COTTON MICRONAIRE MEASUREMENTS BY A SMALL PORTABLE NEAR INFRARED (NIR) ANALYZER J. E. Rodgers SRRC-ARS-USDA New Orleans, LA

<u>Abstract</u>

Micronaire is a key quality and processing parameter for cotton fiber. Much interest has been shown in small, portable instruments (e.g., Near Infrared or NIR) that have the potential to monitor cotton fiber micronaire both in the laboratory and in or near the field. A new, small (fits in your hand) NIR spectrometer—the JDS Uniphase MicroNIR—was evaluated for micronaire measurements in the laboratory (pre-field analyses). The NIR measurement was fast (< 2 minute per sample) and easy to perform. Distinct spectral differences with increasing micronaire were observed. The initial micronaire results for the laboratory trials were very encouraging, with few outliers and low residuals.

Introduction

In the U.S., cotton classification is performed by the USDA's Agricultural Marketing Service (AMS), using the Uster[®] HVITM high volume instrument. Micronaire is a key quality and processing parameter for cotton fiber, and it is a key parameter used to class cotton fiber in the U.S. (USDA, 2005). Micronaire is a function of two fiber parameters—maturity (degree of development of the fiber's secondary wall) and fineness (often expressed as the fiber's linear density) (Wakelyn et. al., 2007). The HVITM has been in use for several years, and it is the global "workhorse" for the classification/quality assessment of cotton fiber. For proper classification and quality assessment results, the HVITM measurements must be performed in the laboratory under stringent environmental conditions. In the global marketplace, one key area of quality assessment emphasis is the development of new quality tools for laboratory, field, and at-line measurements so as to integrate cotton quality measurements from the cotton field to the final fabric.

Much interest has been shown in small, portable instruments (e.g., Near Infrared or NIR) that have the potential to rapidly and accurately monitor cotton fiber micronaire both in the laboratory and in or near the cotton field. The capabilities and universal nature of NIR techniques to rapidly and accurately measure fiber micronaire in the laboratory has been demonstrated previously. (Montalvo and von Hoven, 2004; Rodgers et. al., 2010a; Rodgers et. al., 2010b) The potential and capabilities of using portable NIR analyzers to measure fiber micronaire in and near the cotton field was established in a series of field trials. (Rodgers et. al., 2010c) The portable NIR analyzer used in the previous laboratory and field evaluations concentrated on the Brimrose 5030 unit. (Figure 1) Although the Brimrose unit rapidly and accurately measured fiber micronaire (both on ginned lint and cotton bolls), concerns were expressed about the cost and size of the unit. In addition, the technology has been in use for several years, and many improvements and innovations have been observed in the area of portable instrumentation and optics.



Figure 1. Brimrose 5030 portable NIR analyzer.

A new micro-size NIR unit has been introduced by JDS Uniphase (JDSU). A program was implemented to determine the capabilities of the JDSU MicroNIR portable NIR instrument for monitoring cotton fiber micronaire both in the laboratory and outside the laboratory (e.g., in or near the cotton field). The research presented in this paper focuses on method feasibility evaluations in the laboratory.

Material and Methods

The samples used in this evaluation were a set of 102 well-defined, wide-range, diverse fiber samples. (Hequet et. al., 2006) The micronaire values for all samples were obtained from the HVITM-1000 at the Southern Regional Research Center of the Agricultural Research Service (USDA-ARS-SRRC). All HVITM and JDSU microNIR measurements were performed in a conditioned laboratory at standard conditions $(70\pm2^{\circ}F, 65\pm2\%$ relative humidity or RH). The samples were split into two samples sets—a set used to calibrate the NIR instrument (Calibration Set, n=69) and a set used to validate/verify the calibration for the NIR instrument (Prediction Set, n=33).

The JDSU MicroNIR portable NIR instrument is very small, lightweight, and low cost (< \$10K). (Figure 2) The instrument uses a Linear Variable Filter (LVF) technology, which makes it rugged and very fast (< 15 seconds per spectra/measurement). The glass face of the unit is placed against the surface of the cotton lint sample, and the measurement taken. Five measurements were made per sample on the JDSU unit, with the unit sampling a different portion of the sample with each measurement. The five spectra/measurements were averaged for chemometirc analyses. Vendor recommended operational procedures and protocols were used.



Figure 2. JDSU MicroNIR portable NIR instrument.

The JDSU MicroNIR laboratory results were compared to those previously obtained by the Brimrose 5030 NIR unit. (Rodgers et. al., 2010b) The comparative parameters for this evaluation included R^2 , Standard Error of Calibration/Standard Deviation of Differences residual analysis [SEC/SDD; the standard deviation of the differences between the reference and measured micronaire value for each sample in the calibration (SEC) and prediction (SDD) sets], and the number of "outliers." An outlier was any sample in which the difference between the HVITM micronaire and NIR-determined micronaire was > ±0.3 micronaire units. The higher R^2 , the lower SEC/SDD, and the fewer the number of outliers, the better the NIR instrument performed. In discussions with key industry stakeholders, the end-state criterion for outliers was established at a maximum of 30% outliers.

