

Preliminary Study of Determining Trash Components in Lint Cottons by Near Infrared Spectroscopy Technique

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Abstract

The transfer of Near Infrared (NIR) calibration models for the determination of total trash, leaf trash and non-leaf trash components in cotton fibers was conducted between two sets of samples. These samples to be analyzed are inhomogeneous in a bulky state whereas the samples used as calibrations were homogeneous in a ground state. The efficacy of the model transfer was evaluated based on instrumental leaf grade readings of "as is" diverse samples, because current-in-use trash tests cannot generate the trash amount for individual trash components. Results indicated that the predictions from the direct model transfer were unreliable, but they might be acceptable after the correction or conversion of original predictions with standard samples.

Keywords: Cotton total trash; Leaf trash; Non-leaf trash; Near infrared spectroscopy; NIR; Model transfer

Introduction

More automatic machines have been used to pick cotton fibers in the U.S. and other countries, mostly due to the economic factor and the speed [1]. Commonly, the harvested cottons contain some degree of cotton plant-related contaminants and other irregular foreign matter [2,3]. During subsequent ginning and cleaning practices, great efforts have been made to remove these undesired contaminants as much as possible [4]. Excessive processing may lower some end-use qualities of lint fibers (that were acquired during the ginning process to remove the cotton seed), such as the fiber length due to breakage; thus, assessment of cotton trash is important in optimizing the cleaning treatment too.

Trash in commercial cotton bales has been one of several concerns in cotton industry, as it discounts the value of cotton, requires more cleaning, and influences the quality of yarn and fabric. Over the years, the Shirley analyzer (SA) [5], high volume instrument (HVITM) [6], advanced fiber information system (AFIS) [7], micro dust and trash analyzer (MDTA) [8], and FibroLab [9] have been developed to determine cotton trash content in the U.S. and other countries. Generally, these methods only produce the amount of total trash, instead of the content for such individual or targeted cotton plant trash components as leaves (leaf and bract), seed coats, hulls, and stems. In large part, this limitation arises from the instrumental systems that analyze various trashes simultaneously in commercial-ready cotton bales; in other words, these trash testing methods cannot measure the amount of a specific trash component and identify the type of trash.

To evaluate the possibility of a rapid and low-cost technique that can be used, away from the laboratory, in places such as ginning sites, near infrared (NIR) spectroscopy has been attempted for the

quantitative prediction of trash contents represented as HVITM [10] and SA index [11,12], and also individual trash components [13]. In this latest study [13], a set of mixtures with known amounts of both clean lint fibers and individual trash components (leaves, seed coats, hulls, stems, and sand/soil) was prepared physically and then the visible/NIR spectral response was related to corresponding trash contents. The observation revealed the potential of visible/NIR technique in the precise and quantitative determination of total trash, leaf trash, and non-leaf trash components, but it also indicated the difficulty in the prediction of non-leaf trash components such as stem, hull, seed coat, and sand/soil. As a different approach, Fortier et al. [14] reported NIR spectral feature of individual or pure plant parts and further for their identifications.

There are considerable studies to explore the likelihood of predictive model transfer between instruments of the same or different types [15,16]. Even though instruments are the same, they do not generate exactly the same wavelength (horizontal axis) or absorbance (vertical axis) response when measuring the identical sample, due to both the aging/changing parts of instruments and the variations of environmental conditions. To maintain the efficiency of transferred calibration models, there are a number of approaches, including more calibration and standard validation samples [15], and also better spectral pretreatments and wavelength selection methods [16,17]. In these earlier studies, concentration or content of targeted compounds were commonly used for reference laboratory values for NIR calibration development. As a comparison, the true reference values of individual trash components in regular cottons were extremely difficult to acquire, mostly due to either current-in-use trash protocols cannot provide such information or it is labor intensive to amass different types and sizes of trash manually. In this study, lint cotton samples with various instrumental leaf grades were used, since the instrumental leaf grade was determined by an equation that utilizes HVITM trash readings of percent area and particle count on a sample's surface [18] and so that the magnitude of instrument leaf grade should

