USE OF NEAR INFRARED SPECTROSCOPY IN COTTON MICRONAIRE ASSESSMENT

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Abstract

Micronaire is one of the more important cotton properties as it reflects fiber maturity and fineness. Automation-based high volume instrumentation (HVI™) measurement has been well established as a primary and routine tool of providing fiber micronaire and other quality properties to cotton breeders and fiber processors. This study examined the potential of near-infrared (NIR) spectroscopy for the prediction of cotton micronaire, by validating the calibration model with a validation set and also with a different crop-year fiber set. Results indicated that the development of a robust NIR model for micronaire assessment is feasible and could be utilized to screen micronaire component of new crop-year cottons at remote / breeding locations.

Introduction

Cotton micronaire is one of the more important cotton properties as it reflects fiber maturity (degree of secondary cell wall development) and fineness (weight per unit length) (Lord, 1956). Currently, automation-based high volume instrumentation (HVI™) measurement has been well established as a primary and routine tool of providing fiber micronaire and other quality properties to cotton breeders, fiber processors, and market regulators (ASTM, 2012; Frydrych and Thibodeaux, 2010). To acquire the micronaire value, conditioned fiber samples are measured by HVI™ protocol dominantly at limited and controlled locations where the system is available.

Near infrared (NIR) spectroscopy technique has been explored extensively for determining fiber micronaire (Liu et al., 2010; Montalvo and Von Hoven, 2004; Rodgers et al., 2010), because of its rapid and low-cost attribute that can be used, away from the standard laboratory, in places such as ginning and breeding sites as well as warehouses. This method largely measures the physical scattering of light from near-surface area of a fiber sample and requires a great number of training samples to build accurate and reliable calibration equations (models) through multivariate regression procedure. It takes time collecting the diverse samples and measuring the referenced micronaire values by the standard method beforehand. Previous studies by various researchers have demonstrated the ability of NIR technique in the determination of micronaire component with a high degree of success.

The main objective of this study was to examine NIR model performance, by validating the calibration models with an independent validation set and also with additional crop-year fibers.

Materials and Methods

Cotton Fibers

During 2011 and 2012 crop years, 20 entries (16 elite breeding lines and 4 commercial cultivars) were examined at the Clemson University Pee Dee Research and Education Center (Florence, SC), the Clemson University Edisto Research and Education Center (Blackville, SC), and the North Carolina State University Sandhills Research Station (Sandhills, NC). Each trial was arranged in a randomized complete block design with four replications. Each entry was grown in a two-row plot 10.7 m long with 96.5 cm spacing between rows. These plots were managed conventionally and followed the established local practices.

From each plot, 50 bolls were hand harvested and then were ginned on a 10-saw laboratory gin. Collected cotton fibers were conditioned at a constant relative humidity of 65 ± 2% and temperature of 21 ± 2 ºC for at least 24 hours, prior to subsequent fiber and yarn quality measurement as well as visible/NIR spectral acquisition.

Fiber Micronaire Measurement

An Uster® HVI™ 1000 system (Uster Technologies Inc., Knoxville, TN) was used to collect micronaire component of cotton fibers from five replicates on each sample. All measurements were performed at the Southern Regional Research Center of USDA's Agricultural Research Service (USDA-ARS-SRRC). The same instrument was utilized for continuous 2 crop-year samples and was calibrated throughout the study following the manufacturer's recommendation.

Visible/NIR Reflectance Spectral Measurement

Visible/NIR reflectance spectra were acquired on a Foss XDS rapid content analyzer (Foss NIRSystems Inc., Laurel, MD). Approximately 10 g of cotton fibers were pressed into a Foss coarse granular cell (3.8-cm wide x 15.2-cm long x 4.8-cm deep). Background was recorded with the use of an internal ceramic reference tile before scanning the samples. The log (1/Reflectance) readings were acquired over the 400 to 2500 nm wavelength range at 0.5 nm interval and 32 scans. Two spectra were collected for each of the cotton samples by repacking and the mean spectrum was obtained.