Results and Discussion

A program was implemented to determine the capabilities of the JDSU MicroNIR portable NIR instrument for monitoring cotton fiber micronaire both in the laboratory and outside the laboratory (e.g., in or near the cotton field), with initial emphasis on laboratory measurements. Distinct NIR first derivative spectral differences were observed with the JDSU MicroNIR for samples with distinct micronaire differences (Figure 3). The JDSU MicroNIR measurement of micronaire was relatively fast (< 2 minute per sample, 5 measurements per sample). The measurement was easy to perform, and no sample preparation was required.



Figure 3. NIR first derivative spectral intensities differences, JDSU MicroNIR (micronaire = 2.70 for TT3089, 4.31 for TT2888, and 5.65 for TT3074).

Compared to the reference HVITM micronaire results, both the portable Brimrose 5030 and the portable JDSU MicroNIR NIR measurements for micronaire yielded overall very acceptable results for both the calibration and prediction sample sets, with high R^2s (≥ 0.84), low SDDs (≤ 0.25), and a low number of outliers ($\leq 15\%$ outliers), as shown in Table I (Calibration Set) and Table II (Prediction Set). The higher SDD for the JDSU MicroNIR Prediction Set results was due primarily to one major outlier at ~2.5 micronaire. All end-state criteria were achieved. The method agreement between the Brimrose 5030 and JDSU MicroNIR calibration and prediction results were excellent. Since the field/near field measurement potential and capabilities of the Brimrose 5030 unit has previously been established, these comparative results for the portable JDSU system indicate a very promising potential for at-line/near cotton field measurements of fiber micronaire to complement the current Brimrose 5030 measurements of fiber micronaire in the cotton field.

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ITEM	CALIBRATION, MICRONAIRE (n = 69)			
	HVITM	PORTABLE NIR		
		BRIMROSE 5030	JDSU MicroNIR	
AVG	4.33	4.33	4.34	
SD	0.48	0.46	0.39	
\mathbf{R}^2	NA	0.90	0.84	
SEC	NA	0.15	0.17	
$\% > \pm 0.3$ mic	NA	6	5	

ITEM	PREDICTION, MICRONAIRE (n = 33)				
	HVI TM	PORTABLE NIR			
		BRIMROSE 5030	JDSU MicroNIR		
AVG	4.01	4.06	4.05		
SD	0.64	0.61	0.56		
\mathbf{R}^2	NA	0.91	0.85		
SDD	NA	0.19	0.25		
% > ± 0.3 mic	NA	12	15		

Table II. Micronaire comparison results, Prediction Set

Summary

A new micro-size, lightweight, and cost-effective portable NIR instrument has been introduced by JDS Uniphase (JDSU)—the MicroNIR. A program was implemented to determine the capabilities of the JDSU MicroNIR portable NIR instrument for monitoring cotton fiber micronaire both in the laboratory and outside the laboratory (e.g., in or near the cotton field), with initial emphasis on laboratory measurements. The JDSU MicroNIR instrument measurement of fiber micronaire was easy to perform, rapid (analysis time of less than 2 minutes per sample, 5 measurements per sample), and no sample preparation was required. Distinct NIR spectral differences were observed between samples with distinct micronaire differences. An initial NIR method for the laboratory measurement of cotton fiber micronaire with the MicroNIR was developed, with the end-state criterion for outliers exceeded. The outlier/statistical results were in excellent agreement with those previously obtained with the portable Brimrose 5030 NIR analyzer. The results demonstrated the very promising potential for at-line/near cotton field analyses of micronaire with the JDSU unit.

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Disclaimer

The use of a company or product name is solely for the purpose of providing specific information and does not imply approval or recommendation by the United States Department of Agriculture to the exclusion of others.

References

Hequet, E., B. Wyatt, N. Abidi, D.P. Thibodeaux. 2006. Creation of a Set of Reference Material for Cotton Fiber Maturity Measurements. Textile Research Journal, 76 (7), 576-586.

Montalvo, J.G., and T. Von Hoven. 2004. Analysis of cotton. pp. 671-728. *In* C. Roberts, J. Workman, and J. Reeves (eds.) Near-Infrared Spectroscopy in Agriculture. American Society of Agronomy, Agronomy Monograph No. 44, Madison, WI.

Rodgers, J.E., J.G. Montalvo, G. Davidonis, T. Von Hoven. 2010a. Near Infrared measurement of cotton fiber micronaire, maturity, and fineness—A comparative investigation. Textile Research Journal, 80(9): 780-793.

Rodgers, J.E., S.Y. Kang, C.A. Fortier, J.G. Montalvo, X. Cui, V.B. Martin. 2010b. Near Infrared measurement of cotton fiber micronaire by portable Near Infrared instrumentation. Textile Research Journal, 80(15): 1503-1515.

Rodgers, J.E., S.Y. Kang, C.A. Fortier, X. Cui, G. Davidonis, E. Clawson, D. Boquet, W. Pettigrew. 2010c. Preliminary field measurement of cotton fiber micronaire by portable NIR. Spectroscopy, 25(9): 38-44.

USDA AMS Cotton Program Brochure. April 2005. Cotton classification, understanding the data.

Wakelyn, P.J., N.R. Bertoniere, A.D. French, D.P. Thibodeaux, B.A. Triplett, M.A. Rousselle, W.G. Goynes, J.V. Edwards, L. Hunter, D.D. McAlister, G.R. Gamble. 2007. Physical properties of cotton, Chapter 7. pp. 107-114. *In* M. Lewin (ed.) Cotton Fiber Chemistry and Technology. CRC Press, Boca Raton, FL.