be proportional to trash level in a sample; unfortunately, present HVITM leaf grade measurement cannot provide the trash type, such as leaf trash and non leaf trash. The accumulated knowledge could be of value as a rapid analytical tool to cotton breeders for cotton variety enhancement and also to cotton ginning engineers for trash-removal cleaning device improvement. The main objective of this study was to investigate the feasibility of NIR spectroscopy in the non-destructive and rapid prediction of total trash, leaf trash, and non-leaf trash components in regular bulky cottons, by transferring the NIR models built from grounded mixtures of lint fiber and trash.

Materials and Methods

Grounded clean fibers, cotton trashes and their mixtures

The details on the collection of clean lint fibers and five types of trashes, and the subsequent preparation of their mixtures were described previously [13]. Briefly, clean lint fibers were obtained from routine SA (Shirley Developments, Ltd., Stockport, UK) processing of different lint cotton varieties, whereas each of the five cotton trash components, namely leaves (including bracts), seed coats, hulls, stems, and sand/soil, was collected either from the trash remains (or wastes) of the SA processing of commercial cottons or from the unginning (or seed) cottons manually. Both clean fibers and five trashes were grounded in a Wiley mill and passed through a 20-mesh screen with a sieve hole size of 0.841 mm. Then, 100 mixtures were prepared subjectively in the way of as homogeneous as possible by re-passing the screen a second time. Each mixture weighted 5.0 g in total and consisted of cut fibers and five trashes at varying concentration in percentage (%).

Bulky cotton fibers and instrumental leaf grade reading

Three hundred lint cotton samples from the 2010 crop-year, grown in the U.S. and with instrumental leaf grade assignment of 1 to 6 were utilized. They represented the diversities in Upland cotton varieties, growth locations, and ginning practices within the U.S. These bulky or uncut fibers were measured under a standard conditioning procedure of $65 \pm 2\%$ relative humidity and $21 \pm 2^\circ\text{C}$ temperatures.

Visible/NIR reflectance acquisition

A Foss XDS rapid content analyzer (Foss NIRSystems Inc., Laurel, MD) was used to acquire visible/NIR reflectance spectra. Approximately 1.3 g of individual grounded mixture was loaded into a round sample cell with internal dimension of 1 cm-deep x 3.8 cm-diameter. For regular bulky cottons, about 10 g of fibers was pressed into a Foss coarse granular cell with internal dimension of 3.8 cm-wide x 15.2 cm-long x 4.8 cm-deep, and a 750 g of extra weight was loaded on the top of fiber samples consistently to keep a good contact between the cotton sample and the optical window. In either module, $\log(1/\text{Reflectance})$ or $\log(1/R)$ readings were obtained in the 400 - 2500 nm visible/NIR range at 0.5 nm intervals. Three spectra, 32 scans per spectrum, were obtained for each sample by repacking, and their mean spectrum was utilized in the analysis.

Actually, the instrumental's optical window of scanning the samples is less than 2.5 cm in diameter. Unlike the sample was in a stationary module when using a round sampling cell, the sample in granular cell was moved across the optical window and scanned at 8 locations. This led to eight times more scanning surface area using a granular cell than

a round cell. Under the experimental setting, it took about 1 min to scan one sample.

Partial least squares (PLS) models from grounded samples and transfer to bulky fibers

Two spectral datasets were imported into PLS plus/IQ package in Grams/AI (Version 7.01, Thermo Scientific, Waltham, MA) for PLS regression model development. For individual trash components, 33 (every 3rd sample on the order of the smallest to largest in trash content) spectra were chosen to validate the model that was built from the remaining 67 spectra. In order to compare the prediction models, different combinations of both spectral ranges (e.g., full and narrow regions) and spectral pretreatments (e.g., mean centering (MC), multiplicative scatter correction (MSC), and the first and second derivatives) were attempted. During the protocol, leaving one-sample-out cross-validation method was used, and the suggested optimal factor that generally corresponded to the minimum of the predicted residual error sum of squares (PRESS) was taken. The performance of models was evaluated in the validation set by the parameters of the coefficient of determination (r^2), Root Mean Square Error of Validation (RMSEV), and Residual Predictive Deviation (RPD) [19]. Next, the models developed from the grounded mixtures were applied to the spectra of bulky samples with various leaf grade assignments.