Model Development

All visible/NIR spectra were imported into GRAMS IQ application in Grams/AI (Version 9.1, Thermo Fisher Scientific, Waltham, MA) for partial least-squares (PLS) regression model development. On the order of the smallest to largest in micronaire property, two-thirds of spectra (or samples) were selected for calibration equation development and the remaining one-third (every 3rd sample) spectra were used for model validation. To optimize the accuracy of prediction models, the spectra were subjected to different combinations of both the spectral ranges (e.g., full and narrow regions) and the spectral pretreatments (e.g., mean centering (MC), multiplicative scatter correction (MSC), and the first and second derivatives). Full (one-sample-out rotation) cross-validation method was used, and the number of optimal factors chosen for the regression equation generally corresponded to the minimum of the predicted residual error sum of squares (PRESS). The saved regression equations were subsequently applied to both the validation samples that were harvested from the same crop-year and the test samples that were harvested from differing crop-year. Model accuracy and efficiency were assessed in the validation and independent set on the basis of the coefficient of determination (r^2), root mean square error of calibration (RMSEC), validation (RMSEV), or test (RMSEE), and also bias between NIR predicted and referenced micronaire values.

Results and Discussion

Cotton Fiber Micronaire Component and Visible/NIR Spectral Response

Figure 1 shows the representative micronaire-dependent log(1/R) spectra of cotton fibers in the spectral region between 400 and 2500 nm by averaging the spectra of neighboring micronaire values in the respective range of <3.5, 3.5-4.2,4.3-5.0, and >5.0. Although it suggests that cotton fibers with low micronaire have NIR bands in common with fibers having high micronaire, there appear to be some intensity changes in the entire spectral region induced by rising micronaire. There are at least five intense and broad bands with one (< 600 nm) in visible region (400-750 nm) and four (1490, 1935, 2105, and 2340 nm) in the NIR region (750-2500 nm). In this study, cotton fibers were processed at a small scale, thus the interferences from cotton plant parts could be presented. In general, the visible region of 400-750 nm contains the color information and represents a mixture of contributions from the pigmentations in cotton fibers (Liu et al., 2010), for example, flavonoids, degraded products between a reducing sugar and an amino acid, and also chlorophyll and its degradation derivatives in cotton plants. Whereas the origins of NIR bands differ from those in the visible region, they are mainly due to the (1st and 2nd) overtones and combinations of OH and CH stretching vibrations of cotton fiber cellulose (Burns and Ciurczak, 2001). The broad absorptions between 1150 nm and 1300 nm are from the 2nd overtones of CH stretching modes and their 1st overtones appear in the 1675-1860 nm region. Features in the 1300-1400 nm region are ascribed to combination bands of the CH vibrations. Broad and intense bands in the 1400-1675 nm region are due to the overlap of the 1st overtones of the OH stretching modes in hydrogen bonded forms. The strong bands at 1935 and 2105 are most likely attributed to the combination of OH stretching and deformation mode and the combination of OH and CO stretching vibrations in cellulose, respectively.

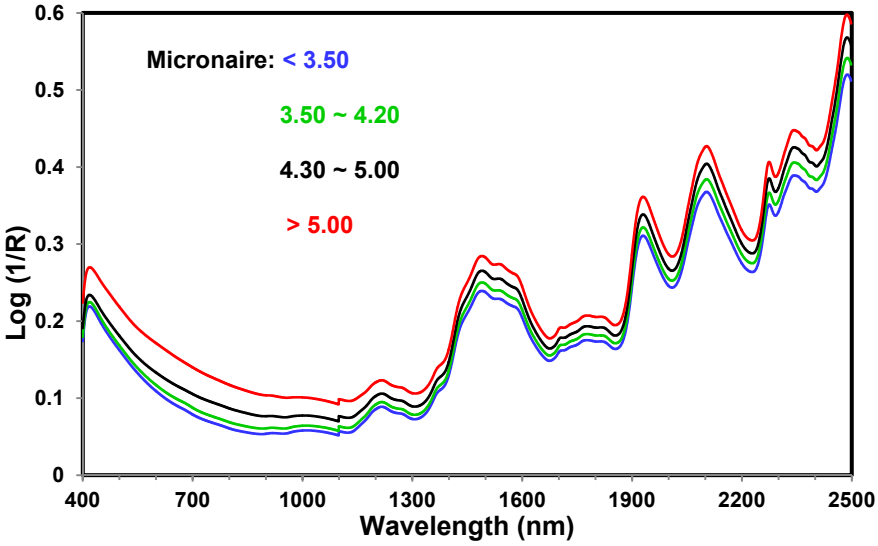

Figure 1. Typical visible/NIR log (1/R) spectra of cotton fibers with various micronaire readings.

