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TEXTILE TECHNOLOGY

Fiber Micronaire, Fineness, and Maturity Predictions Using NIR Spectroscopy Instruments on Seed Cotton and Cotton Fiber, in and Outside the Laboratory

Jimmy Zumba, James Rodgers, and Matthew Indest

ABSTRACT

Micronaire is an important fiber quality parameter in the cotton and textile industry. Micronaire is a function of maturity (the degree of the fiber secondary wall development) and fineness (linear density). In prior research, bench-top near infrared (NIR) spectroscopy demonstrated the ability to measure micronaire, maturity, and fineness in and out of the laboratory. Small, portable handheld NIR instruments have been introduced and a program was established to measure micronaire in and outside the laboratory on seed cotton fiber and cotton lint, and consequently to measure maturity and fineness in the fiber. Adding new data to the original commercial lint-only samples, including data from different environments (laboratory and greenhouse) and fiber type conditions (laboratory ginned lint and seed cotton) made the calibration more robust, increasing the accuracy of the two NIR instruments (MicroNIR 2200 and Luminar5030) used in this experiment. Each instrument has its individual strengths. It is advisable to use the instrument that best fits the laboratory research objectives.

Micronaire is an important fiber quality parameter in the cotton industry (USDA-AMS, 2005). High or low fiber micronaire measurements can impact the downstream fiber processing and final product quality. The current traditional fiber quality analysis for micronaire uses a compression method, such as a high volume instrument (e.g., Uster® HVI™) and Fibronaire. HVI is a modular system that measures several cotton fiber quality parameters at once, including micronaire; Fibronaire is an instrument that measures micronaire only. The HVI is used by the USDA-Agricultural Marketing Service (AMS) as the primary instrumental method for cotton classing and it is used widely internationally for cotton classing and cotton quality measurements, including micronaire. The sample size for the HVI micronaire measurement is approximately 10 g; often, for samples whose sample size is not sufficient for HVI micronaire measurement or for laboratories that do not have a high volume instrument system, the Fibronaire is used (3.24-g sample size). The HVI and Fibronaire micronaire values are similar and in close agreement—often within ±1 micronaire unit (Rodgers et al., 2015). Both instruments use pressurized air and a compressed sample, measuring the air flow resistance in a given fiber weight and volume, but HVI can be expensive and both require controlled lab conditions. To perform cotton fiber micronaire measurements by traditional methods, the cotton first has to be harvested in the field, then the seed cotton is ginned and fiber samples for analyses labeled. Once labeled, the fiber samples are sent to the fiber lab for quality analysis. In the laboratory, fiber testing is performed under controlled conditions (21 ±1 °C and 65 ±2% relative humidity [RH]) after the fiber has been conditioned a minimum of 24 h (ASTM, 2015). Thus, from field harvesting to laboratory analysis, the fiber quality analysis is a tedious and lengthy process that can take days to complete.

Fiber micronaire is the function of maturity (the degree of the fiber secondary wall development) and fineness (linear density) (Wakelyn et al., 2007). An instrument used globally for measuring cotton fiber maturity and fineness (along with several other properties) is the Uster® Advanced Fiber Information System (AFIS). A recently introduced laboratory instrument, the Cottonscope, accurately and precisely measures cotton fiber fineness, maturity, and ribbon width using polarized light microscopy and image analysis on individual longitudinal fibers in a water medium (Gordon and Naylor, 2012; Paudel et al., 2013; Rodgers et al., 2012). The maturity and fineness results from the Cottonscope were more responsive to known changes in fiber maturity and

J. Zumba (formerly) and M. Indest, Oak Ridge Institute for Science and Education (ORISE), 1100 Robert E. Lee Blvd., New Orleans, LA 70124; and J. Rodgers*, USDA-ARS–SRRC, 1100 Robert E. Lee Blvd., New Orleans, LA 70124.
*Corresponding author: cottondoc1948@gmail.com

fineness than the AFIS (Rodgers et al., 2013). The Cottonscope also has the capability to measure micronaire, and the differences between the Cottonscope and HVI, and Fibronaire micronaires are acceptable and often within ±0.3 micronaire units (Rodgers and Delhom, 2015). The Cottonscope is especially useful for fiber measurements where only a small quantity of cotton sample is available (e.g., a single cotton boll down to a few milligrams of fiber). Indest (2015) demonstrated that only instruments that analyze individual fibers or fiber sections (e.g., Cottonscope) can capture how variable a cotton variety genotype is within an environment and that micronaire alone is a poor representation of both fiber maturity and fineness.

