COMPREHENSIVE INVESTIGATION OF NIR TECHNIQUE IN COTTON FIBER QUALITY ASSESSMENT

Yongliang Liu
Gary R. Gamble
Devron P. Thibodeaux
Cotton Quality Research Station, ARS, USDA
Clemson, SC

Abstract

Near infrared (NIR) spectroscopy, with an extension to UV and visible region, has been considerably applied for the quantitative measurements of key color and physical characteristics in cotton fibers. However, the results have been inconsistent, mostly due to the use of different spectral regions. This work examined and compared the NIR model performances built from various regions for the prediction of cotton color (Rd and +b), physical properties (micronaire, strength, and length indexes), and visible trash content. On the basis of results, improvement in reference determination and consideration of other spectroscopic approach were suggested.

Introduction

Various techniques, including optical, physical and chemical methods, have been developed to classify and grade cotton fibers. Following the introduction in 1960s, high volume instrumentation (HVI) (Gordon, 2007), together with other recently developed instrumental methods, has continued to be a viable tool for determining a number of effective cotton quality parameters. Although they can measure many different quality indexes and are used throughout the cotton industry, the procedures usually are destructive, time consuming, and prone to day-to-day and location-to-location variations. Considerable efforts have been made to address several concerns on these instrumental methods, for example, the reproducibility and repeatability of within and between HVI evaluations (Knowlton, 2002a & 2002b), the relationship between HVI color parameter and globally recognized CIELAB color system (Rodgers et al., 2008), and also the correlation of specific physical properties between two similar readings from different instruments (Thibodeaux et al., 2008).

Since instrumental methods measure the color and / or physical property information, it will be of interest to obtain independent and complementary information on cottons from other non-destructive spectral sensing approaches. Among them, near infrared (NIR) is desired due to the speed, ease of use, and potential on-line/off-line implementations (Burns and Ciurczak, 2001). It has been successfully applied for the quantification of fiber moisture and chemical component (Ghosh and Roy, 1988; Taylor and Godbey, 1994), for the prediction of color and physical attributes (Montalvo et al., 1994; Ramey, 1982; Rodgers et al., 2009; Thomasson and Shearer, 1995), and for the detection of foreign contaminants (e.g., trash) (Taylor, 1980; Thomasson and Shearer, 1995). Although these studies indicated that NIR technique is feasible and promising in the quality evaluations of cotton fibers, the results have been inconsistent. Also, color is one of the most important fiber quality indexes in cotton grading and classification, and few studies have been conducted by including the ultraviolet (UV) and visible (UV/visible) region.

The main objective of this study was to examine the potential of NIR technique, with an extension to UV/visible (220-750 nm) region, for the prediction of cotton color and physical attributes and also cotton trash component. The ultimate goal is to develop this technique for rapid, accurate, nondestructive, and routine determination of cotton fiber qualities in cotton fields, ginning sites, and classing offices.

Materials and Methods

Cottons, Reference Measurement, and UV/Visible Spectral Collection

A total of 123 cotton samples were collected from different portions of 21 cotton bales that consisted of eight cotton varieties and also from different portions of six cotton standards. This type of sampling might be reasonable, because cotton fibers from different locations of a bale (480 lb) have shown great variations in fiber properties (Van Dalfsen and Alberts, 1953). The color and physical readings were measured by an Uster HVI 900A system (Zellweger Uster, Inc., Knoxville, TN, USA).

From different cotton bales, 160 cotton wastes were collected at ARS's Cotton Quality Research Station's (Clemson, SC) processing and cleaning line that included such devices as Axi-Flo (coarse cleaner), GBRA (mixer), RN (coarse cleaner), opener motes (fine opener), and card waste (deduster). Non-lint visible trash content was then determined according to ASTM method D2812 with the use of Shirley Analyzer (Shirley Developments, Ltd., Stockport, UK). In addition, 5 cleaned cotton fibers from 5 types of cotton wastes were compiled into the data set and were assigned the visible trash content of 0%.