Referenced Micronaire Values

Table 1 summarizes the range, mean, and standard deviation (SD) of referenced micronaire values for 2011 and 2012 cotton fibers in calibration and validation sets. Among a total of 238 2011 crop-year cottons, their fiber micronaire readings ranged from 4.10 to 5.68, while respective values of 143 2012 crop-year cottons were much smaller (3.44 to 4.84). Despite of the fact that two crop-year cottons were grown with nearly identical varieties, same locations, and similar agricultural practices, there were apparent and expected discrepancies in fiber micronaire. For either crop year fiber set or combined sample set, the variations of referenced micronaire values were in narrow range and cannot cover most of the variability in commercial cotton bales.

Table 1. Summary of range, mean, and SD for cotton micronaire component in calibration and validation sets.

Micronaire		Range	Mean	SD
2011 crop	Calibration set (n = 160)	4.10 - 5.68	5.00	0.32
	Validation set (n = 78)	4.26 - 5.59	5.01	0.30
2012 crop	Calibration set (n = 96)	3.44 - 4.84	4.12	0.28
	Validation set (n = 47)	3.56 - 4.66	4.13	0.26
Combined	Calibration set (n = 256)	3.44 - 5.68	4.67	0.52
	Validation set (n = 125)	3.56 - 5.59	4.68	0.51

Prediction Model

PLS models for micronaire constituent were developed from combinations of such spectral pretreatments as MC and 1st derivative in the 1105-2495 nm region. The use of 2nd derivative, along with other data processing, yielded relatively poor results (not shown). The statistics in calibration, validation, and independent test sets are compared in Table 2.

Table 2. Statistics of NIR model for micronaire prediction in calibration, validation, and test sets.[a]

Micronaire	Calibration Set			Validation Set			Test Set		
	R^2	RMSEC[b]	Bias[c]	r^2	RMSEV[b]	Bias[c]	r^2	RMSET[b]	Bias[c]
2011 cottons only	0.96	0.096	0.000	0.87	0.108	-0.004	0.72	0.160	-0.017
2012 cottons only	0.83	0.104	-0.002	0.63	0.159	-0.005	0.57	0.558	-0.519
Combined	0.95	0.120	0.000	0.93	0.134	-0.003			

[a] All spectral processing with mean centering (MC) and the first (1st) derivative. 6 optimal factors were used for all models.
[b] Root mean square error of calibration (RMSEC), validation (RMSEV), and test (RMSET).
[c] Bias = NIR predicted – HVI measured.

As anticipated, the model built from 2011 year-crop cottons exhibited high R^2 and r^2 as well as low RMSEC and RMCEV in calibration and validation set. When applying the model to independent 2012 cotton test set, r^2 decreased and RMSET increased expectedly. In addition, such a parameter as bias (defined as the difference between NIR predicted and measured micronaire) was used to assess the performance of calibration model. It is very reasonable to observe a greater elevation in bias within independent test set than among calibration and validation sets, mostly because these samples were measured one-year apart from calibration / validation samples and were not included in the model development. Comparative scatter plot of measured and NIR predicted micronaire in validation and independent test sets is given in Figure 2.

