PRELIMINARY EVALUATIONS OF MICRONAIRE MEASUREMENTS BY NEAR INFRARED (NIR) SPECTROSCOPY FOR FIELD APPLICATIONS

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Abstract

Micronaire is a key quality and processing parameter for cotton fiber. The most common measurement of micronaire is a laboratory air resistance measurement with the Uster® High Volume Instrument (HVI). A program was implemented 1) to determine the capabilities of bench-top and portable Near Infrared (NIR) instrumentation to monitor cotton fiber micronaire and its component fiber properties (maturity/maturity ratio, fineness) and 2) to determine the ability of advanced portable instrumentation to monitor critical cotton fiber properties in the field. Comparative studies on optimum laboratory instrumental settings were performed. For laboratory measurements, the 400 Scans/Gain = 2/Glass Head condition was shown to be the best overall portable NIR method for fiber micronaire, and it was implemented for all portable laboratory micronaire measurements. The NIR measurement was fast (< 5 minutes per sample) and easy to perform. Field trials for micronaire on cotton bolls were successfully performed in 4 fields in Louisiana and Mississippi. Two field sampling systems were developed. Distinct micronaire differences were detected between cotton varieties and bolls with known micronaire differences.

Introduction

U.S. cotton fiber is classed by the Agricultural Marketing Service (AMS) of the USDA with the Uster® High Volume Instrument (HVI) for domestic and international use. Micronaire is a key cotton fiber quality property obtained with the HVI, expressed as a measure of the cotton fiber's resistance to air flow per unit mass. (Mogahzy and Broughton, 1992; USDA, 2005) The primary components of micronaire are fiber maturity and fiber fineness. Today, much of the U.S. cotton production is exported overseas. Increasing pressure is being placed on U.S. cotton to achieve high quality levels, which leads to the need for improved quality measurements. An important quality need is the development of new breeder quality tools for field and at-line measurements. At-line measurements are performed in the "manufacturing" or production area; field measurements are performed directly on the cotton boll and directly in the cotton field. Recent advances in portable spectroscopic instrumentation have the potential for achieving improved quality measurements, to include portable Near Infrared (NIR) instrumentation.

The development and use of NIR spectroscopy and applications continue to grow in the agriculture, fiber, and textile industries, to include cotton fiber maturity and micronaire. (Ghosh, 1985; Mogahzy, et. al., 1998; Montalvo and von Hoven, 2004; Rodgers and Ghosh, 2008). The NIR spectral region is normally considered to be from 1100-2500 nm, between the visible and infrared (IR) spectral regions, and its spectra are composed primarily of combination and overtone bands. The NIR method is secondary method and must be calibrated to a reference method.

A program was implemented 1) to determine the capabilities of bench-top and portable Near Infrared (NIR) instrumentation to monitor cotton fiber micronaire and its component fiber properties (maturity/maturity ratio, fineness) and 2) to determine the ability of advanced portable instrumentation to monitor critical cotton fiber properties of breeder samples both at-line and in the field. The program contains 3 components—a comparative program of bench-top and portable NIR analyzers (micronaire, maturity ratio/MR, fineness), laboratory trials of portable NIR analyzers for micronaire measurements, and initial filed/at-line trials of portable NIR analyzers for micronaire measurements.

Previous evaluations addressed the comparative program. (Rodgers, 2006) Cotton fiber samples were analyzed on four bench-top and two portable NIR analyzers from four vendors. The fiber samples were selected ginned cotton samples with wide property ranges for micronaire, maturity ratio (MR), and fineness, and the same sets of samples were measured on each NIR analyzer. Good spectral agreement was observed between the bench-top and portable NIR analyzers for the cotton fibers. For well-defined samples, the rapid measurement fiber micronaire by multiple bench-top and portable NIR analyzers was shown to be feasible. (Figure 1) No sample preparation was required (measure directly on the fiber). Analysis time was less than 5 minutes per sample. *The "universal" nature of NIR measurements of micronaire and its components were verified.*