Reflectance spectra were recorded in the 220-2200 nm or 220-2500 nm region at 1 nm interval and with a NIR cup loaded with ca. 0.5 g sample by using an JASCO V-670 UV/visible/NIR spectrometer (JASCO, Eastern Shore, MD, USA). Before determining the references and collecting the spectra, all samples were well conditioned at a constant relative humidity of 65% and temperature of 72 ± 2 ºF.

PLS Models

All UV/visible/NIR spectra were imported into PLSplus/IQ package in Grams/AI (Version 7.01, Thermo Fisher Scientific, Waltham, MA) and were smoothed with a Savitzky-Golay function (polynomial = 2 and points =13), prior to model development. All samples were ordered with the sequence of acquisition. Every 3rd samples were used for model validation and the remaining for model calibration. To optimize the accuracy of models, the data were subjected to different combinations of both spectral regions (e.g., full and narrow) and spectral pretreatments (e.g., mean centering (MC), multiplicative scatter correction (MSC), standard normal variate (SNV), the first and second derivatives). Full (one-sample-out rotation) cross-validation method was used, and the number of optimal factors selected for the regression equation generally corresponded to the minimum of the predicted residual error sum of squares (PRESS). The saved regression equations were subsequently applied to the validation samples. Model accuracy and efficiency were assessed in the validation set on the basis of coefficient of determination (R^2), root mean square error of validation (RMSEV), and residual predictive deviation (RPD) (Williams, 2001).

Results and Discussion

UV/Visible/NIR Spectra of Cotton Fiber and Cotton Waste

Representative log (1/R) spectra of cotton fibers (Fig.1) reveals at least four intense and broad bands with one (< 600 nm) in the UV/visible region (220-750 nm) and three (1490, 1935, and 2105 nm) in the NIR region (750-2200 nm). In general, the UV/visible region contains the color information and represents a mixture of contributions from the pigmentation compounds present in cotton fibers. Whereas the origins of NIR bands differ from those in the UV/visible region, and they are mainly due to the (1st and 2nd) overtones and combinations of OH and CH stretching vibrations of cellulose, which comprises more than 94% of cotton fiber mass.

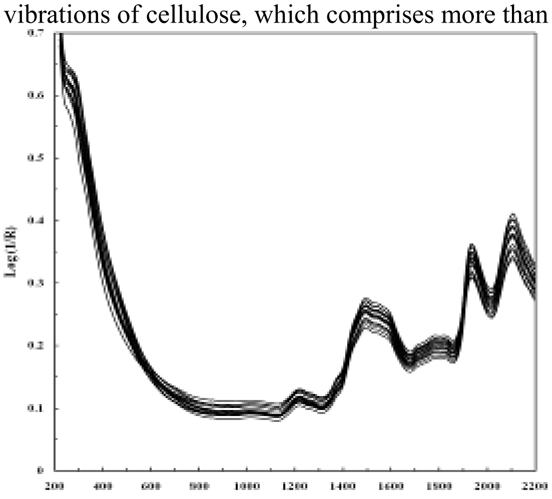

Fig.1. Typical log (1/R) spectra of cotton fibers.

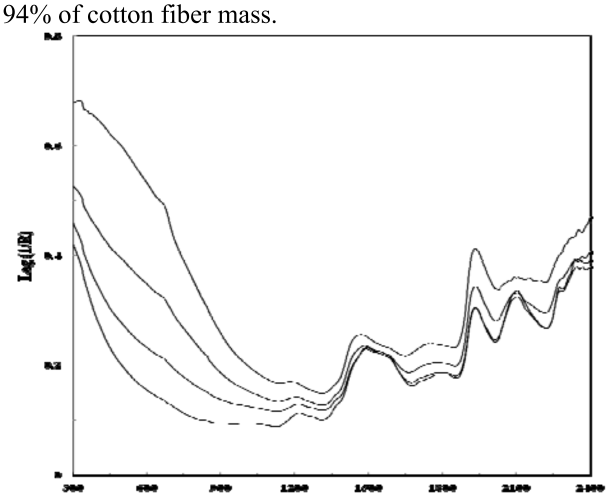

Fig.2. Spectra of trash (0, 9.8, 45.4 &100%, bottom to up).

