PRELIMINARY COMPARISONS OF PORTABLE NEAR INFRARED (NIR) INSTRUMENTATION FOR LABORATORY MEASUREMENTS OF COTTON FIBER MICRONAIRE

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Abstract

Micronaire is a key quality and processing parameter for cotton fiber. A program was implemented to determine the capabilities of portable Near Infrared (NIR) instrumentation to monitor cotton fiber micronaire both in the laboratory and in/near the field. Previous evaluations on one NIR unit demonstrated that acceptable laboratory measurement of cotton fiber micronaire by a portable NIR instrument was indeed feasible. A key component of this program is the demonstration of the robustness of NIR technology and methodology for micronaire measurements through the use of multiple portable NIR analyzers with different measurement techniques. In order to ascertain the robustness of the portable NIR technique, three commercial portable NIR analyzers were compared for instrument agreement of micronaire in the laboratory (laboratory measurement) on a large fiber sample set. Good spectral agreement was observed between the scanning portable NIR units. The preliminary micronaire results for the laboratory trials with multiple portable NIR units were very encouraging; with end-state criteria met for most of the portable NIR analyzers. Measurement time for all units was <3 minutes, and all units were easy to use and operate. Method optimization is continuing.

Introduction

Micronaire is a key cotton fiber property, and the primary components of micronaire are fiber maturity and fiber fineness. (Mogahzy and Broughton, 1992; Montalvo and Von Hoven, 2004; USDA, 2005; Wakelyn et. al., 2007) The Uster® High Volume Instrument (HVI) is used by the Agricultural Marketing Service (AMS) of the USDA to class (quality assessment) U.S. cotton fiber (lint), and the HVI is now being using globally for cotton classing. The HVI measures fiber micronaire using an air resistance technique, in which the fiber's resistance to air flow per unit mass is used to calculate the fiber's micronaire. The HVI method is well established, but the instrumentation is very expensive, requires a very stable laboratory environment, and requires well trained operators. In the global marketplace, cotton quality (and "goodness" of the quality assessment methods) is of primary importance. A need exists for improved quality assessment methods and measurements that would require minimum operator training and be more available and cost effective for cotton producers, ginners, textile mills, and laboratories. Recent advances in Near Infrared (NIR) instrumentation, to include portable NIR analyzers, have the potential for achieving rapid, accurate, and cost effective quality measurements that could act as a complement to the HVI measurements.

The application of NIR spectroscopy and technologies for textile products and fibers (including cotton) continue to grow. (Beck, 1996; Rodgers and Ghosh, 2008) The spectral region between 1100-2500 nm, between the visible and infrared (IR) spectral regions, is normally considered to encompass the NIR spectral region. The NIR spectra are composed primarily of combination and overtone bands, primarily for the NH, CH_i, and OH chemical groups. The NIR method is a secondary method and must be calibrated to a reference method.

Many studies have examined the NIR technique's capability to measure cotton fiber micronaire. (Ghosh, 1985; Mogahzy et. al., 1998; Montalvo and von Hoven, 2004; Rodgers and Ghosh, 2008) These studies relied primarily on bench-top, research grade NIR instruments. A previous evaluation on one portable NIR unit (Brimrose Luminar 5030) examined the ability of a portable NIR analyzer to monitor cotton fiber micronaire in the laboratory. (Rodgers et.al., in press) Optimal instrumental conditions for laboratory measurements were established, and the acceptable laboratory measurement of cotton fiber micronaire by a portable NIR instrument was shown to be feasible, with high R^2s, low residuals, and with ≤ 15% outliers (HVI-NIR micronaire agreement for ≥85% of the

samples was within ± 0.3% micronaire units). (Figure 1 for an example) All end-state criteria were met (≤ 30% outliers, analysis time < 5 minutes, measurement easy to perform).

