

Using NIRS To Predict Fiber and Nutrient Content of Dryland Cereal Cultivars

TAMI L. STUBBS,^{*,†} ANN C. KENNEDY,[‡] AND ANN-MARIE FORTUNA[†]

[†]Crop and Soil Sciences Department, Washington State University, Pullman, Washington 99164-6420 and

[‡]USDA-ARS, Land Management and Water Conservation Research Unit, Pullman, Washington 99164-6421

Residue from cultivars of spring wheat (*Triticum aestivum* L.), winter wheat, and spring barley (*Hordeum vulgare* L.) was characterized for fiber and nutrient traits using reference methods and near-infrared spectroscopy (NIRS). Calibration models were developed for neutral detergent fiber (NDF), acid detergent fiber (ADF), acid detergent lignin (ADL), carbon (C), sulfur (S), nitrogen (N), and C:N. When calibrations were tested against validation sets for each crop year, NIRS was an acceptable method for predicting NDF (standard error of prediction (SEP) < 0.87; R^2 > 0.90) and ADF (SEP < 0.81; R^2 > 0.92) and moderately successful for ADL in 1 year of the study (SEP = 0.44; R^2 = 0.81) but less successful for C, S, N, and C:N (R^2 all < 0.57). These results indicate that NIRS can predict the NDF and ADF of cereal residue from dryland cropping systems and is a useful tool to estimate residue decomposition potential.

KEYWORDS: NIRS; neutral detergent fiber (NDF); acid detergent fiber (ADF); acid detergent lignin (ADL); cultivars; dryland cropping systems; wheat; *Triticum aestivum* L.; barley; *Hordeum vulgare* L.

INTRODUCTION

Wheat (*Triticum aestivum* L.) and barley (*Hordeum vulgare* L.) are grown on more than 275 million hectares worldwide (1). The straw residue from these crops protects soils from erosion, contains nitrogen, phosphorus, sulfur, and potassium that may benefit future crop production, and can be utilized for livestock feed or bedding, biofuel production, composting, or mushroom production (2). Despite these benefits, difficulties with residue management and crop germination caused by excessive residue often results in growers choosing to burn crop residues, a detriment to air quality, or utilize intensive forms of tillage to prepare a seedbed for subsequent crops, leaving soils vulnerable to erosion. In contrast, residue returns in low-rainfall regions (150–300 mm annual precipitation) are often too low to prevent soil loss.

Conservation farming methods, including no-till farming, which retain surface crop residue have been slower to catch on in some regions of the world due to a lack of specialized equipment for farming on steep slopes or adequately managing large quantities of residue produced in areas of higher precipitation. In lower rainfall areas, it is difficult to establish a winter wheat crop without first creating a “dust mulch” fallow using intensive tillage. This dry layer of fine soil is intended to conserve moisture in the seed zone for crop emergence under dry conditions. High-disturbance tillage methods used to create the fallow nearly eliminate surface crop residue. In order to preserve soil organic matter and soil quality by encouraging adoption of conservation-farming methods, crop cultivars that decompose rapidly are needed in higher rainfall

areas, while cultivars that are more resistant to decomposition are needed in lower rainfall zones.

Numerous residue traits are responsible for determining the rate of residue decomposition; among them are hemicellulose, cellulose, lignin, carbon, and nitrogen content, as well as C:N ratio (3–5). Traits differ by crop type (6), location, and cultivar (7, 8). The wet chemistry methods used to predict hemicellulose, cellulose, and lignin content by measuring neutral detergent fiber (NDF), acid detergent fiber (ADF), and acid detergent lignin (ADL) are time-consuming, expensive, and environmentally detrimental due to the large amounts of chemicals used and waste generated. Likewise, the determination of carbon (C), sulfur (S), and nitrogen (N) by wet chemistry or dry oxidation is labor intensive and expensive and requires destruction of the crop sample.

Near-infrared spectroscopy (NIRS) is a secondary method to determine fiber and nutrient content, which is rapid, low-cost, and nondestructive to the crop sample. NIRS requires very little sample preparation and no chemicals, is reliable and accurate (9), allows a larger range of samples to be tested, and can be used to analyze multiple properties at one time (10). NIRS uses the near-infrared absorbance of a sample to measure organic functional groups and quantitatively predict a particular factor. NIRS is an accepted method to predict forage fiber traits of barley straw (11), rice (12, 13), flax stems (14), green cereal crops (15), leguminous shrubs (16), and oat hulls (17). NIRS accurately predicted the total N of sewage sludge (18), red fescue (*Festuca rubra* L.), and perennial ryegrass (*Lolium perenne* L.) (19), as well as the total C and N of forest floor materials (20). However, NIRS did not adequately predict dry matter and ash in maize plants (21) or N and P content in poultry litter (22).

*To whom correspondence should be addressed. Tel: (509)335-3453. Fax: (509)335-3842. E-mail: tlstubbs@wsu.edu.