As noted above, the current laboratory micronaire techniques demonstrate the need for new, rapid, reliable, and accurate complementary techniques for the measurement of fiber micronaire and its components (maturity and fineness) that can be performed both in and outside the laboratory, without the need of a substantial amount of fiber, fiber preparation, and laboratory conditioning. A technology that addresses these needs is near infrared (NIR) spectroscopy. Burns (1985) described the NIR spectral region as the region between the visible and mid-infrared spectral region, from 800 to 2500 nm with the primary NIR spectra region occurring between 1100 to 2500 nm. NIR absorbance is obtained from the light interaction with the sample; the intensity of the diffuse reflectance from the sample received by the detector is dependent on both the sample chemical components (molecular absorbance) and physical properties (scattering/specular reflectance). NIR spectra are composed of overtones and valence vibrations of XH_n groups (CH_n, NH_n, and OH) and the combinations of the valence and bending vibrations of such groups (Perkampus, 1995; Workman, 2001). There are many advantages of using NIR analyzers that include rapid, accurate, and precise measurements; nondestructive sample analyses; no sample preparation needed; easy maintenance and operation; and ability to measure simultaneously several properties of interest. The main disadvantages of NIR are its need of a large number of samples to calibrate the instrument and its use of statistical modeling for the calibration.

NIR Spectroscopy in Cotton Fiber Analysis. NIR has been used extensively for both quantitative and qualitative (classification or identification) measurements in several industries (Fernández de la Ossa et al., 2014; Moffat et al., 2010; Montoya et al., 2013; Riccour, 2011; Sorak et al., 2012), including the textile and cotton industries (Devos et al., 2008; Rodgers and Beck, 2009; Tincher and Luk, 1985). Most of the NIR techniques reported for measuring cotton fiber micronaire in the laboratory concentrate on the use of bench-top, research grade NIR spectrometers (Montalvo and Von Hoven, 2004; Rodgers et al., 2010a). In prior cotton fiber studies using NIR, Liu et al. (2015) and Rodgers et al. (2010a) concluded that the broad absorptions between approximately1150 to 1300 nm were from the second overtones of CH stretching modes, and their first overtones appear in the approximately 1675 to 1860 nm spectral region; the absorptions between approximately1300 to 1400 nm spectral region was ascribed to combination bands of CH_n vibrations. The absorptions between approximately 1400 to 1500, 1900 to 2000, and 2050 to 2250 nm spectral regions were due primarily to OH overtone and combination bands, with the absorptions at approximately 1490 and 2100 nm due primarily cotton and the absorption at approximately 1930 nm often denoted as a "moisture" peak due to the OH group. Ramey (1982) performed exploratory investigations to estimate cotton fiber quality components, including micronaire. Thomasson and Shearer (1995) examined the relationships between NIR reflectance and fiber quality characteristics, including micronaire, using a bench-top, scanning NIR instrument from 1100 to 2500 nm. Montalvo et al. (1993, 1994), and Montalvo and Von Hoven (2004) examined the ability of a bench-top, scanning visible-NIR instrument (400-2500 nm) to predict fiber micronaire on cleaned cottons from successive growing seasons (2001 and 2002) in three states (Texas, Georgia, Mississippi). Liu et al. (2010) also examined cotton fiber quality attributes, including micronaire, using a bench-top, scanning UV-visible-NIR instrument (220-2200 nm). In addition, they examined the UV-visible-NIR instrument's capability to classify fibers according to their micronaire region. Ge et al. (2010) determined useful reflectance spectral wavebands and bandwidths for predicting several cotton fiber quality parameters, including micronaire. Rodgers et al. (2010a) established the universal nature of the NIR measurement of fiber micronaire, maturity, and fineness through a comparison of several bench-top, scanning instruments (Fourier transform and dispersive) on a small but property diverse sample set.

The miniaturization and portability of spectrometers have opened NIR technology to new applications. Research using small, portable NIR instruments on cotton fiber micronaire and its components (maturity and fineness) has not been extensive and has been performed primarily in the laboratory. Rodgers et al. (2010b, c) demonstrated the potential and capabilities of portable NIR analyzers to measure micronaire using a portable NIR instrument in the laboratory and in the cotton field. In laboratory evaluation, a comparative evaluation was used to develop and establish the laboratory measurement of fiber micronaire with a portable NIR instrument (high R^2, low residual, and few outliers). Vogt et al. (2011) concluded that excluding the water band from 1900 to 2000 nm during the data analysis had a minimal impact on the NIR micronaire prediction. The moisture band was of serious concern for field/outside the laboratory measurements as fluctuations in the relative humidity and temperature in field conditions could result in different moisture content in the fiber and could impact instrument performance, which could impact the water band and, thus, possibly the micronaire prediction capabilities of the portable NIR instrument. Rodgers et al. (2010b) performed preliminary field evaluations at three locations over two crop years or growing seasons. Measurements were made directly on the cotton bolls both in the cotton field and at nonlaboratory locations near the cotton field. Distinct micronaire trend levels (high-medium-low micronaire) were identified for 80% of the samples. In addition, the impact of different laboratory ginning methods (saw, roller, hand ginned) on the reference micronaire measurements and portable NIR instrument micronaire measurement was shown to be minimal (Rodgers et al., 2015).

