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Rapid Prediction of Acid Detergent Fiber, Neutral Detergent Fiber, and Acid Detergent Lignin of Rice Materials by Near-Infrared Spectroscopy

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A rapid predictive method based on near-infrared spectroscopy (NIRS) was developed to measure acid detergent fiber (ADF), neutral detergent fiber (NDF), and acid detergent lignin (ADL) of rice stem materials. A total of 207 samples were divided into two subsets, one subset (∼136 samples) for calibration and cross-validation and the other subset for independent external validation to evaluate the calibration equations. Different mathematical treatments were applied to obtain the best calibration and validation results. The highest coefficient of determination for calibration (R^2) and coefficient of determination for cross-validation (1-VR) were 0.968 and 0.949 for ADF, 0.846 and 0.812 for NDF, and 0.897 and 0.843 for ADL, respectively. Independent external validation still gave a high coefficient of determination for external validation (r^2) and a low standard error of performance (SEP) for the three parameters; the best validation results were SEP = 0.933 and $r^2 = 0.959$ for ADF, SEP = 2.228 and $r^2 = 0.775$ for NDF, and SEP = 0.616 and $r^2 = 0.847$ for ADL, indicating that NIR gave a sufficiently accurate prediction of ADF and ADL content of rice material but a less satisfactory prediction for NDF. This study suggested that routine screening for these forage quality parameters with large numbers of samples is possible with NIRS in early-generation selection in rice-breeding programs.

KEYWORDS: Rice; near-infrared spectroscopy; acid detergent fiber; neutral detergent fiber; acid detergent lignin

INTRODUCTION

During the development of the rice plant, the stem supports the plant in growth, in bearing grain, and in resisting lodging. Maize stalk strength is related to lignin synthesis (*1*), a component of the cell wall that is very important to support the plant (i.e., to avoid lodging) and to protect plants against the external environment. After grain harvest, rice straw (stems and leaves) becomes essentially a waste byproduct of the rice grain industry. Effective utilization of rice straw will definitely increase the income of rice producers, whereas improper disposal of rice straw, such as by burning, will produce pollution. Some novel approaches have been developed to extend the utilization of rice straw. In addition to returning it to the field as manure (*2*), rice straw has the potential to be used for bio-oil extraction by pyrolysis and steam pyrolysis (*3*), for papermaking (*4*, *5*), for mushroom growth (*6*), and even for dietary fiber preparation (*7*), etc. However, the most important application is use for feed of livestock, such as cattle, goats, and sheep. For this purpose, acid detergent fiber (ADF), neutral detergent fiber (NDF), and acid detergent lignin (ADL) have been established as important and good indicators of forage quality because they are well correlated to digestibility for livestock animals (*8*−*13*). ADF, NDF, and ADL are traditionally analyzed according to the method proposed by Van Soest et al. (*14*). This method involves a long period of detergent washing, needs intensive labor input, and produces pollution. For rice-breeding programs, to improve rice straw quality or rice stem resistance to lodging, a rapid and cost-effective method to analyze these traits needs to be developed.

Near-infrared spectroscopy (NIRS) is a rapid, accurate, and nondestructive technique that has been extensively used both quantitatively and qualitatively in the analysis of forage quality (*15*−*19*). Determining ADF content in grasses by NIRS has been adopted as an AOAC International Official Method (*20*, *21*). Research is still underway to develop predictive models

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Table 1. Range, Mean, and SD of Rice Stem ADF, NDF, and ADL Content (Percent Dry Weight) by Analytical Methods in the Calibration and Validation Subsets[a]

parameter	calibration				external validation			
	no.	range	mean	SD	no.	range	mean	SD
ADF	138	26.29–44.94	38.16	3.95	69	26.30–45.76	36.76	4.62
NDF	136	45.00–75.29	59.18	5.18	68	48.58–69.33	58.07	4.50
ADL	135	4.48–11.60	7.27	1.23	68	4.14–11.59	6.95	1.57

[a] ADF, acid detergent fiber; NDF, neutral detergent fiber; ADL, acid detergent lignin; no.: number of samples; SD, standard deviation.

for these parameters for more crops. It is reported that different drying methods affected chemical composition (including ADF and NDF) and NIR spectra of pasture silage and forage; freeze-drying gave consistent results and was suitable for NIRS analysis (*22*, *23*). Bruno-Soares et al. (*16*) developed satisfactory NIR calibration models for ADF, NDF, and ADL with coefficient of determination for calibration (R^2) values of >0.87 in cereal crops including oat, barley, triticale, wheat, ryegrass, and sorghum. Berardo et al. (*15*) studied chemical constituents in pigeon pea by NIR analysis and found strong relationships of NIR data with ADF, NDF, and ADL. In addition to forage quality, NIR has been also successfully used to predict ADF in the seed of oilseed Brassica (*24*), ADF and NDF of green asparagus (*25*), lignin content in wood meal of loblolly pine (*26*), and total dietary fiber in cereal food products (*27*).

NIRS has been widely used in the determination of chemical compositions in rice, such as starch content and properties (*28*−*30*), protein content (*31*), etc. To our knowledge, no study has been conducted to establish NIRS calibration equations for analysis of ADF, NDF, and ADL contents of rice materials. The objective of the present study was to develop a rapid and accurate NIRS measurement of these quality parameters and to evaluate its potential application in rice breeding to improve forage quality or to improve lodging resistance.