The dryland cereal cropping region of eastern Washington state is characterized by diverse topography and wide variation in annual precipitation. The easternmost part of the region is distinguished by steep slopes and receives >450 mm average annual precipitation. The western portion of the cropping area is characterized by gently rolling slopes and receives <450 mm average annual precipitation. Soils of both regions are vulnerable to erosion when residue cover is inadequate. Development of NIRS as a rapid method for predicting residue decomposition traits in cereal residue is attractive because of the diverse growing conditions in eastern Washington and because more than 50 cultivars of wheat and barley are currently grown (23), with new cultivars continually being developed for release each year. There is a need for a faster and more accurate method to screen residue from cultivars presently grown, as well as those that will soon be utilized. The application of NIRS technology as a rapid method for predicting residue decomposition traits across a range of cereal cultivars would enable growers to better predict the persistence or loss of these materials from the field and allow growers to consider these traits when designing crop rotations.

The objectives of this research were to develop NIRS calibration models for spring wheat, winter wheat, and spring barley residue NDF, ADF, ADL, C, S, N, and C:N and to evaluate the ability of NIRS to predict these traits among different crop cultivars grown under diverse conditions. Our goal is to use NIRS to rank cultivars on their decomposition potential in soil and on their contributions to soil fertility.

MATERIALS AND METHODS

Sample Collection and Preparation. Straw residue from 16 spring wheat, 17 winter wheat, and 9 spring barley cultivars was collected at harvest from the Washington State University Cooperative Extension Variety Testing nurseries at Ritzville (282 mm average annual precipitation), Dusty (406 mm), Dayton (498 mm), and Pullman (521 mm). Residue was collected each of two consecutive crop years (2003 and 2004), and the experimental design was a completely randomized split split-plot, where time was the main plot and the location was the sub plot. Further information on climate, soils, and plot management can be found in the report by Stubbs et al. (8). Leaves and nodes were removed from the straw, and only the internode portion was used in these studies. Internode straw was oven-dried at 60 °C and ground to pass a 1 mm sieve.

Reference Methods. Neutral detergent fiber, ADF, and ADL contents were determined in stepwise fashion using the VanSoest et al. (24) procedure modified by using an ANKOM automated system with filter bags (ANKOM Technology Corp., Fairport, NY). Neutral detergent fiber includes hemicellulose, cellulose, and lignin, which are the insoluble cell wall components. Acid detergent fiber consists of the cellulose and lignin portions, with hemicellulose removed. Acid detergent lignin is the portion remaining after cellulose is removed. Ground straw samples were analyzed using a LECO CNS-2000 Elemental Analyzer (LECO Corp., St. Joseph, MI) to determine total C, S, and N.

Near-Infrared Spectroscopy. Ground samples were enclosed in stationary metal ring cups (36 mm inside diameter), and the reflectance was determined by scanning with a FOSS XDS Rapid Content Analyzer (Foss NIRSystems, Laurel, MD) using ISIScan software, version 3.10 (Infrasoft International, State College, PA). Samples were scanned twice using the wavelength range 400–2498 nm at 2 nm intervals, with the ring cup rotated 90° between scans. The mean of the two scans was used for data analysis.

Data Analysis. Data from each year were analyzed separately and randomly divided into two sets for developing calibration equations (2003, $n = 324$; 2004, $n = 300$) and for validation of equations (2003, $n = 298$; 2004, $n = 268$). Reference analysis and NIRS analysis were performed on each of the samples in both sets. Random selection of sample sets and calibration and validation statistics were conducted using the WinISI software, version 4.0 (Infrasoft International, State College, PA). Calibration equations were derived using modified partial least-squares (MPLS)

Table 1. Calibration Statistics for Prediction of 2003 and 2004 Crop Residue (Spring Barley, Spring Wheat, Winter Wheat) NDF, ADF, ADL, C, S, N, and C:N using NIRS^a

component	math treatment ^b		n	mean	SD	SEC	R ²	SECV	SD/1-VR	
									SECV	1-VR
(a) 2003 Crop										
NDF	1, 4, 4, 1		319	83.96	3.26	0.82	0.94	0.86	0.93	3.79
ADF	2, 10, 10, 1		321	53.73	3.03	0.74	0.94	0.85	0.92	3.56
ADL	1, 10, 10, 1		322	6.64	0.81	0.43	0.72	0.47	0.67	1.72
C	2, 4, 4, 1		277	47.70	0.71	0.54	0.41	0.61	0.25	1.16
S	1, 4, 4, 1		269	0.039	0.02	0.01	0.80	0.01	0.71	2.00
N	2, 10, 10, 1		273	0.188	0.09	0.05	0.71	0.05	0.64	1.80
C:N	2, 10, 10, 1		271	285.70	112.48	70.30	0.61	76.01	0.55	1.48
(b) 2004 Crop										
NDF	2, 4, 4, 1		294	85.25	2.35	0.68	0.92	0.76	0.90	3.09
ADF	2, 4, 4, 1		293	57.87	2.60	0.58	0.95	0.71	0.93	3.66
ADL	1, 4, 4, 1		294	9.55	0.98	0.34	0.88	0.39	0.85	2.51
C	3, 5, 5, 1		293	47.77	0.71	0.49	0.53	0.55	0.40	1.29
S	2, 4, 4, 1		293	0.050	0.02	0.02	0.59	0.02	0.57	1.00
N	1, 4, 4, 1		292	0.156	0.06	0.04	0.65	0.04	0.53	1.50
C:N	2, 10, 10, 1		291	340.51	129.74	91.34	0.50	95.09	0.46	1.36