As previously described, research on cotton fiber micronaire and its components (fiber maturity and fineness) by NIR spectroscopy has been performed primarily using bench-top NIR spectrometers, with emphasis on micronaire alone. In addition, the current cotton fiber quality analysis process, using HVI, AFIS, or Fibronaire, is a laboratory-only process, but measurements of micronaire and its components are desired in nonlaboratory environments and with small samples. These factors have revealed the need for rapid and accurate in-field and outside the laboratory/on-site analysis for cotton fibers micronaire and its components maturity and fineness. The outside the laboratory NIR measurements would complement, not replace or supplant, the laboratory fiber quality measurements and process. Recently, small and micro NIR spectrometers have been introduced. Cotton industry stakeholders from Cotton Incorporated, cotton breeders, and international researchers have shown increasing interest in the measurement of fiber micronaire, maturity, and fineness using small, portable NIR spectroscopy instruments for both laboratory and outside the laboratory micronaire. A program was implemented to determine the ability of new portable NIR instruments to accurately measure micronaire, fineness, and maturity in cotton lint and seed cotton, to develop protocols and to increase the robustness of the calibration data set, and to reduce variability and improved predictability of the NIR results. All measurements were performed in the greenhouses and laboratories at the Southern Regional Research Center of the Agricultural Research Service of the United States Department of Agriculture (USDA, ARS, SRRC) in New Orleans, LA.

MATERIAL AND METHODS

Cotton Samples and NIR Measurements. NIR measurements were made using two portable units, the Viavi MicroNIR 2200 and the Brimrose Luminar 5030, on seed cotton (laboratory and greenhouse) and saw-ginned cotton lint (laboratory). The vendor's recommended operational procedures for each NIR instrument were followed. The portable Brimrose Luminar 5030 NIR (Brimrose Corp. America, Sparks, MD) uses Acousto-Optic Tunable Filter (AOTF) technology and the spectra are filtered by the interaction between ultrasonic waves and light in an acousto-optic crystal (Brimrose, 2015). The Brimrose Luminar 5030 has a spectral range of 1100 to 2300 nm. Viavi Solutions (formerly JDS Uniphase; San Jose, CA) introduced new miniaturized spectrometers called the MicroNIR spectrometers. The MicroNIR 2200 was used in this evaluation, as it has an extended spectral range of 1150 to 2150 nm. The ultra-compact MicroNIR spectrometers uses thin-film linear variable filter (LVF) technology for the light dispersing element, and the miniaturization of spectrometers are partly driven by microelectromechanical systems (MEMS) (O'Brien, et al., 2012). The instrument is small, with the light source, collection optics, and electronics housed in a case that is less than 2 in. in diameter and height (JSDU, 2014). The NIR results from MicroNIR 2200 were compared to the NIR results from the Brimrose Luminar 5030 instrument (used in previous field evaluations) to determine the feasibility of the MicroNIR 2200 for laboratory and

outside the laboratory measurements of micronaire, maturity, and fineness on individual cotton bolls.

A new accession (LA2010) composed of three cotton check varieties (FM958, DP393, and SG105) from the Cotton Incorporated Regional Breeders Testing Network (RBTN) 2010 crop grown in Alexandria, LA., were used for this evaluation (n = 222). Three environmental conditions–cotton-type measurement conditions were made: greenhouse seed cotton measurements (GSC, n = 222), laboratory seed cotton measurements (LSC, n = 222), and laboratory lint measurements (LL, n = 222). The seed cotton measurements were made on individual cotton bolls. In the greenhouse, seed cotton measurements were made under high temperature and high humidity (> 27 ˚C and 80% RH) conditions. In the laboratory, the NIR measurements on seed cotton and saw-ginned seed cotton lint were performed in a conditioned laboratory for a minimum of 24 h (21±1 ˚C and 65±2%)(ASTM, 2015). The seed cotton was ginned on a laboratory table-top 10-saw gin (Dennis Manufacturing, Athens, TX).

The Cottonscope instrument was used to obtain the reference maturity and fineness values for individual cotton bolls/lint samples. Because the original laboratory lint-only calibrations for micronaire were based on HVI measurements, Fibronaire micronaire measurements and results were used as the reference values for micronaire (excellent micronaire method agreement between HVI and Fibronaire [Rodgers et al., 2015]). HVI measurements were not used for micronaire reference values for the new three cotton varieties accession because HVI requires a sample size of approximately 10 g; however, Fibronaire micronaire was used because Fibronaire micronaire samples require a minimum of 3.24 g, and single cotton boll lint weighted between 1.0 to 1.5 g each. Due to sample weight needed to perform these Fibronaire measurements, a minimum of three samples/fluffs were combined based on micronaire Cottonscope values (for example, three low Cottonscope micronaire bolls were combined and yielded a low micronaire sample for Fibronaire; three high Cottonscope micronaire bolls were combined and yielded a high micronaire sample for Fibronaire, and so on). After combining the samples to obtain the micronaire reference values using the Fibronaire, those combined samples spectral values were averaged so that the total number of samples were 73 each for GSC, LSC, and LL (Rodgers et al., 2010c, 2015). For complete data analysis the micronaire values were obtained using the Fibronaire, and the maturity and fineness were obtained using the Cottonscope. The samples were split into two samples sets, a set used to calibrate the NIR instruments, and a set to validate and verify the calibration for the NIR instruments (Table I).

