CORRELATION OF HVI VS. STELOMETER FIBER STRENGTH AND ITS APPLICATION Yongliang Liu Gary R. Gamble Devron P. Thibodeaux Cotton Quality Research Station, ARS, USDA, P.O. Box 792 Clemson, SC 29633

Abstract

Cotton fiber strength is an important quality characteristic that is directly related to the manufacturing of quality consumer goods. Currently, two types of instruments have been implemented to assess cotton fiber strength, namely, the automation oriented HVI and the laboratory based Stelometer. Each of them has unique merits and correlation between the two strength readings was reported to be relatively low. In this study, both HVI and Stelometer strength readings were corrected by respective micronaire values in both quotient (strength/micronaire) and product (strength*micronaire) forms, and the modified strength readings were found to have a much improved relationship. Also, the relationship might suggest a way to determine the suitable samples in creating the NIR model for predicting the cotton strength effectively.

Introduction

Cotton fiber strength is an important quality characteristic that is related to the manufacturing of quality goods for consumers. Near infrared (NIR) spectroscopy, a useful technique due to the speed, ease of use, and adaptability to on-line or off-line implementation, has been applied to perform the qualitative classification and quantitative prediction on a number of cotton quality indices, including strength property (Liu et al., 2010; Montalvo et al., 1994; Thomasson and Shearer, 1995). However, resultant strength models from various NIR regions were observed to lack the power for practical applications. The low performance of strength models originates directly from at least one of two factors, one is the NIR spectral absorptions without the strength information and another is inappropriate strength reference measurement. In these investigations (Liu et al., 2010; Montalvo et al., 1994; Thomasson and Shearer, 1995), strength readings from high volume instrumentation (HVI) systems were taken as references for NIR model development, because HVI is a automatic computer-controlled testing equipment, and with $1\sim 2$ minutes, it is possible to provide a number of important fiber characteristics simultaneously, such as micronaire, strength, and color. Meanwhile, it has been introduced by USDA's Agricultural Marketing Service (AMS) as a universal standard instrument to be used for global HVI standardization (Knowlton, 2002).

It has taken time to understand the HVI strength result, with the consideration that (1) the HVI strength measurement was less precise compared to the other HVI quality measurement and (2) the HVI strength readings differed greatly from the traditional laboratory testing method known as Stelometer (Timpa and Ramey, 1989). Major distinctions between HVI and Stelometer strength measurements are due to the differences in the principle of testing and the way of fiber preparation. During the HVI procedure, the fibers were randomly selected and automatically prepared for testing, and the fiber mass was determined by less direct methods of light absorption and resistance to air flow. While in the Stelometer analysis, the fibers were selected, combed and carefully prepared to align them in the clamps, and the mass of the broken fibers was determined by weighing the same specimen. Although both HVI and Stelometer strength readings have been adapted in numerous publications, none has attempted to unravel the relationship between the two.

The objectives of this study were: (1) to correlate the HVI strength with Stelometer strength, in which one strategy was to correct the strength readings by respective micronaire values, and (2) to examine the effect of modified HVI strength values on NIR model performance.

Materials and Methods

Cottons and JASCO UV/Visible/NIR Reflectance Measurement

The origin, HVI strength and micronaire, as well as UV/visible/NIR spectral collection of these cotton fibers were described in detail previously (Liu et al., 2010). In brief, 123 sub-samples were from different cotton bales. Their HVI strength and micronaire properties were measured by the standard HVI procedure at this laboratory.

UV/visible/NIR reflectance spectra were recorded over the 220 - 2200 nm wavelength range at 1 nm increment by loading ca 0.5 g of cotton fibers into a round NIR sample cell (1.0 cm-depth and 5.1 cm-diameter) and using a JASCO V-670 UV/visible/NIR spectrometer (JASCO Inc., Eastern Shore, MD).