Figure 2 shows the spectral feature of two cotton wastes with total trash readings of 45.4% and 9.8 %, together with those of pure visible trash (total trash = 100 %) and cleaned cotton fiber (total trash = 0 %). Distinctive spectral differences suggest high log (1/R) intensity in the UV/visible/short-wavelength (SW) NIR region (< 1200 nm) and apparent intensity reduction from 2020 to 2200 nm, with the increase of visible trash amount.

Univariate Correlation Coefficients

The univariate correlation coefficients between pairs of 8 separate quality indexes are shown in Table 1. In general, eight fiber qualities could be considered as 4 types, color (Rd and +b), fineness (as micronaire), strength, and lengths (as mean length and upper half mean length and associated uniformity and short fiber indexes were derived from the same HVI Fibrogram). Besides the positive and moderate correlations with mean length, uniformity index, strength, and micronaire, Rd correlated with +b negatively and significantly and also with short fiber index negatively and moderately. Meanwhile, +b showed positive and moderate correlation with only 1 (short fiber index) of 7 cotton attributes, indicating both of them are related to low fiber quality.

Table 1. Univariate correlation coefficients for 8 fiber qualities from HVI measurement.
(Absolute values > 0.50, 0.50-0.20, and < 0.20 were to have significant, moderate, and insignificant correlations).

	Rd	**+b**	**Mean length**	**Upper half mean length**	**Short fiber index**	**Uniformity index**	**strength**
+b	-0.59						
Mean length (ML)	0.24	-0.40					
Upper half ML	0.18	-0.39	0.98				
Short fiber index	-0.27	0.35	-0.86	-0.79			
Uniformity index	0.34	-0.24	0.63	0.47	-0.76		
Strength	0.45	-0.39	0.51	0.48	-0.48	0.43	
Micronaire	0.20	-0.10	-0.15	-0.29	-0.15	0.47	0.01

As expected, four length properties (mean length, upper-half mean length, short fiber index, and uniformity index) had stronger correlations with each other than with color, micronaire, and strength. Meanwhile, negative correlations existed between short fiber index and mean length, upper-half mean length, and uniformity index.

Moderate correlations were observed between strength and other fiber indexes except for micronaire, positively with Rd, mean length, upper-half mean length, and uniformity index but negatively with +b and short fiber index. Micronaire had positive and moderate correlations with Rd and uniformity index, and negative and moderate correlation with upper-half mean length. Also, it showed insignificant correlations with +b, mean length, short fiber index, and strength.

Reference Values

Table 2 summarizes the range, mean, and standard deviation (SD) of reference values. Their variations covered most of the variability in fiber properties (Montalvo et al., 1994; Thomasson and Shearer, 1995). The range, mean, and SD values for individual property were similar within calibration and validation sets, indicating that the selection of samples for each set was appropriate.

Table 2. Reference values for 9 fiber indexes (calibration = 110 and validation = 55 for visible trash only).

Cotton characteristics	**Calibration set (n =82)**		**Validation set (n = 41)**	
	Range	**Mean ± SD**	**Range**	**Mean ± SD**
Rd	72.97 - 84.80	78.08 ± 2.68	72.97 - 84.80	78.23 ± 2.65
+b	10.92 - 17.20	14.96 ± 1.57	10.92 - 17.20	14.93 ± 1.55
Mean length (inch)	0.692 - 0.964	0.853 ± 0.069	0.700 - 0.964	0.857 ± 0.066
Upper-half mean length (inch)	0.886 - 1.190	1.062 ± 0.077	0.902 - 1.190	1.067 ± 0.073
Short-fiber index (%)	9.10 - 14.10	11.77 ± 1.75	9.10 - 14.10	11.62 ± 1.72
Uniformity index (%)	76.80 - 83.10	80.28 ± 1.39	77.20 - 82.80	80.26 ± 1.38
Strength (gm/tex)	22.84 - 36.15	28.55 ± 3.08	24.11 - 36.39	29.12 ± 3.16
Micronaire (units)	2.51 - 5.38	4.01 ± 0.84	2.51 - 5.38	4.02 ± 0.83
Visible trash (%)	0 - 65.20	28.5 ± 19.8	0 - 60.20	27.9 ± 19.0