Results and Discussion

Development of PLS models from grounded samples

In an earlier study [13], combinations of full / narrow spectral regions and different spectral pre-processing were utilized to optimize the PLS models for the constituents in grounded samples. Statistics in calibration and validation sets from four spectral regions (405 - 2495 nm, 405-1095 nm, 1105-2495 nm, and 900-1700 nm) were cited in Table 1 for the comparison. In general, the models from a narrow 900-1700 nm region showed the potential of NIR technique for the precise and quantitative determination of total trash and non-leaf trash components, due to their great RPDs. The term "RPD" has been used to evaluate the ability of a spectroscopic model in predicting a property [19], and a value of greater than 3.0 suggests the acceptability of the model for a quantitative implementation.

Despite the fact that leaf trash could be better predicted by the inclusion of 405-750 nm visible region ($\text{RPD} > 3.5$), these is a concern that the natural color of cotton fibers may affect the leaf trash model. This visible range reflects the color information and represents the contributions from the pigmentation compounds present in natural fibers, for example, flavonoids and / or degraded products between a reducing sugar and an amino acid [20,21]. Figure 1 compares normalized spectral response among four clean standard fibers that were used to calibrate HVITM micronaire measurement. Their values of micronaire (determined by both fiber maturity and fineness) were 5.34, 4.91, 4.00, and 2.52, respectively. Larger spectral intensity variation in the 405-750 nm regions than in other region originates from the subtle difference in HVITM color +b (yellowness) readings ranging from 15.4 to 17.0 among these samples. Hence, natural color occurrence in cotton fibers could hinder the PLS model transfer efficiency and thus the visible region of 400-750 nm was excluded in this preliminary study.

Chlorophyll and its degradation derivatives in cotton plant parts significantly contribute to the bands in this visible region, as indicated

by the intense band below 750 nm in the spectra of both leaf and non-leaf trash (Figure 2). Relative to that of clean cotton fibers, the spectra of leaf and non-leaf trash exhibited the larger log (1/R) readings in the 400-1000 nm region and the weaker intensity in the 2020-2200 nm regions. Probably, more chlorophyll (represented by the 675 nm band) and its degradation derivatives in leaf and non-leaf trashes than in cotton fiber are responsible for intense bands in the 400-1000 nm region.

Application of PLS models to bulky cotton fibers

Uncorrected predictions

Applying the PLS models built from the grounded samples to bulky cotton fibers, unreliable predictions of targeted constituents were within the line of expectation. For example, predicted total trash and non-leaf trash in cottons with instrumental leaf grade 1 to 6 were negative values

(Figure 3), implying the models could not be applied to unknown samples directly. Nevertheless, as higher leaf grade indicates relative greater trash amount in a sample, it is reasonable to observe an increase of total trash, leaf-trash and non-leaf trash with elevating instrumental leaf grade.

Corrected predictions

Because of significant difference in sampling grounded or bulky fibers between two cells, resultant spectral difference was observed (Figure 4), in which the round sample cell generate slightly high ranges in absorbance than the granular sample cell. Probably, the sample surface on the round sample cell is not similar to that on the granular sample cell due to differing state of samples that were used for the calibrations and the predictions. Therefore, the predicted values in Figure 3 should be corrected by using the standard or known samples.