Cottons and FOSS Visible/NIR Reflectance Measurement

A total of 168 cotton samples and associated HVI quality readings were provided by Dr. James Knowlton at AMS's Cotton Classing office in Memphis, TN. These 2009 cottons represented a diverse distribution in cotton variety and growing locations. After conditioning for 48 hrs, visible/NIR spectra were acquired on a FOSS XDS rapid content analyzer (FOSS NIRSystems Inc., Laurel, MD). Approximately 10 g of cotton fibers was pressed into a FOSS coarse granular cell, which is rectangular with internal dimensions of 3.8 cm-wide x15.2 cm-long x 4.8 cm-depth. To keep a good contact between the cotton fibers and optical window, 750 g of extra weight was loaded on the top of fiber samples consistently throughout the entire experiment. A background was recorded with a built-in internal reference before scanning the samples. The log (1/Reflectance) readings were acquired over the 400 - 2500 nm wavelength range at 0.5 nm interval and 32 scans. Three spectra were collected for each of cotton samples by repacking and then mean spectrum was available for model development.

International Cottons and HVI Qualities

Additional 17 cotton samples from 5 foreign countries of China, South Africa, Pakistan, Zambia, and Ivory Coast were generously donated by a third party. Their HVI micronaire and strength properties were measured by the same HVI system as those cottons scanned by JASCO spectrometer.

Stelometer Strength Determination

Part of the cotton samples scanned by JASCO and FOSS spectrometers and all international cottons were chosen for Stelometer strength measurement. A Stelometer flat bundle tester was used to perform the measurement according to ASTM D-1445-90 (ASTM, 1995). Stelometer strength value of individual cotton sample was obtained as an average of six bundle breaks by two experienced operators. It took 10 minutes to perform three tests on one sample for each operator.

Partial Least Squares Models

All 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 calibration / validation model development. For JASCO spectral model (Liu et al., 2010), the samples were ordered with the sequence of spectral acquisition (2001 crop year samples, then international cotton calibration standards), and also were random within each sample set. Eighty-two of 123 spectra were used for calibration equation development, and the remaining 41 (every 3rd sample) spectra were used for model validation. For the FOSS spectral model, on the order of the smallest to largest in HVI strength readings, 112 spectra were selected for calibration equation development and the remaining 56 (every 3rd sample) spectra were used for model validation. To optimize the accuracy of prediction models, the spectra were subjected to different spectral pretreatments (e.g., mean centering (MC), multiplicative scatter correction (MSC), standard normal variate (SNV), the first and second derivatives) in the entire spectral region. 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). Model accuracy were assessed in validation set on the basis of coefficient of determination (r^2), root mean square error of validation (RMSEP), and residual predictive deviation (RPD) (Williams, 2007).

Results and Discussion

HVI Strength vs. Stelometer Strength

Figure 1 shows the plot of Stelometer strength values against HVI strengths for a total of 99 cotton fibers from diverse sources, with 55 from 2009 crop year and grown in U.S., 21 from 2001 crop year and grown in U.S., 17 from 2009 crop year and grown outside the U.S., and 6 from standard cottons that were used to calibrate the HVI systems around the world. As anticipated, Pearson correlation (r) between Stelometer strength and HVI strength was relatively low (r = 0.75), but still suggested some degree of correlation between the two testing devices.



Figure 1. HVI vs. Stelometer strength. Figure 2. Micronaire corrected HVI vs. Stelometer strength.

Stelometer strength is calculated using the following equation:

STESTR = (Kp x 15) / Wt ------(1)

Where STESTR stands for Stelometer strength, Kp is the breaking force, and Wt is the weight of the sample. As Wt represents a total of cotton mass, it has contributions simply from two components, one is celluloses fraction related with strength information and another is non-cellulose species unrelated with strength. Wt should be the amount of cellulose in total mass and might be estimated by correcting the total mass with the cellulose percentage or cotton maturity. Since there are not fast and reliable measurements to determine the cellulose percentage and cotton maturity (Hequet et al., 2006), we might utilize the micronaire value because it can be predicted by NIR models with a relatively high degree of success (Liu et al., 2010; Montalvo et al., 1994; Thomasson and Shearer, 1995). Hence, Eq. (1) could be rewritten as