Calibration and Prediction Models

Partial least-squares (PLS) regression models for all properties were developed using the different combinations of full / narrow spectral regions and a variety of data pre-treatments. The statistics of optimal results in calibration and validation sets from three spectral regions are summarized in Table 3. In addition to the entire region, the log(1/R) spectra were analyzed subjectively in two narrow regions: 226-1100 and 1100-2194 or 1100-2494 nm. For each property in specific spectral region, the best model was determined by lower RMSEV and higher R^2 in the validation set, respectively.

Table 3 reveals that the best prediction models were obtained from the combinations of such spectral pretreatments as mean centering (MC), multiplicative scatter correction (MSC), standard normal variate (SNV), and the first (1^{st}) derivative. The use of 2^{nd} derivative, along with other data processing, yielded much poorer results for all properties (not shown). This observation is in good agreement with that reported by Montalvo et al. (1994). Comparison of the RMSEV and R^2 in validation set indicates that the models from the full (226-2194 or 226-2494 nm) region produced the optimal predictions for +b, short fiber index, uniformity index, and visible trash, the models representing the 226-1100 nm region yielded the best results for Rd, mean length, upper-half mean length, and strength, and the model in the 1100-2194 nm NIR region had the best performance for micronaire. Notably, optimal models for fiber properties resonated very well with the univariate correlations from HVI values alone in Table 1. For instance, micronaire, with the best model from the 1100-2194 nm NIR region, had much lower correlation with two color indexes than strength / lengths, which had the best models from regions including UV/visible absorptions.

Examination of RMSEC, RMSEV, and R^2 in Table 3 also suggests that micronaire could be predicted closely by two models, either from the 226-2194 nm full region or from the 1100-2194 nm NIR region. Together with other models, it indicated that all cotton properties, considered together, could be best assessed by the utilization of the entire UV/visible region. Meanwhile, the predictive models in Table 3 were similar to or better than those previously reported (Montalvo et al., 1994; Thomasson and Shearer, 1995), in which the “dissimilar” samples were removed from calibration and validation sets by cluster analysis or the optimum regression models were not validated by independent samples.

RPD, ratio of the standard deviation (SD) of reference value against RMSEV, is often used as a dimensionless gauge of the ability of a spectroscopic model to predict a property (Williams, 2001). An RPD value of greater than 2.5 indicates that the spectroscopic model might be suitable for screening programs, and a value of greater than 5.0 is potentially useful in quality control. From the scale of RPD in Table 3, the models for micronaire and +b as well as from Rd, mean length, upper-half mean length, and visible trash could be used for quality control and screening applications, respectively. With the RPD value not greater than 2.0, the strength, short fiber index, and uniformity index could not be modeled as effectively as other fiber indexes investigated.

Spectral Response and Fiber Quality Characteristics

Within 41validation samples in micronaire, mean length, and strength models, there were 1, 6, and 8 samples that had prediction error (or difference) greater than the permitted ranges of 0.30 units, 0.04 inches, and 3.00 gm/tex (USDA, 2005), respectively. Among these samples, none was observed simultaneously in the models for micronaire, mean length, and strength, and only one was presented commonly in the models for mean length and strength. It indicated that the prediction difference probably was not from the spectral measurement, instead, likely it resulted from the degree of precision and reliability in determining the reference value and collecting the appropriate spectra.

The above results suggest that micronaire and +b could be more easily and accurately predicted than other fiber indexes, with the most difficult prediction being that for strength. Micronaire is a function of wall thickness and perimeter, and is related to the relative proportion of the fiber’s cellulose component, while +b is indicative of fiber yellowness originating from organic chromophores and exhibits the characteristic bands in specific NIR or UV/visible regions. On the other hand, fiber strength, and also fiber lengths, might be better determined by the factors that can not be obtained by UV/visible/NIR spectral absorptions. In an earlier study, PC2 scores from principal component analysis (PCA) of FT-Raman spectra of various cotton fibers were found to have a link with the strength of the cotton fibers (Liu et al., 1998). Meanwhile, despite of obvious spectral intensity difference induced by visible trash content, the resultant trash model only can be used for screening purpose. Therefore, improvement on reference measurement and use of other sensing tools, such as IR, Raman, and imaging, should be examined in the future.