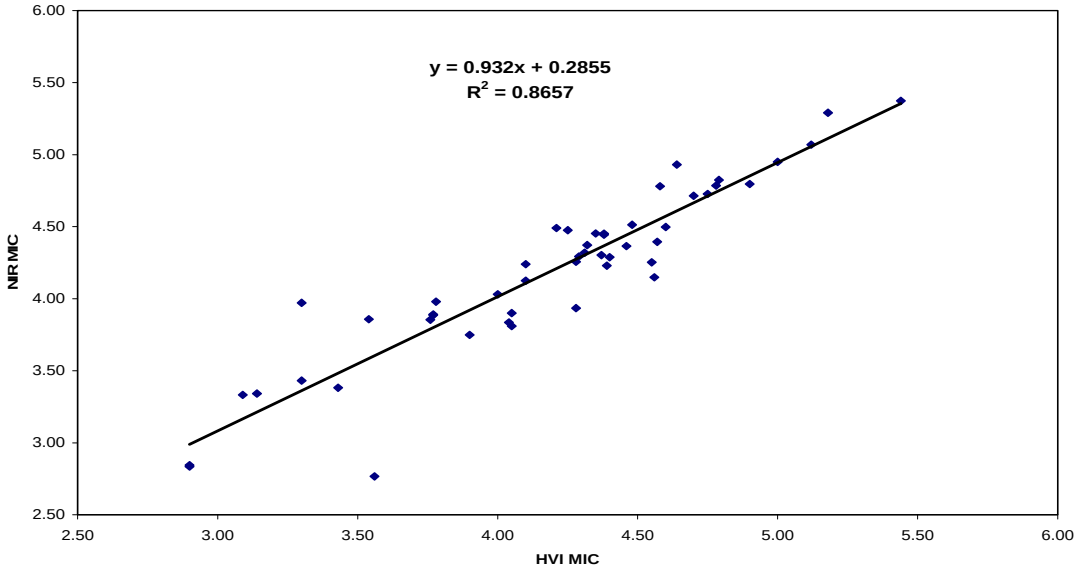

Figure 1. Validation/prediction set, micronaire, Brimrose 5030, 50 Cotton Samples (Rodgers, in press)

A key component of the program for laboratory measurement of micronaire by portable NIR analyzers is the demonstration of the robustness of NIR technology and methodology through the use of multiple portable NIR analyzers with different measurement techniques. The laboratory measurement of cotton micronaire with portable NIR analyzers was expanded to determine the capabilities of multiple portable NIR instrumentation and to ascertain the robustness of the portable NIR technique. Three (3) portable NIR analyzers, representing 3 different measurement technologies, were compared with a large, common cotton fiber sample set on their ability to monitor cotton fiber micronaire accurately, rapidly, and "easily." The preliminary results for this comparative evaluation are presented.

Experimental

For this preliminary comparative evaluation, three commercial portable NIR analyzers were evaluated on a large, well-defined cotton lint sample set (191 samples). The reference micronaire values for all samples were obtained on the HVI unit (5 measurements per sample). 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 (< 3 minutes analysis per sample), and 3) easy to use and operate.

Cotton Samples

The cotton lint samples for the laboratory evaluations consisted of 191 well-defined ginned cotton samples (wide range of micronaire values). Each sample was measured 5 times on the portable NIR analyzer.

Portable NIR Analyzers

Three portable NIR analyzers were compared for their capabilities to monitor cotton fiber micronaire in the laboratory—the Brimrose Luminar 5030, Polychromix Phazir, and Bruker LancIR. (Figures 2-4) A comparison of the primary characteristics of each instrument is given in Table I. The Brimrose 5030 uses an acousto-optic tunable filter (AOTF) technique to generate the diffuse reflectance NIR spectra for each sample, and its NIR wavelength region is 1100-2300 nm; the Polychromix Phazir uses an micro-electro-mechanical-system (MEMS) technique to generate the diffuse reflectance NIR spectra for each sample, and its NIR wavelength region is 1200-2400 nm; the Bruker LancIR uses a diode-array (dispersive) technique to generate the diffuse reflectance NIR spectra for each

sample, and its NIR wavelength region is 1100-2200 nm. All instruments were equipped with a glass sampling port, which facilitates fiber sampling consistency.