The prediction set of the LL, LSC, and GSC sets were evaluated for micronaire, maturity, and fineness using the present laboratory lint-only calibrations (140 calibration cottons). The laboratory lint-only calibration was developed previously from a well-defined and diverse set of lint cottons from a reference set of 104 domestic and international cottons (Hequet et al., 2006) and 36 cottons covering four crop years from Texas, Georgia, and Mississippi, and AMS micronaire standards, covering a wide micronaire range (2.52-5.65 mic units). After the initial evaluation with the laboratory lint-only calibration (140 calibration cottons), the calibration set was expanded with the LL, LSC, and GSC calibration samples to determine if it was feasible to improve the robustness and prediction capability of the original lint-only NIR calibrations. The combined data sets (lint-only plus new accession) were evaluated with the prediction samples of the LL, LSC, and GSC sets (n = 37 each; Table 1).

Table 1. Calibration and validation data sets

Data set		Calibration				Prediction			
		N	MIC	MR	FN	N	MIC	MR	FN
LA2010	Min	36	3.69	0.83	176.71	37	4.02	0.87	178.24
	Max	36	5.93	1.06	246.71	37	5.87	1.07	248.04
	Avg	36	4.99	0.97	209.92	37	5.02	0.97	211.07
	SD	36	0.52	0.05	16.62	37	0.47	0.04	16.17
140 Samples	Min	140	2.52	0.46	136.29				
	Max	140	5.65	1.10	245.11				
	Avg	140	4.23	0.87	181.99				
	SD	140	0.59	0.10	21.05				

MIC = Micronaire; MR = Maturity; FN = Fineness

Data Analysis. The micronaire, maturity, and fineness data were analyzed using SAS PROC GLM. Estimates of means and standard errors were generated through LS MEANS with calibration and prediction as class and micronaire, maturity and fineness as response. Pearson correlation was performed using PROC CORR among micronaire, maturity, and fineness at the 0.05 level of probability (version 9.4; SAS Institute, Cary, NC).

The primary comparison parameters and statistics of interest were the average of the sample sets (AVG), the residual analysis SDD (Standard Deviation of Differences, the standard deviation of the differences between the reference method and the NIR methods for each sample; a residual analysis), and the bias/difference (Δ) between the reference micronaire, maturity, and fineness AVG and the NIR determined AVG for each sample set. The lower the SDD and Δ, the better the method agreement between the reference values and the NIR results. The target Δ values for acceptable NIR performance are ±0.3 for micronaire, ±0.1 for maturity and ±15 for fineness; if Δ values were outside of these property ranges, the samples were considered outliers.

The spectral data from the MicroNIR 2200 and Luminar 5030 units were 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 partial least-squares calibrations. Scatter plots of the residuals were generated in Unscrambler to verify that the assumptions of linearity and normality for residuals were met. Data were transformed and optimal spectral results were obtained using first derivative Savitzky-Golay (SG) with 9-points smoothing algorithms. The MicroNIR 2200 and Luminar 5030 instruments were compared to each other.

RESULTS AND DISCUSSION

Micronaire, Maturity, and Fineness Mean Differences and Correlations. The descriptive statistical values for the calibration and prediction combined data set are shown in Tables 2A and 2B. In this experiment, a new accession of three different commercial varieties (n = 36) was added to the well-defined lint-only calibration data set (n = 140), and then the combined calibrations was validated on the new accession prediction data set (n = 37). For this experiment, as a group, it was preferred not to have a difference statistically between the new accession calibration and the new accession validation (prediction) data sets to reduce the impacts of seed cotton and environmental conditions (e.g., outside the laboratory measurements in the greenhouse) on the NIR results. Table 2A demonstrates that there were no differences statistically between the calibration and prediction data sets for all three fiber parameters (micronaire, maturity, and fineness).

Table 2A. New accession data set mean analysis

Method	N	Micronaire[z]	Maturity[z]	Fineness[z]
Calibration	36	4.99 a	0.97 a	209.92 a
Prediction	37	5.02 [a]	0.98 [a]	211.08 [a]

*$p < 0.05$

Table 2B. Calibration and prediction data set analysis

Method	N	Micronaire[z]	Maturity[z]	Fineness[z]
Calibration	176	4.39 b	0.89 b	187.70 b
Prediction	37	5.02 [a]	0.98 [a]	211.08 [a]

[z] $p < 0.05$

The descriptive statistics for the combined calibration data set and new accession prediction data set were analyzed and shown in Table 2B. Because the combined calibration has a larger number of samples, which includes the lint-only calibration set with 140 samples, it was expected to be different statistically from the group means. For the calibration set, individual samples have a wider range of micronaire, maturity, and fineness values than the average micronaire, maturity, and fineness for the prediction set, so there is not a concern that the micronaire, maturity, and fineness in the prediction data set are different statistically of the calibration set.