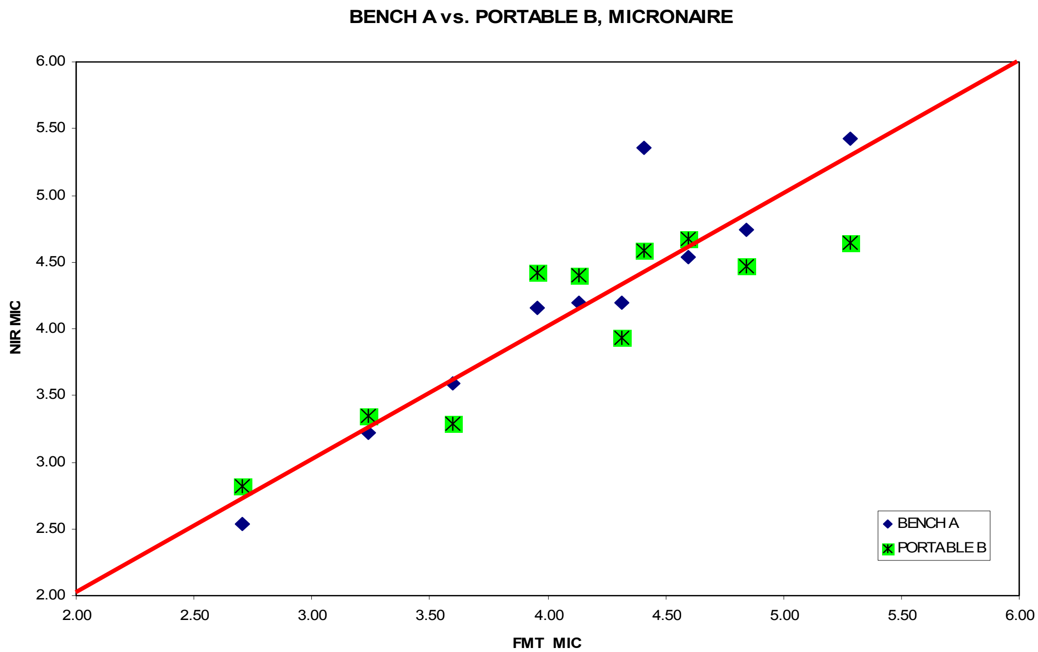

Figure 1. Comparative NIR Analysis of Micronaire, Portable and Bench-top NIR.

<u>Experimental</u>

The experimental information and results for the comparative evaluation was discussed above. The portable NIR analyzer investigations contained two components—laboratory and field evaluations. The reference micronaire values for all samples were obtained on the HVI unit. The end state criteria for both programs were 1) inter-instrument/method agreement (HVI micronaire – NIR micronaire) of ± 0.30 micronaire for ≥ 70% of the samples analyzed, 2) fast analyses (< 5 minutes analysis per sample), and 3) easy to use and maintain.

<u>Cotton Samples</u>

For the laboratory evaluations, 191 selected ginned cotton samples, with a wide range of micronaire values, were used. The 191 samples were from three well-defined sample sets. Each sample was measured 5 times on the portable NIR analyzer.

For the field evaluations, selected seed cottons from several varieties were obtained from 4 cotton fields, with the measurement performed directly on the cotton boll. The four locations were St. Joseph, LA (Louisiana Ag Extension Center, 2006 and 2007), Winnsboro, LA (Louisiana Ag Extension Center, 2007), and Stoneville, MS (Agricultural Research Service/ARS, 2007). Sixteen lots of 14 varieties were measured in the field. Each sample was measured a minimum of 3 times on the portable NIR analyzer.

NIR Analyzer

The Brimrose 5030 portable NIR analyzer was used in this evaluation. (Figure 2) The 5030 uses an acousto-optic tunable filter technique to generate the diffuse reflectance NIR spectra for each sample, and its NIR wavelength region is 1100-2300nm. Two 5 mm sampling ports/"heads" are available with the unit—an open port and a sapphire glass port (a thin glass sheet is placed on the port to facilitate fiber sampling consistency). In the laboratory, the 5030 is operated by a computer, using the SNAP software. Chemometric statistical modeling and calibrations are performed with the Unscrambler software package.

Figure 2. Brimrose 5030 Portable NIR Analyzer.

Results and Discussion

The results and discussion of the comparative program were given previously.

