## COMPARISON OF NIR METHODS FOR MEASURING COTTON FIBER MATURITY AND FINENESS J. E. Rodgers C. A. Fortier X. Cui SRRC-ARS-USDA New Orleans, LA

### <u>Abstract</u>

Maturity and fineness are important cotton fiber properties, as they can impact the fiber's downstream processability and dye performance. Maturity and fineness are often indicated indirectly by the fiber's micronaire. Maturity and fineness can be measured directly, but most available methods are slow and/or require expensive laboratory-only equipment. Much interest has been shown in new direct measurements of fiber maturity and fineness that could be applicable for both laboratory and at-line/field use. A program was implemented to determine the feasibility of using NIR systems (analyzers and measurement methods) to monitor fiber maturity and fineness in the laboratory. Well-defined cotton sample sets were measured on multiple bench-top and portable NIR systems in a conditioned laboratory, and their results compared. NIR instrumental, sampling, and operational procedures and protocols were established. Very good spectral agreement was observed between the NIR units. NIR measurements took less than 3 minutes per sample, and no sample preparation was required. The accurate measurement of fiber maturity by both bench-top and portable NIR analyzers/systems was demonstrated, indicating a very promising potential for cotton field analyses of maturity by portable units. Acceptable fiber fineness results were observed for the bench-top unit only, and additional samples will be required for improved portable NIR unit measurements.

#### **Introduction**

Micronaire is an important classing and quality assessment parameter for cotton fiber. (USDA, 2005) Micronaire is a function of the fiber's maturity and fineness. Maturity is often referred to as the degree of development of the fiber's secondary wall, and fineness is the fiber's linear density (or diameter, cross-section area, perimeter). (Wakelyn et. al., 2007) Maturity and fineness can be measured directly, but most available methods are slow and/or require expensive laboratory-only equipment. Recently, the rapid and accurate direct measurement of fiber maturity and fineness has garnered much interest internationally. Recent advances in Near Infrared (NIR) technology offer the potential for rapid and accurate direct measurements of fiber maturity and fineness both in the laboratory and in remote locations (e.g., at-line, field).

Previous evaluations have demonstrated the capabilities of and established the universal nature of NIR techniques to rapidly and accurately measure fiber micronaire in the laboratory. (Montalvo and von Hoven, 2004; Rodgers et. al., 2010a; Rodgers et. al., 2010b) A preliminary evaluation also demonstrated the potential for NIR measurements of cotton micronaire in remote locations (at-line and in the field). (Rodgers et. al., 2010c) Since NIR techniques have been shown to perform well in the measurement of fiber micronaire, a program was implemented to determine the feasibility of using various NIR techniques to monitor fiber maturity and fineness in the laboratory.

# **Material and Methods**

A set of 104 well-defined, wide-range, diverse fiber samples were used for this comparative evaluation. (Hequet et. al., 2006) The maturity and fineness values from cross-sectional image analysis measurements were used as the reference values. The samples were split into two sampling sets—a 69 sample calibration set (used to calibrate each instrument) and a 35 sample validation set (used to determine the robustness of the calibrations). All measurements were performed in a conditioned laboratory at standard conditions ( $70\pm2^{\circ}F$ ,  $65\pm2\%$  relative humidity or RH)

The maturity and fineness results from three very different NIR analyzer systems were compared (Figure 1). The three NIR systems (instrumental and sampling specifics) were as follow:

- Bruker MPA (bench-top unit; FT-NIR measurement, 1100-2500 nm, fiber optic probe sampling system; Bruker Optics Inc., Billerica, MA)
- Bruker Lancir (portable unit, dispersive measurement, 1100-2200 nm, contact sampling system; Bruker Optics Inc., Billerica, MA)

• Brimrose 5030 (portable unit, Acousto-Optic Tunable Filter/AOTF measurement, contact sampling system; Brimrose Corp of America, Baltimore, MD)

Five measurements were made per sample. As noted above, each instrument was calibrated with 69 samples, and the robustness of the calibrations was validated with 35 samples. Vendor recommended operational procedures and protocols were used.

The comparative parameters for this evaluation included  $R^2$  and Standard Deviation of Differences residual analysis (SDD, the standard deviation of the differences between the reference and measured maturity/fineness value for each sample). The higher  $R^2$  and the lower SDD, the better the NIR instrument/system performed.



BRUKER MPA FT-NIR

BRUKER LANCIR



**BRIMROSE 5030** 



# **Results and Discussion**

A program was implemented to determine the feasibility of using various bench-top and portable NIR instruments and techniques to monitor cotton fiber maturity and fineness in the laboratory. Distinct absorbance spectral differences were observed (Figure 2 for Brimrose 5030 and maturity). The NIR measurement of maturity and fineness was relatively fast (< 3 minutes per sample). The measurement was easy to perform for each instrument, and no sample preparation was required.



Figure 2. NIR absorbance spectral differences, Brimrose 5030 (MR = 0.557, 0.832, 1.087).

Compared to the cross-sectional image analysis maturity values, the NIR measurements for maturity yielded overall very acceptable results, with moderate to high  $R^2s$  (0.75 to 0.92) and low SDDs (0.038 to 0.061), as shown in Table I. The best results were observed with the bench-top MPA unit. A distinct skew/bias was observed at higher MRs. This skew/bias has been attributed to a bias in the image analysis software used to calculate maturity. (Padmaraj et. al., 2011) Even with the error in the reference values, the rapid and accurate measurement of cotton fiber maturity by bench-top and portable NIR system was successfully demonstrated. These results for the portable systems indicate a very promising potential for cotton field measurements of fiber maturity to complement the current measurements of fiber micronaire in the cotton field.

Compared to the cross-sectional image analysis fineness values, the NIR measurements for fineness yielded overall very acceptable results for the bench-top MPA unit only, with a high  $R^2$  and low SDD (0.91 and 4.8 respectively). Only fair method agreement for fineness was observed for the portable Lancir and 5030 units, with  $R^2s \sim 0.60$  and SDDs over 10.0 (Table II). Additional fiber samples will be required to improve the robustness of the portable NIR measurements for fiber fineness.

ITEM	VALIDATION, MATURITY (n = 35)			
	BRUKER MPA	BRUKER LANCIR	BRIMROSE 5030	
AVG	0.844	0.845	0.858	
SD	0.100	0.100	0.094	
SDD	0.038	0.058	0.061	
$\mathbf{R}^2$	0.92	0.78	0.75	

Table I.	Comparison	results.	fiber	maturity

Table II.	Comparison	results.	fiber	fineness
	0011100110011	100001000		

ITEM	VALIDATION FINENESS $(n = 35)$			
	BRUKER MPA	BRUKER LANCIR	BRIMROSE 5030	
AVG	170.8	175.8	171.0	
SD	15.1	16.4	16.6	
SDD	4.8	10.4	11.0	
$\mathbf{R}^2$	0.91	0.63	0.59	

# **Summary**

A program was implemented to determine the feasibility of using NIR techniques to measure cotton fiber maturity and fineness in the laboratory, comparing both bench-top and portable NIR systems. The NIR simultaneous measurement of fiber maturity and fineness was rapid (analysis time of less than 3 minutes per sample), easy to perform, and no sample preparation was required. The accurate measurement of fiber maturity by both bench-top and portable NIR analyzers/systems was demonstrated, with best results for the Bruker MPA bench-top unit. For fiber fineness, only the bench-top unit yielded acceptable results. Additional samples will be required for improved fineness measurements on portable NIR analyzers/systems. For the portable units, the results clearly indicated the very promising potential for cotton field analyses of maturity.

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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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