Component	Range	Optimal factor	Calibration Set		Validation Set		
			R ²	RMSEC (b)	r ²	RMSEV (b)	RPD (c)
Total trash	405-2495 nm	6	0.93	0.76	0.89	0.88	2.9
	405-1095 nm	6	0.93	0.75	0.92	0.75	3.4
	1105-2495 nm	6	0.93	0.76	0.93	0.75	3.4
	900-1700 nm	6	0.94	0.69	0.92	0.72	3.6
Leaf trash	405-2495 nm	4	0.94	0.26	0.92	0.28	3.6
	405-1095 nm	5	0.95	0.23	0.94	0.26	3.9
	1105-2495 nm	6	0.87	0.38	0.84	0.41	2.5
	900-1700 nm	8	0.89	0.35	0.89	0.35	2.9
Non-leaf trash	405-2495 nm	7	0.93	0.67	0.92	0.66	3.2
	405-1095 nm	6	0.91	0.73	0.9	0.68	3.1
	1105-2495 nm	7	0.95	0.54	0.91	0.69	3
	900-1700 nm	8	0.95	0.54	0.92	0.60	3.5

Table 1: Statistics in calibration and validation sets [13(a)]

- a. Spectral pre-treatment with mean entering (MC) and a Savitzky-Golay first derivative function of two degrees and thirteen points for all models.
- b. Root mean square error of calibration (RMSEC) and validation (RMSEV).
- c. RPD = SD / RMSEV.

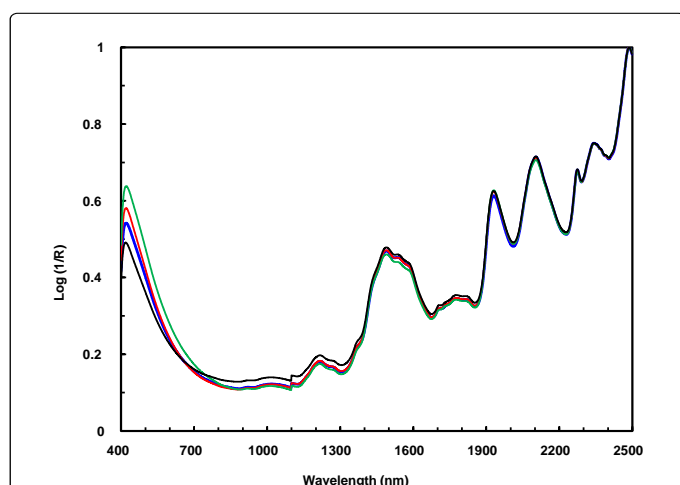


Figure 1: Comparison of normalized visible/NIR log (1/R) spectra of international standard calibration cotton fibers.

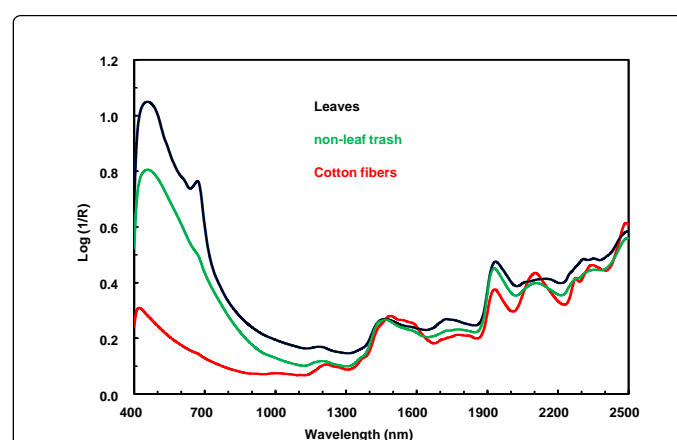


Figure 2: Typical visible/NIR log (1/R) spectra of cut clean "cotton fibers", leaf trash, and non-leaf trash that included stem, seed coat and hull trash.

Additional factors such as sample packing intensity and instrumental fluctuation can impede the model transfer.