Table 3. Statistics in calibration and validation sets.
(Calibration no.=82 and validation no.=41; For visible trash, calibration no.=110 and validation no.=55).

Fiber index / Spectral region	**Spectral processing**	**Optimal factors**	**Calibration set R^2**	**Calibration set RMSEC**	**Validation set R^2**	**Validation set RMSEV**	**Validation set RPD**
Rd							
226 - 2194 nm	MC	8	0.90	0.86	0.82	1.13	2.3
226 - 1100 nm	MC+MSC+1st deri.	10	0.96	0.53	0.87	0.96	2.8
1100 - 2194 nm	MC	6	0.61	1.68	0.51	1.89	1.4
+b							
226 - 2194 nm	MC+MSC	7	0.96	0.30	0.96	0.31	5.0
226 - 1100 nm	MC	8	0.96	0.32	0.94	0.39	4.0
1100 - 2194 nm	MC	5	0.82	0.66	0.82	0.66	2.3
Mean length							
226 - 2194 nm	MC+MSC	7	0.82	0.029	0.78	0.031	2.1
226 - 1100 nm	MC+MSC+1st deri.	8	0.88	0.024	0.81	0.029	2.3
1100 - 2194 nm	MC	4	0.53	0.048	0.55	0.044	1.5
Upper-half mean length							
226 - 2194 nm	MC+MSC	7	0.84	0.031	0.78	0.034	2.1
226 - 1100 nm	MC	9	0.84	0.031	0.82	0.031	2.3
1100 - 2194 nm	MC+MSC	4	0.62	0.048	0.59	0.047	1.5
Short fiber index							
226 - 2194 nm	MC+MSC+1st deri.	4	0.82	0.75	0.75	0.87	2.0
226 - 1100 nm	MC+MSC	7	0.71	0.95	0.71	0.94	1.9
1100 - 2194 nm	MC+1st deri.	2	0.52	1.21	0.55	1.15	1.5
Uniformity index							
226 - 2194 nm	MC	9	0.78	0.66	0.76	0.69	2.0
226 - 1100 nm	MC	8	0.66	0.82	0.74	0.71	1.9
1100 - 2194 nm	MC	2	0.38	1.11	0.49	1.01	1.4
Strength							
226 - 2194 nm	MC+1st	4	0.74	1.59	0.55	2.25	1.4
226 - 1100 nm	MC	12	0.74	1.59	0.63	2.11	1.5
1100 - 2194 nm	MC	4	0.31	2.58	0.20	2.91	1.1
Micronaire							
226 - 2194 nm	MC	7	0.97	0.14	0.97	0.14	5.9
226 - 1100 nm	MC	12	0.96	0.17	0.91	0.25	3.3
1100 - 2194 nm	MC	4	0.97	0.15	0.98	0.13	6.4
Visible trash							
226 - 2494 nm	MC	5	0.86	7.32	0.90	6.42	3.0
226 - 1100 nm	MC+SNV	6	0.87	7.08	0.88	7.04	2.7
1100 - 2494 nm	MC+SNV	4	0.86	7.42	0.86	7.27	2.6

Summary

The results of the present study demonstrate the usefulness of HVI and UV/visible/NIR spectroscopy in the characterization and determination of cotton fiber qualities. Univariate correlation coefficients among 2 color and 6 physical attributes from HVI measurement indicated several significant / moderate correlations. For example, strength had moderate correlations with 2 color indexes and fiber lengths, and micronaire had much lower correlation with color than strength and lengths. PLS regression models from the spectra and HVI / Shirley Analyzer data were individually developed in three spectral regions. The best models for nearly all properties were obtained with the inclusion of UV/visible region and corresponded well with the univariate correlations from HVI data alone, indicating the importance of cotton color in the characterization of other cotton physical properties. Meanwhile, the results suggested that UV/visible/NIR models can be used to predict micronaire and +b for quality control applications, and to assess Rd, mean length, upper-half mean length, and visible trash content for screening programs. However, for the quantitative measurement of strength, more work is needed to reflect the spectral response and / or improve the reference method.