Table 3. Mean analysis for the calibration data set

Groups	N	Calibration[z]		
		Micronaire	Maturity	Fineness
Original lint-only	140	4.23 [a]	0.87 [a]	181.99 [a]
DP 393	12	5.29 [c]	1.00 [b]	203.55 [b]
FM 958	12	4.67 [b]	0.97 [b]	206.07 [bc]
SG 105	12	5.02 [bc]	0.95 [b]	220.14 [c]

[z] All were statistically different at $p < 0.0001$

In Table 3, the results demonstrate that the average property values for the new varieties are higher than the average property values for the original lint-only samples (n = 140). The well-defined, original lint-only sample group was statistically different compared to the new fiber accession (FM958, DP393, and SG105) groups for micronaire, maturity, and

fineness, and it was the lowest average value on all three variables. Thus, within the calibration and prediction data set, average differences among the original lint-only and the new varieties were expected to improve the robustness of the calibration data set. The well-defined lint-only sample group exhibited the lowest average micronaire (4.23) and was statistically different from the mean average micronaires of the new varieties (FM958, DP393, and SG105). The FM958 cotton lint exhibited the lowest mean micronaire (4.67), and the DP393 cotton lint had the highest mean micronaire (5.29) for the new fiber accessions. Further, for maturity and fineness, there were statistical differences between the original lint-only group and the new fiber accession groups. The prediction data set showed similar statistical differences as observed for the calibration data set.

The fiber micronaire results were highly correlated with the maturity and fineness results within the calibration data set (Table 4). The variables in the prediction data set were also highly correlated, with a p value < 0.0001 in both cases. The correlations were expected, as micronaire is determined by the fiber's maturity and fineness. The micronaire and fineness were the highest correlated, with a correlation value of 0.90 for the calibration data set. The correlations between maturity and fineness were the lowest. When the fiber's maturity is increased, the fiber becomes larger, coarser, and has a higher fineness size (higher mtex). However, the maturity and fiber coarseness do not increase at the same rate or extent, with some cottons (e.g., extra long staple) exhibiting low coarseness (very fine) at high maturities.

Table 4. Combined calibration correlations

Groups	N	Mean	SD	Correlations[z]		
				Mic	Maturity	Fineness
1. Micronaire	176	4.39	0.65	1		
2. Maturity	176	0.89	0.1	0.67	1	
3. Fineness	176	187.7	23.13	0.9	0.41	1

[z] All were statistically different at $p < 0.0001$

Micronaire, Maturity, and Fineness Validations Using Original Lint-only Calibrations. NIR measurements were made using both the Viavi MicroNIR 2200 and the Brimrose Luminar 5030 instruments on a new cotton fiber accession of three commercial varieties (FM958, DP393, and SG105) under three conditions: GSC, LSC, and LL under controlled conditions. Previously developed NIR calibrations from a well-defined and uniform set of 140 lint samples (original lint-only laboratory calibrations) were used for validation (projection) of these three varieties under these three conditions.

The samples results under each measurement condition (LL, LSC, and GSC) for micronaire are presented in Table 5, for maturity in Table 6, and for fineness in Table 7 (both instruments and all cotton varieties). Across all three varieties (FM958, DP393, SG105) and within each fiber property (micronaire, maturity, and fineness), large differences were observed between the reference values (MIC) and AVG, as observed for Δ, the SDD, and the number of predicted samples (OUT %) outside of the targeted property ranges for all samples (micronaire Δ > ±0.30; maturity Δ > ±0.10; fineness Δ > ±15.00).

As indicated in Table 5, both instruments exhibited large differences between the reference and NIR predicted values, as indicated for each variety by high Δ, high SDD, and a large number of outliers for all varieties combined. The Δ results for each variety demonstrated a variety effect for all three conditions and for each instrument, with FM958 being distinctly different from DP393 and SG105. Between conditions (LL, LSC, GSC), a definite AVG and Δ difference was observed between the lint and seed cotton cotton-types for all three varieties for the MicroNIR 2200 unit (cotton-type impact); the AVG and Δ difference between the lint and seed cotton results (under the same condition) was much smaller for the Luminar 5030 unit (minimal cotton-type impact). The rationale for the different instrumental responses is that the two instruments employ a different measuring surface. The MicroNIR 2200's measuring surface is flat and wider than that of the Luminar 5030, which is similar to a tapered cone; thus, the MicroNIR 2200 measuring surface compresses the entire sample, permitting some cotton seed to be present at the measuring surface, whereas the Luminar 5030's cone measuring surface is smaller in contact size and able to move around the cottonseed more readily, resulting in only slight or no measurement of the seed surface. In addition, between conditions (LL, LSC, GSC), a definite AVG and Δ difference was observed between the lab and greenhouse environmental conditions for all three varieties for the Luminar 5030 unit (environmental condition impact); the AVG and Δ difference between the lab and greenhouse results for seed cotton was much smaller for the MicroNIR 2200 unit (minimal environmental condition impact). Differences in hardware between the two NIR instruments result in the observed environmental condition impact differences observed for the instruments.