^aThe scatter correction SNV and detrend was used. Legend: NDF, neutral detergent fiber; ADF, acid detergent fiber; ADL, acid detergent lignin; C, carbon; S, sulfur; N, nitrogen; C:N, carbon to nitrogen ratio; NIRS, near-infrared spectroscopy. ^bLegend: math treatment: derivative number, gap (nm), smooth (number of smoothing points), second smooth; SD, standard deviation; SEC, standard error of calibration; R², coefficient of determination; SECV, standard error of cross validation; 1-VR, 1 minus variance ratio.

and cross-validation techniques. The scatter correction of standard normal variant and detrend (SNV-D) was applied, along with several different math treatments for derivative order number, gap, and first smoothing. The second smoothing was set at 1 to indicate no second smoothing. Principal component analysis was used to identify and remove spectral outliers. Samples having spectra with Mahalanobis distance (H) values greater than 3.0 were considered outliers and were removed from the file. The appropriate calibration equation for each component was determined by selecting the one with the lowest standard error of cross validation (SECV) and the 1-variance ratio (1-VR) closest to 1 (9) (Table 1). The ratio of standard deviation (SD)/SECV was calculated (25) and used to determine calibration equations that are acceptable for quantitative prediction of fiber characteristics (Table 1). Correlations between NIRS-predicted values and reference values (wet chemistry or LECO) for each fiber component were determined using Pearson correlation coefficients (26).

RESULTS AND DISCUSSION

Calibration Equations. The calibration equations to predict NDF, ADF, ADL, C, S, N, and C:N of cereal crop residue (spring barley, spring wheat and winter wheat) varied with constituent (Table 1). The equations for each constituent also varied with the math treatment used (Table 1). The use of different yet specific calibration equations or math treatments for each data set increases the predictive ability of the data set (27), and for this reason the two crop years were analyzed separately. For each trait, we selected the math treatment that gave the highest 1-VR (closest to 1) and the lowest SECV, which is the standard error between laboratory reference values and NIRS-predicted values for the set of calibration samples (9). For the 2003 crop year calibration equations, the 1-VR values of fiber components NDF, ADF, and ADL were 0.93, 0.92, and 0.67, respectively, and the SECV values were 0.86, 0.85, and 0.47 (Table 1a). The 2003 carbon, sulfur, nitrogen, and C:N 1-VR values were 0.25, 0.71, 0.64, and 0.55 with SECV values of 0.61, 0.01, 0.05, and 76.01 (Table 1a). According to the guidelines utilized by Deville et al. (25), calibrations with

Table 2. Reference Measurements and Validation Results for the Prediction of 2003 and 2004 Cereal Residue (Spring Barley, Spring Wheat, Winter Wheat) NDF, ADF, ADL, C, S, N and C:N ($n = 298$)

component	n	reference measurements			validation results				
		range	measured mean	measured SD	NIRS predicted mean	bias	R^2	SEP	slope
(a) 2003 Crop									
NDF	297	73.86–89.05	83.74	3.55	83.73	0.013	0.93	0.94	1.01
ADF	297	44.93–60.43	53.58	3.10	53.55	0.034	0.93	0.87	0.94
ADL	297	4.67–8.87	6.58	0.77	6.61	–0.025	0.57	0.53	0.79
C	293	44.7–49.49	47.62	0.89	47.69	–0.070	0.30	0.75	0.983
S	293	0.011–0.093	0.039	0.017	0.039	0	0.49	0.013	0.75
N	293	0.049–0.649	0.205	0.12	0.198	0.007	0.48	0.09	1.07
C:N	293	74.87–953.79	309.93	166.62	281.16	28.78	0.24	147.79	0.89
(b) 2004 Crop									
NDF	268	71.6–89.46	85.03	2.78	85.13	–0.099	0.90	0.87	1.03
ADF	268	45.96–63.93	57.58	2.95	57.65	–0.067	0.92	0.81	1.01
ADL	268	7.29–12.76	9.50	0.99	9.59	–0.085	0.81	0.44	0.98
C	268	45.07–49.75	47.83	0.70	47.83	0.004	0.39	0.55	0.88
S	268	0.008–0.17	0.051	0.03	0.051	0.001	0.54	0.02	1.08
N	268	0.06–0.704	0.17	0.09	0.16	0.006	0.55	0.06	1.16
C:N	268	66.16–792.98	339.51	132.14	343.62	–4.115	0.47	96.43	0.97

SD/SECV ratios > 3.0 are acceptable for quantitative prediction, ratios > 2.5 and < 3.0 indicate equations that might be useful for screening, and ratios < 2.5 indicate the threshold where an equation is not useful. For the 2003 residue calibrations, only NDF (3.79) and ADF (3.56) SD/SECV ratios indicated equations with acceptable performance. When calibration equations were compared for the 2004 data set, the relationships are similar to the 2003 equations, with NDF and ADF having the highest 1-VR values of all the constituents, 0.90 and 0.93 (Table 1b). The SECV values for NDF and ADF were 0.76 and 0.71. For ADL, C, S, N, and C:N, 1-VR values were 0.85, 0.40, 0.57, 0.53, and 0.46, with SECV values of 0.39, 0.06, 0.02, 0.04, and 95.09 (Table 1b). The SD/SECV ratios for 2004 calibrations indicate that NDF (3.09) and ADF (3.66) equations are acceptable for quantitative prediction, while the ADL (2.51) equation would be useful for screening purposes. On the other hand, SD/SECV ratios for C, S, N, and C:N were too low, and the comparison between NIRS-predicted values and LECO-generated values may not be appropriate to use for C, S, and N estimation, especially for differences at the cultivar level.