Acknowledgements

We are grateful to Ms. D. Sewell, N. Carroll, and M. Marris (ARS, Clemson) for their excellent technical support.

References

Burns, D.A. and E.W. Ciurczak Eds. 2001. *Handbook of Near-Infrared Analysis*. Marcel Dekker, New York.

Ghosh, S. and R. B. Roy. 1988. Quantitative near-infrared analysis of reducing sugar from the surface of cotton. J. Textile Inst. 79: 504-510.

Gordon, S. 2007. Cotton fiber quality. P. 68-100. *In* S. Gordon and Y.-L. Hsieh (eds.) Cotton: Science and Technology. Woodhead publishing limited, Cambridge, England.

Knowlton, J. L. 2002a. Development in cotton standards. *Proceedings of 2002 Beltwide Cotton Conferences.* Memphis, Tenn.: National Cotton Council of America.

Knowlton, J. L. 2002b. Module averaging the short fiber measurement. *Proceedings of 2002 Beltwide Cotton Conferences.* Memphis, Tenn.: National Cotton Council of America.

Liu, Y., S. Kokot, and T.J. Sambi. 1998. Vibrational spectroscopy investigation of Australian cotton cellulose fibers. Part 1. A Fourier transform Raman study. Analyst. 123:633-636.

Montalvo Jr., J.G., S.E. Buco, and H.H. Ramey Jr. 1994. Studies to measure cotton fiber length, strength, micronaire and color by vis/NIR reflectance spectroscopy. Part II: Principal components regression. J. Near Infrared Spectrosc. 2:185-198.

Ramey Jr., H.H. 1982. Estimating quality components of natural fibers by near-infrared reflectance. Textile Res. J. 52: 20-25.

Rodgers, J., D. Thibodeaux, X. Cui, V. Martin, M. Watson, and L. Knowlton. 2008. Instrumental and operational impacts on spectrometer color measurements. J. Cotton Sci. 12:287-297.

Rodgers, J., J. G. Montalvo Jr., D. Gayle, and T. von Hoven. 2009. Near infrared measurement of cotton fiber micronaire, maturity and finesses – a comparative investigation. Textile Res. J. 79 : xxx-xxx.

Taylor, R.A. 1980. Cotton trash and contamination measurements for quality classification using near infrared reflectance. *Proceedings of 1980 Beltwide Cotton Conferences.* Memphis, Tenn.: National Cotton Council of America.

Taylor, R.A., and L.C. Godbey. 1994. Evaluation of high speed methods to measure cotton moisture for HVI testing. *Proceedings of 1994 Beltwide Cotton Conferences.* Memphis, Tenn.: National Cotton Council of America.

Thibodeaux, D., H. Senter, J. L. Knowlton, D. McAlister, and X. Cui. 2008. A comparison of methods for measuring the short fiber content of cotton. J. Cotton Sci. 12:298-305.

Thomasson, J.A. and S. A. Shearer. 1995. Correlation of NIR data with cotton quality characteristics. Transactions of the ASAE, 38:1005-1010.

USDA, AMS. 2005. Available from: http://www.ams.usda.gov/AMSv1.0/getfile?dDocName=stelprdc5074569. Accessed November 4, 2009.

Van Dalfsen, J.W. and J. Alberts. 1953. Raw cotton: variations within bales and within shipments. Textile Res. J. 23: 121-122.

Williams, P.C. 2001. Implementation of near-infrared technology. P. 145-169. In P. Williams and K. Norris (eds.) Near-Infrared Technology in the Agriculture and Food Industries, 2nd ed. American association of cereal chemists, St. Paul, MN.