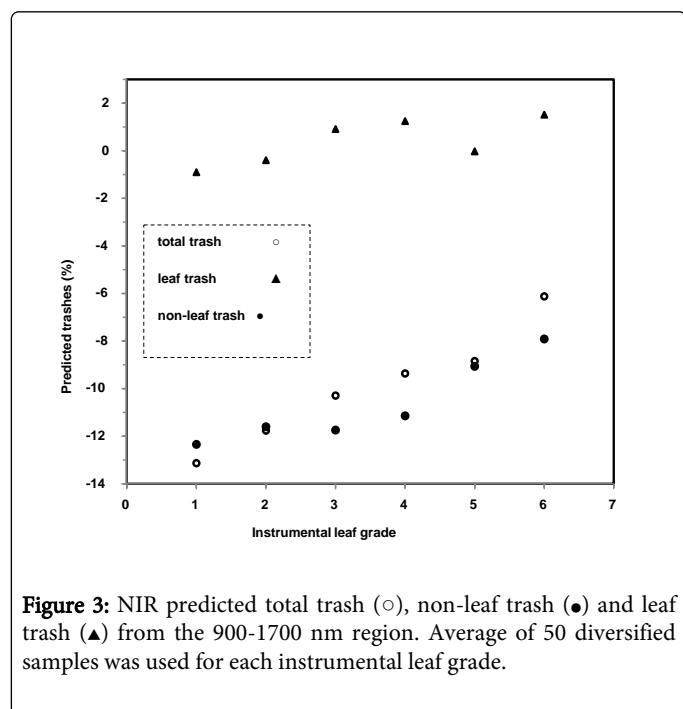


Figure 3: NIR predicted total trash (○), non-leaf trash (●) and leaf trash (▲) from the 900-1700 nm region. Average of 50 diversified samples was used for each instrumental leaf grade.

In this preliminary study, both one HVITM micronaire standard fiber (micronaire value = 5.34; color +b = 17.0) and pure trash mixture were utilized to correct the predictions in Figure 3. Undoubtedly, the selection of this specific calibration cotton is subject to more discussion and possible impacts of micronaire value and color on model transfer need further investigation. Figure 5 shows the corrected NIR predictions of total trash, non-leaf trash and leaf trash for different instrumental leaf grade samples. These conversions were simply calculated from the following general equation,

$$\text{Trash (\%)} = 100 \cdot (P_s - P_c) / (P_t - P_c)$$

where Trash (%) stands for the corrected content of total trash, or non-leaf trash, or leaf-trash

Through NIR model transfer, P_s , P_c , and P_t are the predicted values on bulky samples, uncut clean cotton fibers, and uncut trash materials from NIR models based on grounded mixtures. Clearly, P_c and P_t change with total trash, non-leaf trash, and leaf-trash variable. In this approach, the clean cotton fiber and trash components are considered to have the trash values of 0.0 and 100%, respectively.

The results in Figure 5 are promising, but undoubtedly it still has the space to improve the predictions in the future. One ultimate question is how to verify the results in Figure 5, in which the validation of model transferability was based on the instrumental leaf grade instead of measured trash contents. As stated earlier [13], trash determination tools can be categorized into two main groups: (1) gravimetric methods that assess the trash content by weight (g) or percentage (%) and (2) geometric method that estimate the trash content by trash particle count and percentage area on a sample's surface. Because of the heterogeneous distribution of trash type, density, and particle size, it is a challenge to link the trash amount from either type of two trash measurements with NIR spectra at satisfactory level, as revealed by relatively low NIR model performance

for referenced SA and HVITM trash indices [11,12]. As an alternative to overcome the challenge in NIR prediction of trash contents, seven-class SIMCA/PCA (soft independent modeling of class analogy / principal component analysis) classification models in different visible/NIR spectral regions were developed to optimize the identification efficiency of cotton samples with various leaf grade categories [18]. It was observed that using the discrimination model in the 1105–1700 nm NIR regions could distinguish one class of leaf grade fibers from other six groups at a satisfactory level of ~ 95.0%.