Validation Results. Reference measurements and validation results for the 2003 and 2004 crop residue samples are shown in Table 2. In 2003, R^2 values for the constituents ranged from 0.24 for C:N to 0.93 for NDF and ADF (Table 2a). Besides NDF and ADF, R^2 values for all other constituents were too low to be acceptable. For 2004, R^2 values ranged from 0.39 for C to 0.92 for ADF (Table 2b). The NDF R^2 value for 2004 was 0.90, and the ADL R^2 value was 0.81 (Table 2b). The R^2 values for all other constituents of 2004 samples were too low to be acceptable. Laboratory reference data compared to NIRS-predicted results for NDF, ADF, and ADL (Figure 1) had slopes that approached 1.0 in both crop years, with the exception of ADL for 2003 samples, when the slope was 0.79 (Table 2a; Figure 1c). Because the relationships for NDF, ADF and ADL were similar for both 2003 and 2004, only the 2003 data is shown (Figure 1).

In evaluating NIRS as an acceptable prediction method of fiber and/or nutrient content, some researchers used criteria in addition to R^2 values. Mathison et al. (11) calculated RPD, which is the standard deviation of laboratory reference data divided by the standard error of prediction (SEP). They considered RPD values of 2.5 or greater to be adequate for acceptable prediction. For the validation results in 2003 and 2004, only the RPD values

for NDF (3.78, 3.2) and ADF (3.56, 3.64) would be considered successful following the Mathison et al. (11) guidelines. In addition to R^2 values and RPD, Malley et al. (28) include RER, which is the range of reference values divided by the SEP in their guidelines. Our results for NDF and ADF in both years would be considered successful according to the Malley et al. (28) guidelines (NDF RER = 16.1, 20.5; ADF RER = 17.8, 22.2). In addition, ADL for 2004 would be considered moderately successful ($R^2 = 0.81$, RPD = 2.25, RER = 12.4) under the Malley et al. (28) guidelines. Roggo et al. (29) utilized RPD and RER statistics in their screening of sugar beet quality and found that RPD values of 1–3 and RER values greater than 10 may indicate the usefulness of NIRS for screening. In our study, RPD and RER values for N in 2004 samples were 1.5 and 10.7. Validation results for the remaining constituents (C, S, C:N) would be useful for screening purposes only, rather than prediction of values, under the Malley et al. (28) guidelines.

NIRS values were significantly correlated ($P > 0.05$) with reference method values for most traits over all years, locations, and crops (Table 3). NDF, ADF, and ADL were well correlated with one another. Pearson correlation coefficients were greater than 0.95 for comparisons between methods for each trait and ranged from 0.76 to 0.32 for comparisons among those three traits. NDF was correlated with all other traits, with Pearson correlation coefficients ranging from 0.57 to 0.15. ADF did not correlate with carbon and sulfur but was correlated with nitrogen (0.55) and C:N (0.54). ADL was correlated with each of the traits (0.65 to 0.13). Carbon was not correlated with S, N, or C:N. We found lower and less consistent correlations between LECO C, N, and S content and NIRS-predicted C, N, and S than results for fiber fractions.

Several researchers have developed calibration equations for NDF, ADF, and ADL of various plant materials (17, 25, 30). Strong correlations between NIRS prediction and laboratory reference data have been found for NDF and ADF of barley (11) and green cereal crops (15). Weaker correlations, however, were found for ADL of barley (11), green cereal crops (15), rice straw (13), and leguminous shrubs (16). Shepherd et al. (31) reported high R^2 values for prediction of N and total soluble polyphenols in plant materials but a less robust prediction for lignin using NIRS. They reported only R^2 in their investigations and no other calculations, which may have limited their results.