In the laboratory, all of the portable NIR analyzers were connected to a computer, using vendor-specific software (OPUS for Bruker, SNAP for Brimrose, PHAZIR MG for Polychromix). Chemometric statistical modeling, calibration development, and prediction were performed with the Camo® UNSCRAMBLER software package for the 5030 and Phazir and OPUS for the LancIR.

Figure 2. Brimrose 5030 Portable NIR Analyzer.

Figure 3. Polychromix Phazir Portable NIR Analyzer.

Figure 4. Bruker LancIR Portable NIR Analyzer.

Table I. Comparison of key characteristics, portable NIR analyzers

PROPERTY	PORTABLE NIR ANALYZER		
	BRIMROSE 5030	POLYCHROMIX PHAZIR	BRUKER LANCIR
TECHNOLOGY	AOTF	MEMS	DISPERSIVE
WAVELENGTH REGION (nm)	1100-2300	1200-2400	1100-2200
INSTRUMENT ANALYSIS TIME (sec)	20	5	30
RESOLUTION (nm)	2	10.5	4.3
SAMPLING AREA (mm Diameter)	6	6	25

Results and Discussion

The objective of the comparative laboratory evaluations was to determine the capabilities of multiple portable NIR analyzers to monitor critical cotton fiber properties in the laboratory, with initial emphasis on micronaire, in order to establish the robustness of portable NIR technology for laboratory fiber measurements. Measurements were performed on the same 191 cotton lint sample set for each portable analyzer, and their results compared.

For cotton fiber, the portable NIR analyzers were compared for their spectral agreement. Very good spectral agreement was observed between the 3 portable analyzers over their respective NIR spectral range up to 2200 nm. The key spectral features (absorbances) of cotton were observed for each analyzer.

Partial Least Squares (PLS) calibrations were performed on the spectral data from each analyzer for all 191 cotton samples (full calibration comparison). The PLS NIR calibrations were prepared over the entire NIR spectral region for each unit. For the 5030 and LancIR analyzers, very good calibration statistics were obtained, with over 85% of the samples agreeing to within ± 0.30 micronaire. (Table II, Figure 5) For NIR, the measurement was fast (< 3 minutes per sample for the slowest analyzer), and each analyzer was very easy to use and operate.

Table II. Comparison of calbiration results, multiple portable NIR analyzers, n=191

ITEM	CALIBRATION, MICRONAIRE (MIC), 191 SAMPLES			
	HVI	NIR		
		5030[1]	PHAZIR[2]	LANCIR[3]
AVG	4.22	4.22	4.22	4.22
R^2	NA	0.91	0.70	0.91
SEC	NA	0.17	0.28	0.18
No. > ± 0.3 (%)	NA	12.0	31.0	9.7