Laboratory Evaluations

A primary objective of the laboratory evaluations was the establishment of the optimal instrument settings. The main instrument variables were instrument gain, number of scans, and type of measuring head/sample port (with or without glass). Four conditions with these instrument variables were evaluated: 1) 300 scans/gain=1, No Glass Head (300/NG); 2) 300 scans/gain=1, Glass Head (300/G); 3) 400 scans/gain=2, No Glass Head (400/NG); and 4) 400 scans/gain=2, Glass Head (400/G). All 191 cotton samples were run under each of the four conditions on the 5030 unit, and their results compared.

Partial Least Squares (PLS) calibrations were completed for the 4 conditions with all 191 fiber samples ("best case" scenario). Often, NIR calibrations for micronaire incorporate the entire NIR spectral region from 1100-2300 nm. However, the moisture content for all cottons is not constant, even in a controlled laboratory, and moisture differences are expected in field applications. The primary moisture peak/band for cotton occurs at 1920 nm. Thus, in addition to developing calibrations over the entire NIR spectral range, calibrations were developed with the moisture peak at 1920 nm removed. For all conditions, very good calibration statistics were obtained, and over 75% of the samples agreed within ± 0.30 micronaire. (Figure 3) The best calibration statistics were observed with the moisture peak removed from the calibration. The best calibration results were observed with the 400/G condition (400 scans/gain=2, Glass Head). Sample analysis was very fast (< 5 minutes per sample), and the 5030 unit was easy to use. Thus, the NIR measurement of cotton micronaire by portable NIR analyzer is feasible.

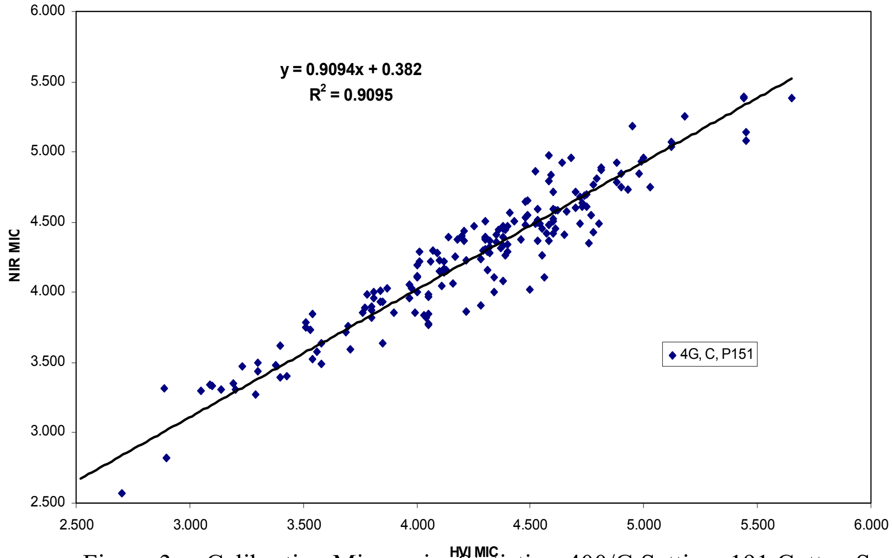

Figure 3. Calibration Micronaire Statistics, 400/G Setting, 191 Cotton Samples

To determine the full potential of the NIR measurement, calibrations should be tested against fiber samples that were not part of the NIR calibration (a "prediction set"). The 191 samples were split into two sub-sets—a 141 sample calibration set and a 50 sample prediction set. New PLS NIR calibrations were developed for each of the 4 instrument settings, and the calibrations were tested with the 50 sample prediction set. For all conditions, very good prediction statistics were obtained with the new PLS calibrations, with over 75% of the samples agreed within ± 0.30 micronaire (Figure 4). The best prediction results were observed with the 400/G condition (400 scans/gain=2, Glass Head), with 90% of the samples agreed within ± 0.30 micronaire. *For laboratory measurements, the 400/G condition was shown to be the best overall portable NIR method for fiber micronaire, and this condition was implemented for laboratory micronaire measurements with the Brimrose 5030.*