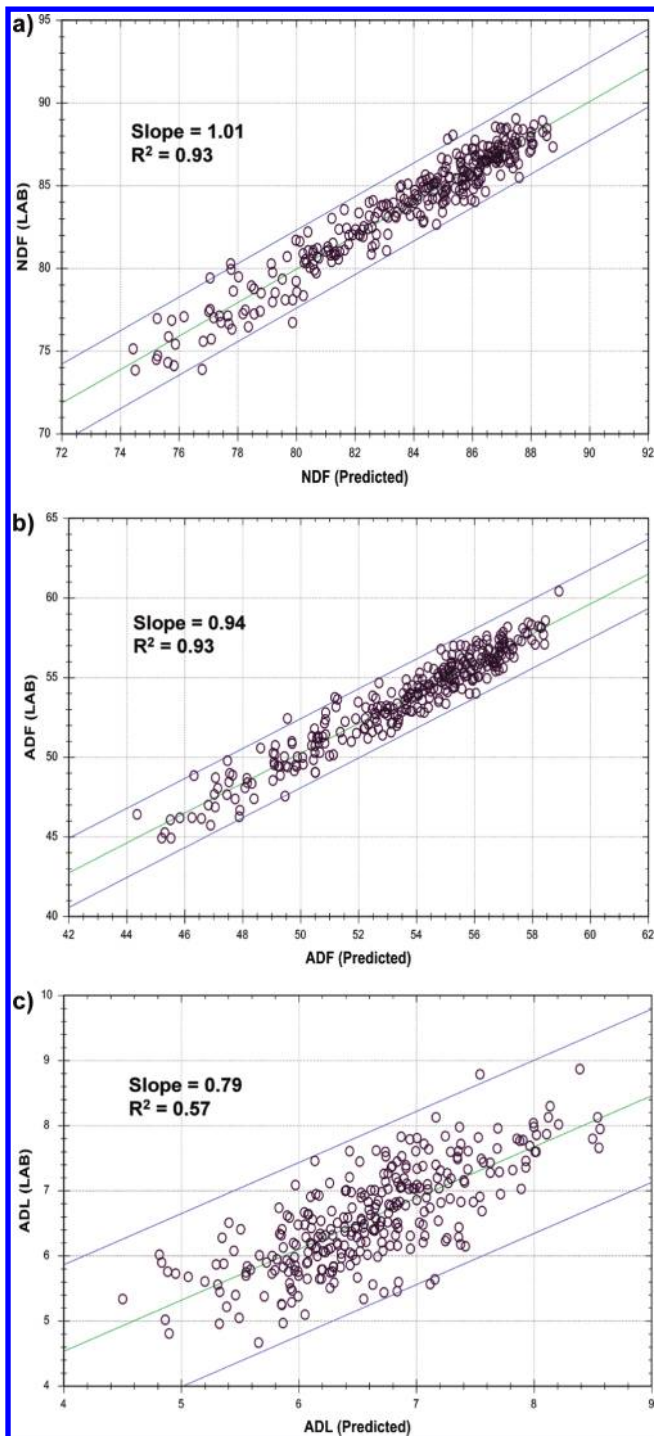


Figure 1. Linear regression relationship between laboratory reference methods and NIRS-predicted values for (a) percent neutral detergent fiber (NDF; $R^2 = 0.93$), (b) percent acid detergent fiber (ADF; $R^2 = 0.93$), and (c) percent acid detergent lignin (ADL; $R^2 = 0.57$) of spring barley, spring wheat, and winter wheat residue from 2003 on a dry weight basis.

Berardo et al. (32) showed that NIRS was capable of predicting the quality of pigeon pea (*Cajanus cajan* L.) NDF, ADF, ADL, and other traits for animal feed. They noted that correlation was generally not as strong for ADL but it was an acceptable prediction in their study, as it was in our study.

Malley et al. (28) showed NIRS to be successful in predicting manure C during composting but moderately successful for C:N and only moderately useful for N and S prediction. Halgerson et al. (33) found that NIRS was not consistent in predicting S in

Table 3. Coefficients of Pearson Correlation between Laboratory Reference Values and NIRS-Predicted Values for Cereal Residue Fiber Traits across All Years, Locations, and Cultivars^a

	NDF	ADF	ADL	C	S	N	C:N
NDF	0.9606 <0.0001	0.7633 <0.0001	0.3185 <0.0001	0.1556 0.0003	-0.3749 <0.0001	-0.5768 <0.0001	0.5332 <0.0001
ADF		0.9731 <0.0001	0.6480 <0.0001	0.0280 0.5201	-0.0547 0.2081	-0.5480 <0.0001	0.5357 <0.0001
ADL			0.9589 <0.0001	0.2925 <0.0001	0.4168 <0.0001	-0.1277 0.0032	0.1897 <0.0001
C				0.5862 <0.0001	0.0618 0.1553	-0.0323 0.4579	-0.0482 0.2674
S					0.7359 <0.0001	0.4997 <0.0001	-0.4764 <0.0001
N						0.7167 <0.0001	-0.6543 <0.0001
C:N							0.5733 <0.0001

^aFor each comparison, the top number indicates the Pearson correlation coefficient and the bottom number indicates the level of significance. Values in boldface type are significant at the $P < 0.05$ level.

Table 4. Coefficients of Pearson Correlation between Laboratory Reference Values and NIRS-Predicted Values for Cereal Residue Fiber Traits across the Four Growing Locations and Three Crops^a

	NDF	ADF	ADL	C	S	N	C:N
location							
Ritzville	0.9687 <0.0001	0.9809 <0.0001	0.9538 <0.0001	0.4413 <0.0001	0.8384 <0.0001	0.8902 <0.0001	0.8451 <0.0001
Dusty	0.9627 <0.0001	0.9628 <0.0001	0.9605 <0.0001	0.4831 <0.0001	0.7524 <0.0001	0.6163 <0.0001	0.2525 0.0031
Dayton	0.9121 <0.0001	0.9398 <0.0001	0.9565 <0.0001	0.3726 <0.0001	0.5662 <0.0001	0.6227 <0.0001	0.6431 <0.0001
Pullman	0.9536 <0.0001	0.9663 <0.0001	0.9644 <0.0001	0.7257 <0.0001	0.5993 <0.0001	0.3044 0.0004	0.4201 <0.0001
crop							
spring wheat	0.9720 <0.0001	0.9805 <0.0001	0.9627 <0.0001	0.5231 <0.0001	0.6568 <0.0001	0.6739 <0.0001	0.3897 <0.0001
winter wheat	0.9460 <0.0001	0.9621 <0.0001	0.9386 <0.0001	0.6018 <0.0001	0.5806 <0.0001	0.3994 <0.0001	0.4321 <0.0001
spring barley	0.9636 <0.0001	0.9759 <0.0001	0.9748 <0.0001	0.5432 <0.0001	0.6686 <0.0001	0.6547 <0.0001	0.5505 <0.0001