NOTE: [1]Brimrose 5030 NIR, [2]Polychromix Phazir NIR, [3]Bruker LancIR NIR

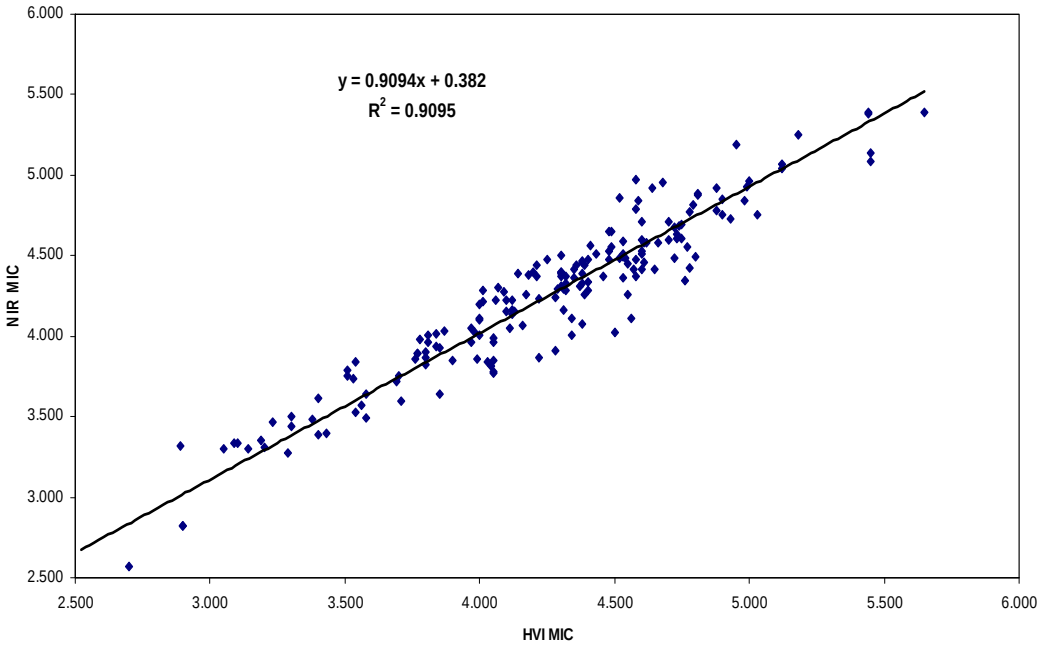

Figure 5. Example of calibration statistics for micronaire, Brimrose 5030, laboratory measurement, 191 Cotton Samples

A 141 sample calibration set and a 50 sample "prediction" or validation set were made from the 191 sample set. A prediction set is often used to determine the full potential of a NIR calibration and measurement, in which fiber samples that were not part of the NIR calibration are tested against the NIR calibrations. For each analyzer, PLS NIR calibrations were developed for the 141 calibration sample set, and the new calibrations were evaluated with the 50 sample prediction set. For the 5030 and LancIR analyzers, very good prediction results were obtained, with over 85% of the samples agreeing to within ± 0.30 micronaire. (Table III) Although the best prediction results were obtained for the 5030 analyzer, the differences between the 5030 and LancIR analyzers were small, and both units handily met the end-state criteria.

Table III. Comparison of prediction results, multiple portable NIR analyzers, n=50

ITEM	PREDICTION, MICRONAIRE (MIC), 50 SAMPLES			
	HVI	NIR		
		5030[1]	PHAZIR[2]	LANCIR[3]
AVG	4.21	4.22	4.14	4.24
R^2	NA	0.89	0.35	0.84
SDD	NA	0.20	0.50	0.24
No. > ± 0.3 (%)	NA	10.0	48.0	14.0

NOTE: [1]Brimrose 5030 NIR, [2]Polychromix Phazir NIR, [3]Bruker LancIR NIR

The Polychromix Phazir portable analyzer yielded poor laboratory results for both the calibration statistics and prediction results, and the end-state criteria for outliers was not met. (Tables II and III) Possible reasons for these results include a low number of scans and low wavelength resolution. Discussions are underway with the vendor.

Thus, the NIR measurement of cotton micronaire in the laboratory by multiple portable NIR analyzers was shown to be feasible, but additional investigations are required to discern the key instrumental and operational parameters that may influence the portable NIR measurement (as demonstrated by the Phazir results). For all analyzers, method optimization is continuing.

Conclusions

A program was implemented to determine the capabilities of multiple portable NIR analyzers to monitor critical cotton fiber properties in the laboratory, with initial emphasis on micronaire, in order to establish the robustness of NIR technology for laboratory fiber measurements. Preliminary comparative studies on 3 portable NIR analyzers, representing 3 different measurement technologies, were completed. Very good spectral agreement was observed between the 3 portable analyzers over their respective NIR spectral range up to 2200 nm. The preliminary results for the laboratory measurement of cotton fiber micronaire by multiple portable NIR units were very encouraging; with end-state criteria met for 2 of the portable NIR analyzers (Brimrose 5030 and Bruker LancIR). For both the full calibration and prediction sample sets, method agreement of ± 0.30 micronaire was achieved for >85% of the samples, with high R^2s and low residuals. Discussions are underway with Polychromix to ascertain possible improvements in the Phazir results. The NIR measurement of cotton micronaire in the laboratory by multiple NIR analyzers was shown to be feasible, but additional investigations are required. Method optimization is continuing.

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