972
Micronaire	.039	0.92	.987
Fineness	3.06	1.79	.952
Perimeter	0.84	1.65	.857
Wall Thickness	.022	0.82	.982
AFIS			
Perimeter	0.49	0.95	.931
Wall Thickness	.042	1.68	.923