Table 5. Micronaire results on the original lint-only calibration

GROUP		MIC	MicroNIR 2200				Luminar 5030			
			AVG	Δ	SDD	OUT(%)	AVG	Δ	SDD	OUT(%)
ALL	LL	5.29	4.82	-0.47	0.29	32	5.97	0.68	0.28	32
	LSC		4.50	-0.79	0.32	32	5.83	0.53	0.47	59
	GSC		4.42	-0.87	0.29	38	4.61	-0.68	0.44	57
DP393	LL	5.17	4.87	-0.30	0.26		5.84	0.67	0.26	
	LSC		4.41	-0.76	0.31		5.39	0.22	0.37	
	GSC		4.43	-0.74	0.31		4.48	-0.69	0.44	
FM958	LL	5.32	4.71	-0.61	0.16		6.11	0.79	0.26	
	LSC		4.51	-0.81	0.14		6.21	0.89	0.32	
	GSC		4.24	-1.08	0.17		4.65	-0.67	0.45	
SG105	LL	5.40	4.86	-0.54	0.34		5.98	0.58	0.28	
	LSC		4.58	-0.82	0.46		5.91	0.51	0.46	
	GSC		4.61	-0.79	0.27		4.72	-0.68	0.47	

MIC = Micronaire; LL = Lab measured lint samples; LSC = Laboratory measured seed cotton samples; GSC = Greenhouse measured seed cotton samples

Table 6. Maturity results on the original lint-only calibration

GROUP		MR	MicroNIR 2200				Luminar 5030			
			AVG	Δ	SDD	OUT(%)	AVG	Δ	SDD	OUT(%)
ALL	LL	0.98	0.98	0.00	0.00	14	1.17	0.19	0.13	46
	LSC		0.84	-0.14	0.07	18	1.21	0.23	0.19	71
	GSC		0.81	-0.17	0.11	30	1.38	0.40	0.32	81
DP393	LL	1.00	0.99	-0.01	0.05		1.11	0.11	0.10	
	LSC		0.85	-0.15	0.08		1.04	0.03	0.08	
	GSC		0.82	-0.18	0.09		1.09	0.09	0.36	
FM958	LL	0.96	0.97	0.01	0.07		1.25	0.29	0.10	
	LSC		0.86	-0.10	0.08		1.38	0.42	0.10	
	GSC		0.81	-0.15	0.14		1.67	0.71	0.18	
SG105	LL	0.96	0.99	0.03	0.07		1.14	0.18	0.11	
	LSC		0.82	-0.14	0.06		1.22	0.27	0.13	
	GSC		0.80	-0.16	0.09		1.38	0.42	0.38	

MR = Maturity; LL = Lab measured lint samples; LSC = Laboratory measured seed cotton samples; GSC = Greenhouse measured seed cotton samples

Table 7. Fineness results on the original lint-only calibration

GROUP		FN	MicroNIR 2200				Luminar 5030			
			AVG	Δ	SDD	OUT(%)	AVG	Δ	SDD	OUT(%)
ALL	LL	210.98	181.11	-29.87	14.22	32	271.65	60.67	14.80	32
	LSC		171.74	-39.24	17.59	35	305.21	94.23	20.55	45
	GSC		164.05	-46.93	22.39	51	157.04	-53.94	40.43	71
DP393	LL	206.10	178.07	-28.03	12.92		274.55	68.45	12.13	
	LSC		170.85	-35.35	13.39		299.45	93.36	19.44	
	GSC		162.80	-43.30	20.23		181.41	-24.69	38.61	
FM958	LL	203.64	179.41	-24.23	11.16		266.62	62.97	14.18	
	LSC		169.81	-33.83	16.15		303.24	99.60	20.85	
	GSC		158.45	-45.19	26.18		123.88	-79.76	22.60	
SG105	LL	223.81	186.14	-37.67	15.21		273.69	49.88	11.66	
	LSC		174.68	-49.13	19.20		313.48	89.67	20.71	
	GSC		171.15	-52.66	19.71		164.70	-59.11	36.80	

FN = Fineness; LL= Lab measured lint samples; LSC = Laboratory measured seed cotton samples; GSC = Greenhouse measured seed cotton samples

For maturity, the results for the LL, LSC, and GSC samples, using the original lint-only calibration, are shown in Table 6 for both instruments by cotton variety and all varieties combined. As observed for the micronaire results, large differences often were observed for each variety between the reference MR values and predicted NIR MR values for AVG and Δ (> ±0.1). In addition, a large number of outliers were observed for all varieties combined. However, the differences observed for maturity were overall in better agreement than observed for micronaire. The MicroNIR 2200 exhibited the best overall Δ agreement with the reference values (lower AVG and Δ), and fewer outliers were observed. The Δ differences for LL observed with the Luminar 5030 were bias-related primarily. The Δ results by variety demonstrated a minimal variety impact MicroNIR 2200 results, but a distinct variety impact was observed with the Luminar 5030 results, especially for FM958. As observed for the micronaire results, between conditions (LL, LSC, GSC), a definite difference was observed between the lint and seed cotton cotton-types for the MicroNIR 2200 unit (cotton-type impact), and a definite difference was often observed between the lab and greenhouse environmental conditions for the Luminar 5030 unit (environmental condition impact).