$$STESTR_{mic} = (Kp \times 15) / (Wt \times MIC) = STESTR / MIC -----(2)$$

In which $STESTR_{mic}$ represents the micronaire corrected Stelometer strength value and MIC is micronaire value determined by HVI measurement. Similarly, HVI strength (HVISTR) could be represented by the quotient form in Eq. (3)

$$HVISTR_{mic} = HVISTR / MIC ------(3)$$

When plotting micronaire corrected Stelometer strength against HVI strength for the same samples as those in Figure 1, a nearly linear relationship with r of 0.96 was exhibited (Figure 2). The improvement of r from 0.75 to 0.96 suggests the consistency between two types of strength testing instruments, and also emphasizes the adaptability of the automated HVI system for practical fiber quality assessment. In addition to variations of cottons in crop year, variety, and growing countries, HVI strength and micronaire values were determined by at least two HVI systems at different locations within one year period, and Stelometer strength was conducted at one location within the past 8 months. Notably, Figure 2 might provide a clue to obtain the appropriate cotton strength references for model development, because the outlier samples could have great differences in strength readings between two independent measurements.

To verify the correlation in Figure 2, two completely different sample sets were applied. First set was from the 2001 crop year and the r was 0.99 on the basis of 21 cotton bales, in which HVI strength, Stelometer strength, and HVI micronaire were measured in 2002. Second set was from a published article (Brushwood, 2003) and the r for the cottons from 5 U.S. growing areas was 0.94.

As a different approach, one might consider the product result by multiplying the strength with HVI micronaire values as shown in Eqs. (4) and (5);

STESTR*MIC = (Kp x 15) / Wt x MIC = STESTR x MIC ------(4)HVISTR*MIC = HVISTR x MIC ------(5)

Pearson correlation between STESTR*MIC and HVISTR*MIC was 0.95 for the identical samples to Figure 1 and 2. It seems that the product presentation of strength and micronaire is nearly as effective as that of quotient form.

NIR Spectral Response and Cotton Fiber Strength Indices

NIR technique, in which the absorption bands arise from the combination and overtones of the fundamental IR bands of cotton cellulose, could be an independent tool for the assessment of three cotton fiber strength indices (HVISTR, HVISTR*, and HVISTR_{mic}). Representative log (1/R) spectra of 2 cotton fibers in different crop years from 2 NIR instruments are shown in Figure 3. There are at least six intense and broad bands with one (< 700 nm) in the UV/visible region (220-750 nm) and five (1490, 1935, 2105, 2270, and 2320 nm) in the NIR region (750-2500 nm). In general, the UV/visible region of 220-750 nm contains the color information and represents a mixture of contributions from the pigmentation compounds present in cotton fibers, whereas the NIR bands are mainly due to the (1st and 2nd) overtones and combinations of OH and CH stretching vibrations of cotton cellulose (> 90% in total mass).



Figure 3. Typical UV/visible/NIR log(1/R) spectra of cotton fibers from FOSS (solid line) and JASCO (dashed line).

Partial least squares (PLS) regression models on 3 HVI strength indices were developed on 2 sample sets from two NIR instruments, respectively. Table 1 compares the statistics of optimal results in calibration and validation sets. NIR spectra of cotton samples used for Stelometer strength measurement were not collected, since the Stelometer samples were too small (0.4 mg x 6 replicates = 0.0024g) to be loaded into the NIR cup and also sample numbers were limited in this study. From either JASCO or FOSS spectral model, R^2 in calibration set and r^2 in validation set were much improved with the use of HVISTR*MIC and HVISTR_{mic} as references compared to those with HVISTR as a reference. Because of different ranges for the three strength indices, it is rather difficult to compare RMSEC or RMSEP directly for model performance.