^aFor each comparison, the top number indicates the Pearson correlation coefficient and the bottom number indicates the level of significance ($P < 0.05$).

alfalfa. Others have found NIRS to be less accurate for prediction of ADF in poultry litter (22) and unreliable for predicting N in poultry litter (22, 34). Reeves (35), on the other hand, found NIRS able to predict total N but not S in poultry litter. LECO was used in C, N, or S analysis for each of these studies, as we did in our study. Gislum et al. (19), Michel et al. (20), and Galvez-Sola et al. (18), among others, have been successful in using NIRS to predict N and/or C but with different methods for determining reference values of plants and other materials. Improved calibration equations, possibly on larger populations of samples, will need to be developed before NIRS can be used to adequately predict elemental content of cereal residue.

Environment, Crop Types, and Cultivars. Cereal residue varies in fiber content with growing location (7, 8, 36) and crop (6, 8). In this study, NIRS and reference methods equally ranked the four growing locations according to predicted fiber quality traits in spring and winter wheat and spring barley. Pearson correlation coefficients were greater than 0.90 for NDF, ADF, and ADL for all locations and crops and were below 0.90 for C, S, N, and C:N (Table 4). The Ritzville location had correlation coefficients

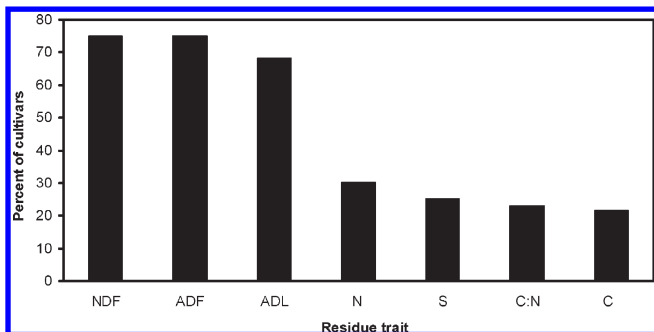


Figure 2. Percent of spring wheat, winter wheat, and spring barley cultivars where NIRS-predicted values were significantly correlated ($P < 0.05$) with laboratory reference values for the residue traits NDF, ADF, ADL, nitrogen, sulfur, C:N, and carbon.

ranging from 0.89 to 0.84 for S, N, and C:N, while correlation coefficients for the same traits at other locations were less than 0.75. Nitrogen and C:N were not well correlated between analysis methods at Pullman, nor was C at Dayton or C:N at Dusty. All other locations and all other analyses were well correlated. We found that NIRS and reference methods were well correlated for all crops across all traits, except for C:N in spring wheat, and N in winter wheat (Table 4). Pearson correlation coefficients were greater than 0.90 for NDF, ADF, and ADL of each crop.

Redaelli and Berardo (17) used NIRS to predict the NDF, ADF, and ADL of oat hulls. They showed that oat cultivars grown at various locations differed in their fiber contents, and overall, cultivar played a stronger role than location in determining variability. Al Haj Khaled et al. (37) used NIRS analysis of leaf blade chemical components (fiber, cellulose, hemicellulose, lignin) to rank grass species based on their nutritive value. For both years of this study, NIRS and wet chemistry values for ADF and NDF were significantly correlated ($P < 0.05$) for 75% of the cultivars (Figure 2). NIRS values for ADL were significantly correlated with wet chemistry values for 68% of the cultivars tested in both years. For C, N, S, and C:N, NIRS and LECO analyses were not well correlated, with less than 25% of the cultivars showing significant correlations (Figure 2). From our data we can conclude that NIRS is able to differentiate NDF and ADF, and ADL to a lesser extent, and NIRS can be used to predict decomposition potential for each cultivar without using the more labor intensive wet-chemistry methods. At this time, NIRS prediction would not be used to replace C, N, and S analysis using the classic LECO method to rank cereal cultivars for their decomposition potential.

Cereal cultivars vary in their fiber composition (7, 8), and we wanted to determine if cultivars could be simply yet accurately ranked according to their decomposition potential. Cultivars of spring wheat, winter wheat, and spring barley were ranked as “fast”, “medium”, or “slow” on the basis of laboratory reference values for fiber traits and again on the basis of the NIRS-predicted values for the same traits. We found that, for ADF, 87% of cultivars were ranked the same with the two methods, 82% for NDF, and 81% for ADL. For each of those traits, there were no cultivars that were ranked opposite by the two methods. The percent of cultivars ranked correctly for C, S, N, and C:N was only 53, 68, 63, and 65. For each of those traits, some cultivars were ranked opposite on the basis of their potential for decomposition when comparing the two methods: 8% for carbon, 2% for sulfur, 4% for nitrogen, and 5% for C:N. This ranking scheme shows that we can most accurately categorize cultivars for their potential to decompose slowly or rapidly according to their NIRS-predicted values for NDF, ADF, and ADL.