For fineness, the results for the LL, LSC, and GSC samples, using the original lint-only calibration, are shown in Table 7 for both instruments by cotton variety. As observed for the micronaire and maturity results, when using the original lint calibrations, large differences were also observed between the reference fineness values and predicted NIR maturity values AVG and Δ (> ±15) for all samples. Based on the AVG and Δ results, a small but distinct variety impact for the three cotton varieties was observed for both instruments, primarily for SG105. Between conditions (LL, LSC, GSC), a distinct difference was observed between the lint and seed cotton cotton-types and between the lab and greenhouse environmental conditions for all three varieties for both instruments, with the MicroNIR 2200 exhibiting the smallest impacts.

New Combined Calibration for Micronaire, Maturity, and Fineness Using Three Conditions. The results above demonstrated that lint-only calibrations required improvements to minimize the observed cotton-type and environmental conditions impacts. The initial calibration was for laboratory measurements of commercially ginned lint only. It did not satisfactorily predict the fiber properties on the hand-picked individual cotton bolls, especially for the seed cotton and outside the laboratory samples. Thus, combining a subset of these diverse samples was required to obtain more robust calibrations that were capable of measuring both laboratory and outside the laboratory cottons, both seed cotton and lint from individual bolls. Samples from the LL, LSC, and GSC data sets were added to the original lint-only calibrations to develop new combined calibrations for micronaire, maturity, and fineness that include lint and seed cotton samples and lab and outside the lab (greenhouse) samples (n = 176). The results for the new and improved combined calibrations using both instruments and on LL, LSC, and GSC are shown in Table 8 for micronaire, Table 9 for maturity, and Table 10 for fineness.

The combined calibration NIR results for micronaire (Table 8) yielded improved method agreement compared to the original lint-only calibrations (Table 5). Significant improvements were observed in the AVG, Δ, and outlier results for all varieties. The Δ targeted limit (±0.3 micronaire units) was achieved for all varieties, cotton-types, and environmental conditions. The best results were obtained with the MicroNIR 2200 instrument with lower Δ and fewer outliers (< 30% outliers for all samples for LL, LSC, and GSC). The combined calibration significantly minimized and reduced the variety, cotton-type, and environmental condition impacts.

For maturity, the combined calibration (Table 9) yielded much improved method agreement compared to the results for the lint-only maturity calibration (Table 6), reducing significantly the difference (Δ) between the reference maturity values and the NIR maturity results (AVG) and with < 10% outliers (Table 9). The Δ targeted limit (±0.1 maturity units) was achieved for all varieties, cotton-types, and environmental conditions. The NIR results for the MicroNIR 2200 and Luminar 5030 instruments were similar for all varieties. The combined calibration significantly minimized and reduced the variety, cotton-type, and environmental condition impacts (Table 9).

For fineness, the combined calibration (Table 10) results yielded much improved method agreement compared to the lint-only calibration (Table 7), with significantly reduced differences (Δ) between the reference fineness values and the NIR fineness results (AVG), and with < 15% outliers for all but one condition for all varieties. The Δ targeted limit (±15 mtex) was achieved for all varieties, cotton-types, and environmental conditions (Table 10). The best results were obtained with the MicroNIR 2200 instrument, with fewer outliers (< 15% outliers for all samples). The combined calibration significantly minimized and reduced the variety, cotton-type, and environmental condition impacts on NIR measured fineness.

Table 8. Micronaire results on the combined calibration

GROUP		MIC	MicroNIR 2200				Luminar 5030			
			AVG	Δ	SDD	OUT(%)	AVG	Δ	SDD	OUT(%)
ALL	LL	5.29	5.28	-0.01	0.22	16	5.32	0.03	0.24	19
	LSC		5.31	0.02	0.29	27	5.24	-0.05	0.38	49
	GSC		5.35	0.05	0.29	24	5.12	-0.17	0.47	57
DP393	LL	5.17	5.17	0.00	0.22		5.10	-0.07	0.23	
	LSC		5.19	0.02	0.28		5.06	-0.11	0.38	
	GSC		5.36	0.19	0.27		4.93	-0.24	0.41	
FM958	LL	5.32	5.29	-0.03	0.14		5.40	0.08	0.20	
	LSC		5.27	-0.05	0.15		5.34	0.02	0.31	
	GSC		5.22	-0.10	0.17		5.21	-0.11	0.51	
SG105	LL	5.40	5.39	-0.01	0.29		5.47	0.07	0.27	
	LSC		5.48	0.07	0.40		5.34	-0.06	0.46	
	GSC		5.45	0.05	0.34		5.24	-0.16	0.53	

MIC = Micronaire; LL= Lab measured lint samples; LSC = Laboratory measured seed cotton samples; GSC = Greenhouse measured seed cotton samples