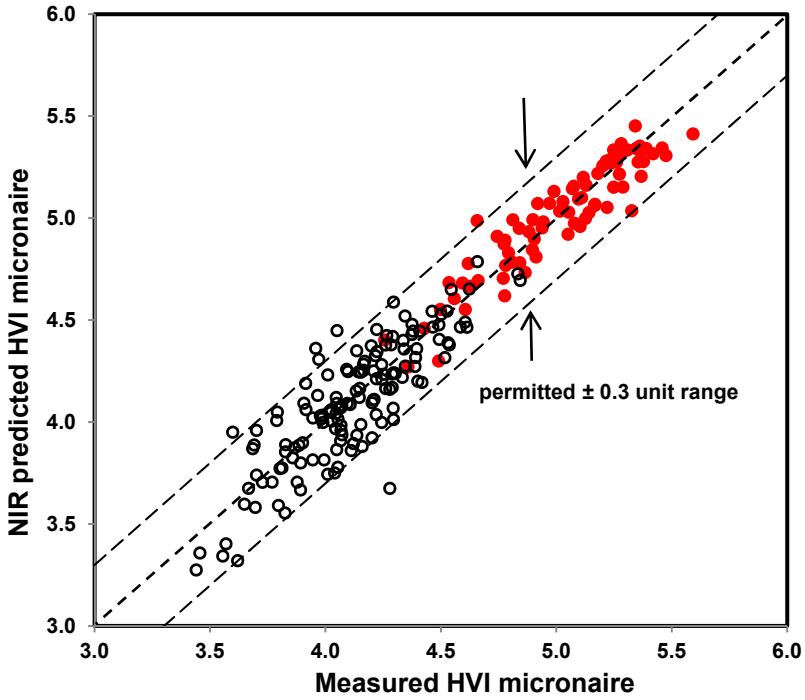

Figure 2. Correlations between measured and NIR predicted micronaire in validation set (●) and test set (○). Samples in test set were not included in the model development.

Another approach to examine the model performance was the use of ± 0.3 micronaire unit role (USDA, 2005). Within the 160 calibration samples, 78 validation samples and 143 test samples, there were 0 (0%), 1 (1.3%), and 5 (3.5%) samples that had prediction error (or difference) greater than the permitted range of 0.30 unit, respectively. In other words, this model resulted in over 96% of micronaire predictions that were within the acceptance range of ± 0.3 micronaire unit.

Similarly, the model was developed from 2012 crop-year cottons sorely and then was applied to independent 2011 cotton test set (Table 2). Apparently, bias in test set was so great (-0.519) that the model cannot be considered for any further application. One of rational concerns might be due to distribution weigh of 2012 cotton micronaire in calibration set.

The 2012 crop-year cottons were divided into calibration and validation samples, and then they were compiled into respective 2011 crop-year sample sets. In general, the recalibrated model was similar to the 2011 crop-year model in R^2, r^2, and bias, but with a greater RMSEC and RMSEV that were associated with a wider distribution of micronaire value. In the line of expectation, majority of the calibration samples (252 of 256, or 98.4%) and validation samples (122 of 125, 97.6%) were within 0.3 micronaire unit. Comparative scatter plot of measured and NIR predicted micronaire in validation set is given in Figure 3.

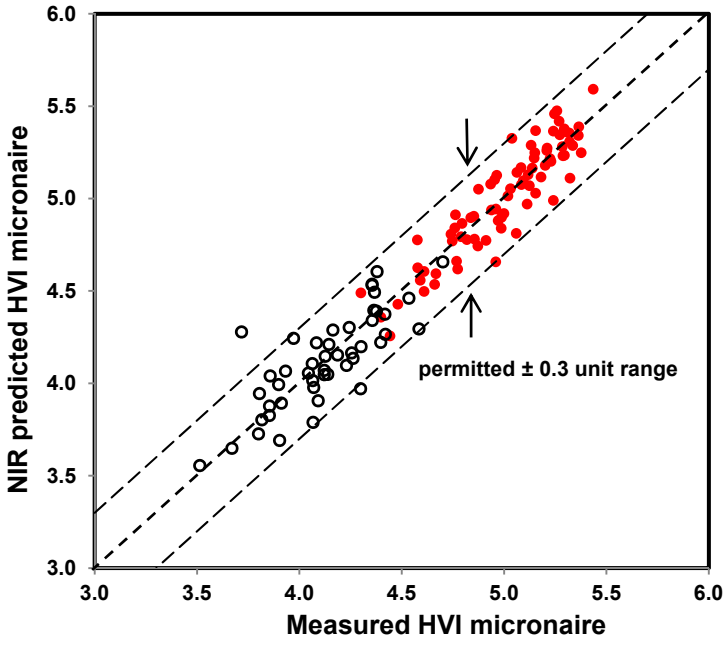

Figure 3. Correlations between measured and NIR predicted micronaire in validation set, 2011 crop-year cottons (●) and 2012 crop-year fibers (○).

Practical implementation of this NIR procedure for rapid and routine micronaire assessment at remote sites, fibers need to be conditioned at a standard environment, a few checking samples are necessary to examine the NIR model performance, and the NIR model is essential to be updated by including new crop-year (or location) cottons.

Summary

Relating NIR spectra with different crop-year cottons, the resultant models showed the potential of NIR technique in the precise and quantitative determination of cotton micronaire, implying its feasibility for micronaire screening at remote sites only when the samples were conditioned at a standard environment. Obviously, a few checking samples are necessary to compare the NIR predicted micronaire with conventionally HVI™ determined micronaire during the period, and also to update the NIR calibration model.

Acknowledgements

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