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Application of near infrared spectroscopy in cotton fiber micronaire measurement☆

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ABSTRACT

The term "micronaire" describes an important cotton fiber property by characterizing both the fiber maturity and fineness. In practice, micronaire is regularly measured in laboratories with the well established high volume instrumentation (HVI™) protocol. In most scenarios, cotton breeders/geneticists sent cotton breeding line field trial samples to laboratories equipped to use the HVI™ systems available for fiber micronaire determination. Researchers have previously investigated the use of NIR as an alternative means of measuring micronaire either at breeding sites or in standard laboratories. As a proof-of-concept investigation, this study collected both near infrared (NIR) spectra and HVI™ micronaire from a total of 381 cottons harvested in the 2011 and 2012 crop years. Partial least square (PLS) calibration model relating NIR spectral information to fiber HVI™ micronaire was developed and then applied to both a validation sample set from identical crop years and an independent test sample set from the 2014 crop year. Results indicated an acceptable bias (or differences between HVI™ measured and NIR predicted micronaire) and an over 97% correctly predicted micronaire (within ±0.30 micronaire unit) in an independent test set. Therefore, the development of a robust and effective NIR model for rapid laboratory micronaire assessment that would be applicable to remote/breeding locations is feasible.

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1. Introduction

Cotton, one of the most important and widely grown crops in the world, is a well-traded agricultural commodity primarily for its naturally produced textile fiber [1]. Cotton fiber's growth or development is considered to include at least four overlapping but distinctive phases: initiation, primary wall formation (elongation), secondary cell wall thickening (cellulose synthesis), and maturation [2]. The day of flowering is referred to as anthesis and the word "days post anthesis" (dpa) is commonly used to describe the cotton fiber growth. The fiber cells initiate at 0 dpa and then elongate to reach a fiber length of 22–35 mm within 20–25 dpa. The secondary cell wall synthesis starts around 15–22 dpa and continues for an additional 30–40 days until maturation, when the fibers dehydrate and collapse into flattened and twisted ribbons. Such a fiber evolution indicates a number of significant changes in fiber chemical composition, structure, and physical properties coinciding with various stages of development. The

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length of fiber growth period between floral anthesis and its harvest cannot be considered as a parameter to describe the degree of fiber maturation [3]. Fiber maturity has been accepted to reflect the degree of the secondary cell wall thickening relative to the diameter or fineness of the fiber [4].

Cotton micronaire is one of the most essential fiber characteristics in the cotton industry [5,6], as it reflects fiber maturity (degree of secondary cell wall development) and fineness (weight per unit length) simultaneously. In practice, 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 [7]. To determine the micronaire value, conditioned fiber samples with constant weight (~10 g) are measured by passing air through the fibers and then measuring the drop in pressure. Overall, the test for micronaire is very fast and accurate, therefore HVI™ measurement has been increasingly and routinely utilized in the cotton and textile industry from cotton breeding program to textile quality control [8–11], in addition to an industrial standard used internationally and domestically to classify commercial-ready cottons.

During the development and testing of advanced breeding lines and candidate cultivars, cotton breeders typically harvest thousands of fiber samples from one crop year. Fiber quality data are routinely collected on these samples, and they must be sent to outside fiber quality laboratories where HVI™ systems are available. It would be both desirable and beneficial to cotton breeders if more robust and low-cost quality measurements were available. One of the potential techniques is near infrared (NIR) spectroscopy, covering the 750–2500 nm (or 13,300–4000 cm^{-1}) region and representing the overtones and combination bands of the fundamental absorptions observed in the mid-IR spectral regions of cotton fiber cellulose [12].

NIR has been explored extensively for determining fiber micronaire over the years [13–19], because of its rapid, low-cost, and portable attribute that can be used away from the standard laboratories. This method largely measures the physical scattering of light from near-surface area of a fiber sample and requires a great number of training or calibration samples to develop accurate and reliable calibration equations (models) through multivariate regression procedure. Clearly, it takes time collecting the diverse samples and measuring the referenced micronaire values by the standard laboratory method in advance. Previous studies by various researchers have demonstrated the potential of NIR technique to determine micronaire with a high degree of success.

The main aim of the current study was to examine the applicability of NIR micronaire model developed from earlier crop year cottons to newly crop year fibers, by testing their micronaire predictions.

2. Materials and methods

2.1. Cotton samples

In each of 2011, 2012, and 2014 crop years, a total of 20 entries (16 elite breeding lines and 4 commercial cultivars) were grown in four replicated field tests at the Clemson University Pee Dee Research and Education Center near Florence, SC (Florence) on a Norfolk loamy sand soil, the Clemson University Edisto Research and Education Center near Blackville, SC (Blackville) on a Barnwell loamy sand soil, and the North Carolina State University Sandhills Research Station near Jackson Springs, NC (Sandhills) on a Candor sand soil. Each trial was arranged in a randomized complete block design with four replications. Each entry was planted in a two-row plot 10.7 m long with 96.5 cm spacing between rows. Plots were managed conventionally and followed the established local practices.

From each plot in each trial, 50 bolls were picked by hand. These boll samples were subsequently ginned on a 10-saw laboratory gin and lint fibers were collected. In every crop year, cotton lint fibers were conditioned at a constant relative humidity of 65 ± 2% and temperature of 21 ± 1 °C for at least 24 h, prior to routine fiber quality and NIR spectral measurement. Table 1 summarizes the fiber information about their origins and data collection at three locations over three crop years. Both fiber quality and spectral measurement were performed in August 2012, January 2013, February 2015 for the respective 2011, 2012 and 2014 crop year cottons.

2.2. Fiber quality measurement

Average micronaire values were obtained from five measurements on each sample by an Uster® HVI™ 900A system (Uster Technologies Inc., Knoxville, TN). All measurements were performed at the Southern Regional Research Center of USDA's Agricultural Research Service (USDA–ARS–SRRC). The same instrument was used for all fibers throughout the multiple year study.

2.3. NIR reflectance spectral acquisition

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 × 15.2-cm long × 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 400–2500 nm wavelength range at 0.5 nm interval and 32 scans. At least two spectra were collected for each of the cotton samples by repacking and the mean spectrum was obtained.

2.4. Micronaire model development

All 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 within each crop year fibers, 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

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