MICRONAIRE MEASUREMENTS ON SEED COTTON AND COTTON FIBER, IN AND OUTSIDE OF THE LABORATORY USING MICRO NIR-INFRARED INSTRUMENTS Jimmy Zumba James Rodgers Cotton Structure & Quality Research Unit (CSQ), Southern Regional Research Center, USDA

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<u>Abstract</u>

Micronaire is a key quality parameter in cotton fiber. NIR-spectroscopy has the ability to measure micronaire in and out of the laboratory. New very small micronaire instruments have recently been introduced. A program was established to measure micronaire in and outside the laboratory on seed cotton fiber and cotton lint using micro portable handheld NIR-Infrared instruments. Adding new data to the original 189 lint samples, including data from different environments (laboratory and greenhouse) and fiber type conditions (lint and seed cotton) made the calibration more robust, increasing the accuracy of the prediction and reducing the variability of the prediction. The accuracy of the prediction was improved with both instrument, but when compared each instrument they differed on the predictability and variability, and it may be due to the precision of each instrument. It is advisable to use the instrument that fits the best the laboratory research objectives, considering its weight or size.

Introduction

Micronaire is an important fiber quality parameter in the cotton industry (Cotton Incorporated, 2013; USDA-AMS, 2005), and quality and processing impacts can occur if the micronaire for the harvested cotton is too high or too low. Acquiring the fiber quality parameters is a lengthy process, and it could take several days before knowing the final fiber quality of the cotton grown in the fields. First, the cotton had to be harvested in the field and samples collected. Next, the seed cotton samples are sent to be ginned and fiber samples labelled correctly; once labelled the fiber samples have to be sent to the fiber lab for quality analysis (USDA-AMS, 2005). In the fiber lab, the fiber has to be conditioned for at least 24 hours $(21\pm1^{\circ}C \text{ and } 65\pm2\%)$ (ASTM, 2015). Once the fiber is analyzed, data can be downloaded from the HVI, then after several days the fiber quality parameters are known. Thus, the need exists for new complementary micronaire fiber measurements that can be performed both in and outside the laboratory, and can provide quick results. One potential technology that addresses these needs is Near Infrared (NIR) spectroscopy analyses (Rodgers et al, 2010a, 2010b and 2010c).

The Near-Infrared (NIR) spectral region is located between the visible and infrared spectral region. It is referred to as the region from 800 - 2500 nanometers (nm), although the primary NIR spectra region is normally from 1100-2500 nm (Burns, 1985). NIR absorbance is due to the light interaction with the sample and the intensity of light received by the detector. The amount of light reflected or transmitted is dependent on both chemical (molecular absorbance) and physical (scattering/reflective) properties of the sample, and they arise from the overtones and valence vibrations of XH_n, groups or combination of the valence of such groups within the sample (CH_n, NH_n, and OH) (Perkampus, 1995; Workman, 2001). There are many advantages of using small NIR spectroscopy analyzers for micronaire such as it allows for quick, accurate and precise measurements; its instrument size; it is a non-destructive method; it requires no sample preparation; and it is easy to maintain and operate. The main disadvantages of NIR are that it relies on a large number of samples for the calibration and the instrument calibration process.

A program was implemented to determinate the ability of new portable NIR instruments to accurately measure micronaire in cotton lint and seed cotton to develop protocols to increase the robustness of the calibration data set, to minimize potential impacts, and to reduce micronaire variability and increase predictability. The Viavi MicroNIR 2200 and Brimrose Luminar 5030 instruments were used in this experiment.

Materials and Methods

Cotton Samples and NIR Measurements

Seed cotton samples of three conventional commercial varieties DP393 (n=75), FM958 (n=74) and SG105 (n=73) were obtained from the 2010 crop grown in Alexandria, LA. NIR measurements were made with both the Viavi MicroNIR 2200 and the Brimrose Luminar 5030 portable units, on the seed cotton in the greenhouse (SCGH) and on

seed cotton in the laboratory (SCL); in addition, NIR measurements were made on the lint from the saw-ginned seed cotton in the laboratory (LL). Prior to NIR measurements in the laboratory, the seed cotton and the saw-ginned seed cotton's fiber were conditioned for at least 24 hours ($21\pm1^{\circ}$ C and $65\pm2\%$) (ASTM, 2015). This study created a subset of spectral data (n= 666) as follow: seed cotton greenhouse (n= 222), seed cotton laboratory (n= 222), and saw-ginned lint laboratory (n= 222). Micronaire lint data of these three varieties was obtained using the Cottonscope.