Table 9. Maturity results on the new combined calibration

GROUP		MR	MicroNIR 2200				Luminar 5030			
			AVG	Δ	SDD	OUT(%)	AVG	Δ	SDD	OUT(%)
ALL	LL	0.98	0.98	0.00	0.04	3	0.97	-0.01	0.05	3
	LSC		0.98	0.00	0.04	3	0.97	-0.01	0.05	5
	GSC		1.00	0.02	0.04	8	0.96	-0.02	0.06	5
DP393	LL	1.00	0.99	-0.01	0.03		0.96	-0.04	0.03	
	LSC		0.99	-0.01	0.04		1.00	-0.01	0.05	
	GSC		1.02	0.02	0.04		0.98	-0.02	0.06	
FM958	LL	0.96	0.97	0.01	0.05		0.99	0.03	0.05	
	LSC		0.98	0.02	0.04		0.96	0.01	0.06	
	GSC		1.00	0.04	0.05		0.95	-0.01	0.05	
SG105	LL	0.96	0.98	0.02	0.03		0.96	0.01	0.04	
	LSC		0.96	0.00	0.03		0.95	-0.01	0.06	
	GSC		0.97	0.01	0.05		0.96	0.01	0.07	

MR = Maturity; LL = Lab measured lint samples; LSC = Laboratory measured seed cotton samples; GSC = Greenhouse measured seed cotton samples

Table 10. Fineness results on the new combined calibration

GROUP		FN	MicroNIR 2200				Luminar 5030			
			AVG	Δ	SDD	OUT(%)	AVG	Δ	SDD	OUT(%)
ALL	LL	210.98	210.60	-0.38	8.57	5	210.68	-0.30	7.65	3
	LSC		210.30	-0.68	9.13	8	210.32	-0.66	11.15	11
	GSC		211.55	0.57	9.35	11	207.94	-3.04	15.12	35
DP393	LL	206.10	206.13	0.03	6.66		205.06	-1.04	5.43	
	LSC		207.43	1.33	7.92		206.41	0.31	10.13	
	GSC		212.29	6.19	6.74		204.87	-1.23	13.24	
FM958	LL	203.64	210.38	6.74	4.99		210.42	6.78	5.51	
	LSC		206.78	3.14	6.37		205.63	1.99	9.71	
	GSC		203.69	0.05	9.97		200.36	-3.28	16.45	
SG105	LL	223.81	215.66	-8.15	6.86		217.04	-6.77	5.44	
	LSC		216.94	-6.87	10.09		219.25	-4.56	13.26	
	GSC		218.62	-5.19	7.95		218.83	-4.98	16.67	

FN = Fineness; LL = Lab measured lint samples; LSC = Laboratory measured seed cotton samples; GSC = Greenhouse measured seed cotton samples

It is interesting to note that the NIR results for maturity have lower Δ, SDD, and far fewer outliers compared to the NIR results for micronaire and fineness. Micronaire is a combination of the fiber's maturity and fineness. For maturity, the NIR is measuring the cellulose content in the fiber, a chemical property; for fineness, the NIR is measuring the scattering and surface reflectance from the fiber, a physical property. NIR responds well to chemical property differences, but differences between pure physical properties can be challenging to determine (Burns, 1985). Thus, the variability in the NIR results for fineness can be a major contributor impacting the micronaire results, resulting in the observed variability observed for micronaire.

SUMMARY

Comparative evaluations were performed to determinate the potential of portable NIR instruments to accurately measure micronaire, maturity, and fineness simultaneously on seed cotton (greenhouse and laboratory measurements on individual cotton bolls) and on ginned lint from the seed cotton (laboratory measurements) using the portable Viavi MicroNIR 2200 and the Brimrose Luminar 5030 NIR instruments. The simultaneous NIR measurements on the fiber of three cotton varieties under three distinct measurement variables (measurement location, environmental conditions, type cotton) were fast (< 1 min. per sample), easy to perform, accurate, and required minimal sample preparation.

For the original lint-only calibration developed from a well-defined and diverse sample set, NIR results on the seed cotton and ginned lint samples often exhibited variety effects for both instruments. For micronaire and maturity, the MicroNIR 2200 exhibited a cotton-type impact (lint vs. seed cotton), and the Luminar 5030 exhibited an environmental condition impact (lab vs. greenhouse measurements). Samples of lint and seed cotton were added to the original lint-only sample set to develop new NIR calibrations for micronaire, maturity, and fineness. Distinct and significant improvements in method agreement (Δ) were obtained, and the significant reduction of number outliers of all samples using the optimized combined calibration data sets was encouraging. These positive results establish that including samples from different environments (laboratory and greenhouse) and fiber type conditions (lint and seed cotton) into ginned lint cotton-only calibration data sets results in stronger, more robust, and improved calibrations for micronaire, maturity, and fineness. In addition, the impact of variety on the NIR results was minimized. The accuracy of the prediction was improved with both instruments. The two portable NIR instruments, the MicroNIR 2200 and the Luminar 5030, yielded similar results, with the MicroNIR 2200 yielding superior results for micronaire and fineness. Due to the similarities in the NIR results with the combined calibrations, both instruments were shown to satisfactorily measure fiber micronaire, maturity, and fineness both inside and outside the laboratory for both ginned and seed cotton.

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DISCLAIMER

The use of a company or product name is solely for the purpose of providing specific information and does not imply approval or recommendation by the United States Department of Agriculture to the exclusion of others.

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