Residue decomposition rate can be predicted by hemicellulose, cellulose, and lignin content of plant residue, among other traits (38), which are calculated from analysis for NDF, ADF, and ADL. We found that NIRS was a successful method for predicting NDF and ADF, and sometimes ADL, in cereal crop residue. The calibrations were less consistent and less successful for prediction of straw C, S, and N; however, we can use the results of NIRS analysis to predict indicators for residue decomposition based on values for NDF and ADF and include LECO values for C, S, and N. Although LECO analysis is time-consuming, it generates no toxic waste and coupled with NIRS analysis of fiber components yields valuable information for the prediction of decomposition potential of cereal residue. Future work will involve building and strengthening calibration equations and validating these equations with additional populations of cereal residue samples. This work might also include prediction of residue tannin content and correlation with residue decomposition in laboratory and field studies.

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LITERATURE CITED

- (1) USDA Foreign Agricultural Service. *World agricultural production*. 2009, <http://www.fas.usda.gov/wap/current/toc.asp> (verified 7/22/2009).
- (2) CIMMYT. *Crop residue management fact sheet*. 2007, <http://www.knowledgebank.irri.org/Wheat/factsheets/Crop%20residue%20management.pdf> (verified 7/22/2009).
- (3) Johnson, J. M.-F.; Barbour, N. W.; Weyers, S. L. Chemical composition of crop biomass impacts its decomposition. *Soil Sci. Soc. Am. J.* 2007, 71, 155–162.
- (4) Goh, K. M.; Tutuna, S. S. Effects of organic and plant residue quality and orchard management practices on decomposition rates of residues. *Commun. Soil Sci. Plant Anal.* 2004, 35, 441–460.
- (5) Baggie, I.; Rowell, D. L.; Robinson, J. S.; Warren, G. P. Decomposition and phosphorus release from organic residues as affected by residue quality and added inorganic phosphorus. *Agrofor. Sys.* 2004, 63, 125–131.
- (6) Smith, J. H.; Peckenpaugh, R. E. Straw decomposition in irrigated soil: Comparison of twenty-three cereal straws. *Soil Sci. Soc. Am. J.* 1986, 50, 928–932.
- (7) Chalaux, N.; Libmond, S.; Savoie, J.-M. A practical enzymatic method to estimate wheat straw quality as raw material for mushroom cultivation. *Bioresour. Technol.* 1995, 53, 277–281.
- (8) Stubbs, T. L.; Kennedy, A. C.; Reisenauer, P. E.; Burns, J. B. Chemical composition of cereal crops and cultivars from dryland ecosystems. *Agron. J.* 2009, 101, 538–545.
- (9) Foss North America. *ISIScan & WinISI Software Training Class*, 2008.
- (10) Stuth, J.; Jama, A.; Tolleson, D. Direct and indirect means of predicting forage quality through near infrared reflectance spectroscopy. *Field Crops Res.* 2003, 84, 45–56.
- (11) Mathison, G. W.; Hsu, H.; Soofi-Siawash, R.; Recinos-Diaz, G.; Okine, E. K.; Helm, J.; Juskiw, P. Prediction of composition and ruminal degradability characteristics of barley straw by near infrared reflectance spectroscopy. *Can. J. Anim. Sci.* 1999, 79, 519–523.
- (12) Kong, X.; Xie, J.; Wu, X.; Huang, Y.; Bao, J. Rapid prediction of acid detergent fiber, neutral detergent fiber, and acid detergent lignin of rice materials by near-infrared spectroscopy. *J. Agric. Food Chem.* 2005, 53, 2843–2848.
- (13) Jin, S.; Chen, H. Near-infrared analysis of the chemical composition of rice straw. *Ind. Crops Prod.* 2007, 26, 207–211.