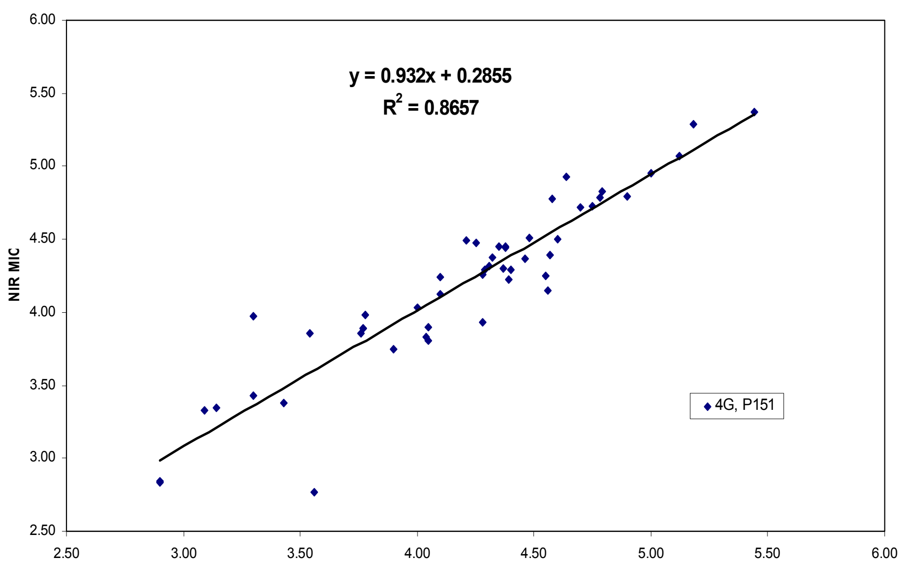

Figure 4. Prediction Micronaire Statistics, 400/G Setting, 50 Cotton Samples

Field Evaluations

Prior to field evaluations with the Brimrose 5030 portable NIR analyzer, a laboratory PLS micronaire calibration was developed and loaded into the 5030 unit. Field operations are performed with a battery attached to the 5030 unit. A macro was developed and installed for field measurements which permitted the display of the sample number and micronaire result, and the spectral results were stored for further data analysis. A backpack harness system was developed for carrying the 5030 unit into the field. Field evaluations were performed in 2006 and 2007 at 4 locations—St. Joseph, LA (Louisiana Ag Extension Center, 2006 and 2007), Winnsboro, LA (Louisiana Ag Extension Center, 2007), and Stoneville, MS (Agricultural Research Service/ARS, 2007). All measurements were taken directly on the seed cotton boll either in the field or near the field. Sixteen sample lots, representing 14 cotton varieties, were measured and collected for further analyses. Distinct micronaire differences between different varieties and known high/low micronaire samples were detected.

Two sampling systems were utilized—"plant measurement" and "vehicle measurement." (Figures 6 and 7) In plant measurements, the 5030 unit was placed on the sampler's back, the unit taken into the field, the unit's measuring head placed on the cotton boll, and the boll measured. In vehicle measurements, the seed cotton samples were collected in the field, the bolls are taken to the vehicle (where the NIR unit was set-up), the unit's measuring head placed on the cotton boll, and the boll measured. After field measurements, the seed cotton samples were placed in a conditioned laboratory. Laboratory measurements of the samples were performed on 5030 unit. The samples will be hand ginned and measured on the 5030 unit and the HVI (reference values).

Figure 6. Plant Measurements

Figure 7. Vehicle Measurements

Conclusions

A program was implemented to determine the capabilities of bench-top and portable NIR instrumentation to monitor cotton fiber micronaire and to determine the ability of advanced portable instrumentation to monitor critical cotton fiber properties of breeder samples in the field. Comparative studies on optimum laboratory instrumental settings were completed and implemented. For the prediction sample set, method agreement of ± 0.30 micronaire was achieved for >75% of the samples for all compared settings. For laboratory measurements, the 400 Scans/Gain = 2/Glass Head condition was shown to be the best overall portable NIR method for fiber micronaire. The NIR measurement was fast (< 5 minutes per sample) and easy to perform. Field trials for micronaire on cotton bolls were performed in 4 fields in Louisiana and Mississippi (16 sample lots representing 14 varieties). A macro was developed and installed for remote field operation (battery power). Two field sampling systems were developed—plant measurements and vehicle measurements. Distinct micronaire differences were detected between cotton varieties and bolls with known micronaire differences.

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