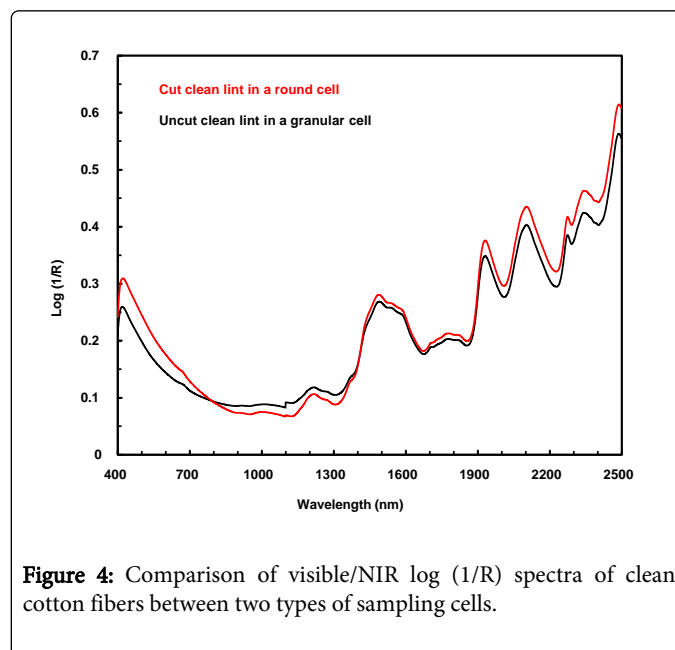


Figure 4: Comparison of visible/NIR log (1/R) spectra of clean cotton fibers between two types of sampling cells.

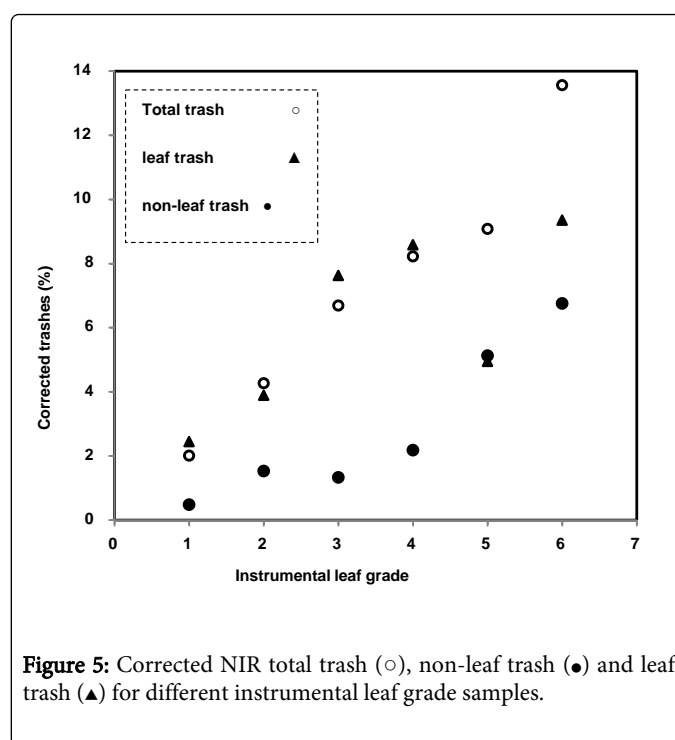


Figure 5: Corrected NIR total trash (○), non-leaf trash (●) and leaf trash (▲) for different instrumental leaf grade samples.

Conclusions

The transfer of NIR calibration models for the determination of total trash, leaf trash and non-leaf trash components in cotton fibers was attempted. In this scenario, the samples to be analyzed are inhomogeneous in a bulky state whereas the samples used as calibrations were much homogeneous in a ground state. Difference in sampling state (ground vs. bulky) generated a differing spectral response, and reasonably, direct transfer of NIR models built from grounded mixtures to bulky cotton fibers encounter some challenges. With the utilization of standard samples to correct or convert the original predictions, the results implied the feasibility of NIR technique for the rapid and quantitative determination of total trash, leaf trash and non-leaf trash components in commercial cotton fibers.

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