RPD, the ratio of SD of reference value to RMSEP in the validation set, is often used as a dimensionless gauge of the ability of a spectroscopic model to predict a given property (Williams, 2007). This parameter was calculated and

is included in Table 1. An RPD value of greater than 3.0 indicates the acceptability of the model for quantitative prediction, a value of greater than 2.5 and less than 3.0 suggests the suitability of the model for screening program, and a value of 1.0 or less means the lack of modeling power (Liu et al., 2010). Hence, modeling power of 3 strength indices was in the increasing order of HVISTR, HVISTR*MIC, and HVISTR_{mic}, which is independent of both sample sets and NIR instruments. Noticeably, the use of HVISTR_{mic} yielded a better result than that of HVISTR*MIC, and the HVISTR_{mic} model from JASCO could be acceptable for quantitative prediction (RPD > 3.0). Comparative scatter plots of both HVISTR and HVISTR_{mic} strength indices vs. the JASCO NIR model predicted ones in validation set are shown in Figure 4. This suggests how well the NIR model predictions agree with the references from a separated measurement. With the quotient form (Eq. 3), the regression line is nearly along the 45-degree direction.

Table 1. Statistics in calibration and validation sets for 3 cotton fiber strength indices ^a							
Strength index	Calibration set ^b		Validation set ^b				
	\mathbf{R}^2	RMSEC	r^2	RMSEP	RPD	Mean	SD
From JASCO ^c							
HVISTR	0.74	1.57	0.62	2.02	1.6	29.12	3.16
HVISTR*MIC	0.95	6.36	0.88	9.61	2.9	116.96	27.85
HVISTR _{mic}	0.94	0.47	0.94	0.47	3.9	7.59	1.83
From FOSS ^d							
HVISTR	0.32	1.36	0.42	1.12	1.3	28.97	1.46
HVISTR*MIC	0.76	7.12	0.76	7.01	2.0	128.25	14.09
HVISTR _{mic}	0.76	0.47	0.87	0.35	2.7	6.63	0.95

^a Mean centering (MC) pretreatment for all; 9 and 6 optimal factors for JASCO and FOSS models.

^b RMSEC, root mean square error of calibration; RMSEP, root mean square error of validation; RPD, ratio of SD to RMSEP; SD, standard deviation.

^c 82 samples in calibration set and 41 in validation set.

^d 112 samples in calibration set and 56 in validation set.



Figure 4. Correlation plot of HVI measured vs. NIR predicted from JASCO, taking HVISTR_{mic} value as a reference (\bullet and solid regression line) or HVISTR reading as a reference (\circ and dashed regression line). The HVISTR readings were divided by 3.5 to fit the scales in both axes for direct comparison.

Different response to 3 strength references between JASCO and FOSS model might result from several factors, such as variations in validation and calibration samples and also spectral regions. The most likely one is the distribution of range and standard deviation (SD) values of the reference in two sets. To examine this, the ratio of SD to mean

(SD/Mean) was estimated. The SD/Mean values of validation samples in JASCO models were 0.11, 0.24, and 0.24 for HVISTR, HVISTR*MIC, and HVISTR_{mic}, while they were 0.05, 0.11, and 0.14 in FOSS models, respectively. The greater SD/Mean value is desired, as it reflects a larger SD, and, in turn, could yield a higher RPD. Therefore, less variation of strength references could be responsible for relatively lower modeling in FOSS than that in JACSO.

In addition, the r between HVI +b (yellowness) modified HVI and Stelometer strength was 0.98 for the identical samples to Figure 1 and 2, but the FOSS NIR model was much poor RPD (\sim 1.7). Hence, this validates the modification of strength by micronaire and suggests a natural relationship between micronaire and strength property.

<u>Summary</u>

Despite relatively low correlation between 2 cotton strength readings directly from the automated HVI and the laboratory based Stelometer device, the present study demonstrates the consistency of cotton fiber strength between two methods if the strength readings were modified by cotton micronaire. The corrected HVI strength indices were found to have better correlations with NIR spectra than uncorrected strength values, through PLS regression. The use of the quotient form resulted in a more improved model performance than that of the product form. This suggests the potential of NIR technique in the prediction of cotton fiber strength for the purpose of quality control. Also, relationship on cotton strengths from two independent methods (HVI and Stelometer) might suggest a way to determine the suitable samples in creating the NIR model for predicting the cotton strength effectively.

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