- (14) Barton, F. E. II; Akin, D. E.; Morrison, W. H.; Ulrich, A.; Archibald, D. D. Analysis of fiber content in flax stems by near-infrared spectroscopy. *J. Agric. Food Chem.* **2002**, *50*, 7576–7580.
- (15) Bruno-Soares, A. M.; Murray, I.; Paterson, R. M.; Abreu, J. M. F. Use of near infrared reflectance spectroscopy (NIRS) for the prediction of the chemical composition and nutritional attributes of green crop cereals. *Anim. Feed Sci. Technol.* **1998**, *75*, 15–25.
- (16) Garcia Ciudad, A.; Fernandez Santos, B.; Vazquez de Aldana, B. R.; Zabalgoceazcoa, I.; Gutierrez, M. Y.; Garcia Criado, B. Use of near infrared reflectance spectroscopy to assess forage quality of a Mediterranean shrub. *Commun. Soil Sci. Plant Anal.* **2004**, *35*, 665–678.
- (17) Redaelli, R.; Berardo, N. Prediction of fibre components in oat hulls by near infrared reflectance spectroscopy. *J. Sci. Food Agric.* **2007**, *87*, 580–585.
- (18) Galvez-Sola, L.; Moral, R.; Moreno-Caselles, J.; Perez-Murcia, M. D.; Perez-Espinosa, A.; Bustamante, M. A.; Paredes, C. Effectiveness of near infrared reflectance spectroscopy in the quick evaluation of nitrogen content in sewage sludge. *Commun. Soil Sci. Plant Anal.* **2009**, *40*, 726–735.
- (19) Gislum, R.; Micklander, E.; Nielsen, J. P. Quantification of nitrogen concentration in perennial ryegrass and red fescue using near-infrared reflectance spectroscopy (NIRS) and chemometrics. *Field Crops Res.* **2004**, *88*, 269–277.
- (20) Michel, K.; Terhoeven-Urselmans, T.; Nitschke, R.; Steffan, P.; Ludwig, B. Use of near- and mid-infrared spectroscopy to distinguish carbon and nitrogen originating from char and forest-floor material in soils. *J. Plant Nutr. Soil Sci.* **2009**, *172*, 63–70.
- (21) Cozzolino, D.; Fassio, A.; Gimenez, A. The use of near-infrared reflectance spectroscopy (NIRS) to predict the composition of whole maize plants. *J. Sci. Food Agric.* **2000**, *81*, 142–146.
- (22) Aiken, G. E.; Pote, D. H.; Tabler, S. F.; Tabler, T. C. Application of near-infrared reflectance spectroscopy to estimate chemical constituents in broiler litter. *Commun. Soil Sci. Plant Anal.* **2005**, *36*, 2529–2539.
- (23) USDA-National Agricultural Statistics Service. *Washington small grains*. **2008**, http://www.nass.usda.gov/Statistics_by_State/Washington/Publications/Small_Grains/index.asp (verified 7/22/2009).
- (24) VanSoest, P. J.; Robertson, J. B.; Lewis, B. A. Methods for dietary fiber, neutral detergent fiber, and nonstarch polysaccharides in relation to animal nutrition. *J. Dairy Sci.* **1991**, *74*, 3583–3597.
- (25) Deaville, E. R.; Humphries, D. J.; Givens, D. I. Whole crop cereals 2. Prediction of apparent digestibility and energy value from *in vitro* digestion techniques and near infrared reflectance spectroscopy and of chemical composition by near infrared reflectance spectroscopy. *Anim. Feed Sci. Technol.* **2009**, *149*, 114–124.
- (26) SAS Institute Inc. *SAS/STAT User's Guide*; SAS Institute: Cary, NC, 2007.
- (27) Ruano-Ramos, A.; Garcia-Ciudad, A.; Garcia-Criado, B. Determination of nitrogen and ash contents in total herbage and botanical components of grassland systems with near infra-red spectroscopy. *J. Sci. Food Agric.* **1999**, *79*, 137–143.
- (28) Malley, D. F.; McClure, C.; Martin, P. D.; Buckley, K.; McCaughey, W. P. Compositional analysis of cattle manure during composting using a field-portable near-infrared spectrometer. *Commun. Soil Sci. Plant Anal.* **2005**, *36*, 455–475.
- (29) Roggo, Y.; Duponchel, L.; Huvenne, J.-P. Quality evaluation of sugar beet (*Beta vulgaris*) by near-infrared spectroscopy. *J. Agric. Food Chem.* **2004**, *52*, 1055–1061.
- (30) Berzaghi, P.; Cozzi, G.; Andrighetto, I. The use of near infrared analysis for *in situ* studies. *J. Dairy Sci.* **1997**, *80*, 3263–3270.
- (31) Shepherd, K. D.; Palm, C. A.; Gachengo, C. N.; Vanlauwe, B. Rapid characterization of organic resource quality for soil and livestock management in tropical agroecosystems using near-infrared spectroscopy. *Agron. J.* **2003**, *95*, 1314–1322.
- (32) Berardo, N.; Dzwowela, B. H.; Hove, L.; Odoardi, M. Near infrared calibration of chemical constituents of *Cajanus cajan* (pigeon pea) used as forage. *Anim. Feed Sci. Technol.* **1997**, *69*, 201–206.
- (33) Halgerson, J. L.; Sheaffer, C. C.; Martin, N. P.; Peterson, P. R.; Weston, S. J. Near-infrared reflectance spectroscopy prediction of leaf and mineral concentrations in alfalfa. *Agron. J.* **2004**, *96*, 344–351.
- (34) Tasistro, A. S.; Shaaban, S.; Kissel, D. E.; Vendrell, P. F. Near-infrared reflectance spectroscopy for the analysis of water and total nitrogen contents in poultry litter. *Commun. Soil Sci. Plant Anal.* **2003**, *34*, 1367–1379.
- (35) Reeves, J. B., III. Near-infrared diffuse reflectance spectroscopy for the analysis of poultry manures. *J. Agric. Food Chem.* **2001**, *49*, 2193–2197.
- (36) Grando, S.; Baum, M.; Ceccarelli, S.; Goodchild, A.; Jaby El-Haramein, F.; Jahoor, A.; Backes, G. QTLs for straw quality characteristics identified in recombinant inbred lines of a *Hordeum vulgare* x *H. spontaneum* cross in a Mediterranean environment. *Theor. Appl. Genet.* **2005**, *110*, 688–695.
- (37) Al Haj Khaled, R.; Duru, M.; Decruyenaere, V.; Jouany, C.; Cruz, P. Using leaf traits to rank native grasses according to their nutritive value. *Rangeland Ecology Management* **2006**, *59*, 648–654.
- (38) Wolf, D. C.; Wagner, G. H. Carbon transformations and soil organic matter formation. In *Principles and Applications of Soil Microbiology*; Sylvia, D. M., Fuhrmann, J. J., Hartel, P. G., Zuberer, D. A., Eds.; Prentice-Hall: Englewood Cliffs, NJ, **2005**; pp 285–332.

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