<u>Data Analysis</u>

The selected evaluation targets were: 1) reduced prediction delta (Δ), 2) reduced standard deviation, 3) less than a total 5 minute analysis time per sample, and 4) minimal sample preparation and user-friendly operation. The primary comparison statistics for the portable NIR units were: Deviation of predicted micronaire with the original HVI micronaire (Δ), and Standard Deviation (SD). For this evaluation, NIR instrument performance was considered to be better with a lower prediction delta (Δ) and lower SD. The spectral data from the Viavi and Brimrose units was transferred to the Camo[®] Unscrambler software package (Version 9.8, Camo Software AS, Woodbridge, NJ). NIR calibrations and predictions were performed with Unscrambler, using derivative mathematics and PLS calibrations.

Results and Discussion

Original Lint-Only Calibration

In a prior study, a well-defined set of 189 samples lint was used (Hequet, E. 2006; Rodgers, J. 2010b) to develop a successful calibration to predict fiber lint micronaire in the laboratory for the Brimrose Luminar 5030. A similar calibration was developed for the Viavi MicroNIR 2200 instrument. Individual variety predictions on the seed cotton lab measurements were made utilizing the original 189 sample lint-only calibration data sets (Table 1). The predictions using both NIR instruments (Viavi and Brimrose) were different, and each showed large micronaire discrepancies (Deltas= Δ) and variability (SD) when compared with the calibration micronaire values obtained from HVI analysis. As shown in Table I, distinct micronaire differences for each variety were obtained for each instrument. Similar results were observed for lint versus seed cotton samples.

Variety	MIC	NIR (1100-2300 nm)	
		Viavi 2200 MicroNIR	Brimrose Luminar 5030
DP393	5.33	4.41 (Δ= -0.92)	5.39 (Δ= +0.06)
SD	0.36	0.34	0.42
FM958	4.64	4.50 (Δ= -0.14)	6.21 (Δ= +1.57)
SD	0.28	0.32	0.63
SG105	5.09	4.58 (Δ= -0.51)	5.92 (Δ= +0.83)
SD	0.47	0.47	0.68

Table 1. Predictions on Seed Cotton Lab using original lint-only calibration

Combined Calibration

The results above indicated that improvements would be required for the NIR micronaire calibrations if one wished to minimize variety and seed cotton impacts on the NIR micronaire results. In addition to the original 189 samples lint, a new subset of spectral data (n=666) from the three commercial varieties was added in order to create an optimized combined micronaire data set (n=855) which included two different environments (laboratory and greenhouse), and two type of fiber conditions (lint and seed cotton). The total optimized data set was split into two samples sets –a set used to calibrate the NIR instrument and a set used to validate and verify the calibration for the NIR instrument.

Individual variety predictions on the seed cotton laboratory measurements were made using the new optimized combined calibration data set with both NIR instruments (Table 2). The predictions utilizing the combined calibrations were more accurate and the discrepancy by variety for each instrument was reduced (Δ) compared to the original lint-only calibration. The prediction improvement was also reflected by a reduced standard deviation. This

combined calibration showed that there is a variety effect that was minimized by the combined sample set calibrations. Thus, individual variety predictions could now be made utilizing either NIR instrument. Similar results were found using the saw-ginned seed cotton lab lint data set.

Variety	MIC	NIR (1100-2300 nm)	
		Viavi 2200 MicroNIR	Brimrose Luminar 5030
DP393	5.33	5.12 (Δ= -0.21)	5.26 (Δ= -0.07)
SD	0.36	0.21	0.33
FM958	4.64	4.80 (Δ= +0.16)	4.64 (Δ= 0.00)
SD	0.28	0.29	0.38
SG105	5.09	4.89 (Δ= -0.20)	4.98 (Δ= -0.11)
SD	0.47	0.36	0.32

Table 2. Predictions on Seed Cotton Lab using the combined calibration

Individual variety predictions on the seed cotton greenhouse measurements were made using the new combined calibration data set with both NIR instruments (Tables 3). The predictions using both NIR instruments (Viavi and Brimrose) were different compared to the predictions performed with the original lint-only calibration data set. It also improved the accuracy of the prediction; reducing the delta and the standard deviation of the predictor varieties. This new combined calibrations showed that there was an environmental condition effects, and this impact was minimized with the combined sample sets calibrations.

Table 3. Predictions on Seed Cotton Greenhouse using the combined calibration

Variety	MIC	NIR (1100-2300 nm)	
		Viavi 2200 MicroNIR	Brimrose Luminar 5030
DP393	5.33	5.39 (Δ = + 0.06)	5.21 (Δ= - 0.12)
SD	0.36	0.27	0.33
FM958	4.64	4.75 (Δ= + 0.11)	4.66 (Δ= + 0.02)
SD	0.28	0.28	0.42
SG105	5.09	5.03 (Δ= - 0.06)	5.03 (Δ= - 0.06)
SD	0.47	0.42	0.37

Summary

The increase of accuracy and the reduction of the standard deviation of the individual variety predictions using the new optimized, combined calibration data sets were very encouraging, and they lead us to believe that including samples from different environments (laboratory and greenhouse) and fiber type conditions (lint and cottonseed) in the data sets makes the calibration more robust. The accuracy of the prediction was improved with both instruments. The two portable NIR instruments yielded different but similar results. It is advisable to use the instrument that fits the best the laboratory research objectives, considering its weight or size.

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References

American Society of Testing Materials. 2015. Standard Practice for Conditioning and Testing Textiles, ASTM D1776/D1776M-15. Book of ASTM Standards, ASTM International, West Conshohocken, PA, 5 pp.

Burns, D.. "Modern Near Infrared Reflectance Analysis". New Orleans, Louisiana: ACS Short Course. 1985. Pp 7-21.

Cotton Incorporated. "The Classification of Cotton". 2013. http://www.cottoninc.com/fiber/quality/Classification-Of-Cotton/Classing-booklet.pdf [accessed December 15, 2015]

Hequet, E., B. Wyatt, N. Abidi, D.P. Thibodeaux. "Creation of a Set of Reference Material for Cotton Fiber Maturity Measurements". Text. Res. J. 2006. 76(7): 576-586.

Perkampus, H.H.,. "NIR Spectroscopy". In: Encyclopedia of Spectroscopy. New York, NY: VCH, 1995. Pp 387-388.

Rodgers, J., S. Kang, C. Fortier, X. Cui, G. Davidonis, E. Clawson, D. Boquet, W. Pettigrew. "Preliminary Field Measurement of Cotton Fiber Micronaire by Portable NIR". Spectroscopy. 2010c. 25(9), 38-44.

Rodgers, J., S. Kang, C. Fortier, J. Montalvo, X. Cui, V. Martin. "Near Infrared Measurement of Cotton Fiber Micronaire by Portable Near Infrared Instrumentation". Text. Res. J. 2010b. 80(15): 1503-1515.

Rodgers, J., J. Montalvo, G. Davidonis, T. Von Hoven. "Near Infrared Measurement of Cotton Fiber Micronaire, Maturity, and Fineness—A comparative Investigation". Text. Res. J. 2010a. 80(9): 780-793.

USDA, AMS. "Cotton Classification, Understanding the Data". 2005. http://www.ams.usda.gov/sites/default/files/media/Cotton%20DB%20Understanding%20the%20Data.pdf [accessed December 15, 2015]

Workman, J.. "NIR Spectroscopy Calibration Basics". In: D. Burns and E. Ciurczak, editors. Handbook of Near-Infrared Analysis. New York, NY: CRC Press, 2001. Pp 123-150.