MATERIALS AND METHODS

Rice Stem Materials Collection and Treatments. Stem samples (207) from an F_6 population developed from a cross between Dongxiang wild rice (*Oryza rufipogon*) and Xieqingzao B (*Oryza sativa* L.) were obtained at China National Rice Research Institute in September 2003. Stems were taken from only those plants bearing effective spikes. They were first washed to remove soil on the base and then dried in a forced-air oven for 72 h at 65 °C. The sheath and leaves were removed, and only three nodes from the base up were collected. Prior to grinding of the samples to pass a 1 mm sieve, they were dried again for another 24 h at 65 °C for moisture control and to enhance grinding.

NIR Spectroscopic Analysis. NIR System model 5000 monochromator (Foss-NIR System, Inc., Silver Spring, MD) was used to obtain the NIR reflectance spectra of each dried ground sample under the control of the software WinISI II Project Manager version 1.50. The sample was placed in the ring cup (35 mm inner diameter, 8 mm depth) ~80% full and was scanned in duplicate (rotating the cup to a different angle to get another spectrum of the same sample) with the wavelength range of 1100−2498 nm. The spectra were recorded as log(1/*R*) at 2 nm intervals.

Calibration and Validation. All sample spectra were divided into two subsets, one of 136 samples used to develop the calibration equation and the other used to evaluate the calibration equation through different mathematical treatments (**Table 1**). Calibration and validation were conducted with the WinISI II Project Manager version 1.50 software. Different math treatments with scatter correction of standard normal variate and detrend (SNV-D) were applied for calibration (*32*). Taking math treatment, D = 1, G = 4, S1 = 4, S2 = 1, for example, D is the derivative order number (that is, 0 indicates no derivative operation, 1 means first derivative, and so on); G is gap (the number of data points over which derivation is computed), S1 is the number of data points in the first smoothing, and S2 is the number of data points in the second smoothing, which is normally set at 1 for no second smoothing. Modified partial least squares (MPLS) were used to develop the regression equations. The major statistics are standard error of calibration (SEC) and the coefficient of determination (R^2) for calibration, coefficient of determination (1-VR), and standard error of cross-validation (SECV) for cross-validation (*33*). The prediction ability of equations was tested based on the following statistics: coefficient of determination (r^2), slope, bias, and standard error of performance (SEP) (*33*). In addition, the parameters SD/SEP ratio and SEP/SEL ratio (i.e., the SEP with respect to the standard error of laboratory) (*24*, *33*, *34*) were also used to evaluate the precision of an NIR equation.

Reference Analysis. After all NIR spectra were recorded, ADF, NDF, and ADL contents on the dry weight basis were measured according to the method described by AOAC (*35*) and Van Soest et al. (*14*). To get enough residuals for ADL analysis, the ADF were measured in triplicate and the others in duplicate.

RESULTS AND DISCUSSION

ADF, NDF, and ADL Contents by Reference Methods. The average, range, and standard deviation (SD) values of ADF, NDF, and ADL in the samples used in the calibration subset and external validation subset are summarized in **Table 1**. Wide diversity in the component values was found within the calibration subset (**Table 1**); ADF ranged from 26.29 to 44.94%, NDF from 45.00 to 75.29%, and ADL from 4.48 to 11.60%, respectively. The ranges of ADF and NDF were a little larger than those in rice landraces reported by Agnihotri et al. (*9*), who found ADF varied from 42.70 to 54.58% and NDF varied from 63.64 to 78.46%. The difference might result from different rice genotypes used; Agnihotri et al. (*9*) used rice landraces and planted them across various locations, whereas the present study used lines derived from a wild rice that has much lower ADF and NDF contents than cultivated rice. Another reason was that Agnihotri et al. (*9*) used whole fodder, whereas we only used stem to measure these parameters, and ADF and NDF in leaves were higher than those in stems. Similar ranges for ADF and NDF contents were found in other cereals (barley, oat, triticale, wheat, ryegrass, and sorghum), but these samples were taken at different maturities (*16*). The ranges of the validation subset were as wide as those of the calibration subset (**Table 1**).

Spectral Analysis for Typical Samples. In general, the spectral traces of rice stem materials (**Figure 1A**) were very similar to those of other grasses (*16*, *23*). Weak bands between 1420 and 1530 nm, stronger bands from 1890 to 2024 nm, and several combined slopes thereafter were characteristic spectra of grasses, which indicated the energy absorption of bonds from residual water (which absorbs at 1420 and 1850−1980 nm), alcohols (1400−1460, 1975−2125 nm), phenolic compounds (1415−1512, 1955−2035 nm), amines (1449−1538, 1965−2025 nm), and protein bonds (2275−2500 nm) (*23*, *36*). Samples a and d had different ADF contents, but they had similar NDF and ADL contents. Likewise, samples a and c had different NDF contents, but their ADF and ADL contents were similar, whereas samples b and c had different ADL contents but similar ADF and NDF contents. **Figure 1B**−**D** shows the curves in which the scatter correction method SNV + D was performed in the whole spectral region of 1100−2498 nm. Distinct spectral differences between them could be attributed to the difference in ADF (**Figure 1B**), NDF (**Figure 1C**), and ADL (**Figure 1D**). The absorbance at 2308 and 2348 nm was reported to be highly related to oilseed ADF (*24*); this region was also related to rice stem ADF in the present study because it was different in **Figure 1B**, whereas they were the same in **Figure 1C**,**D**. However,

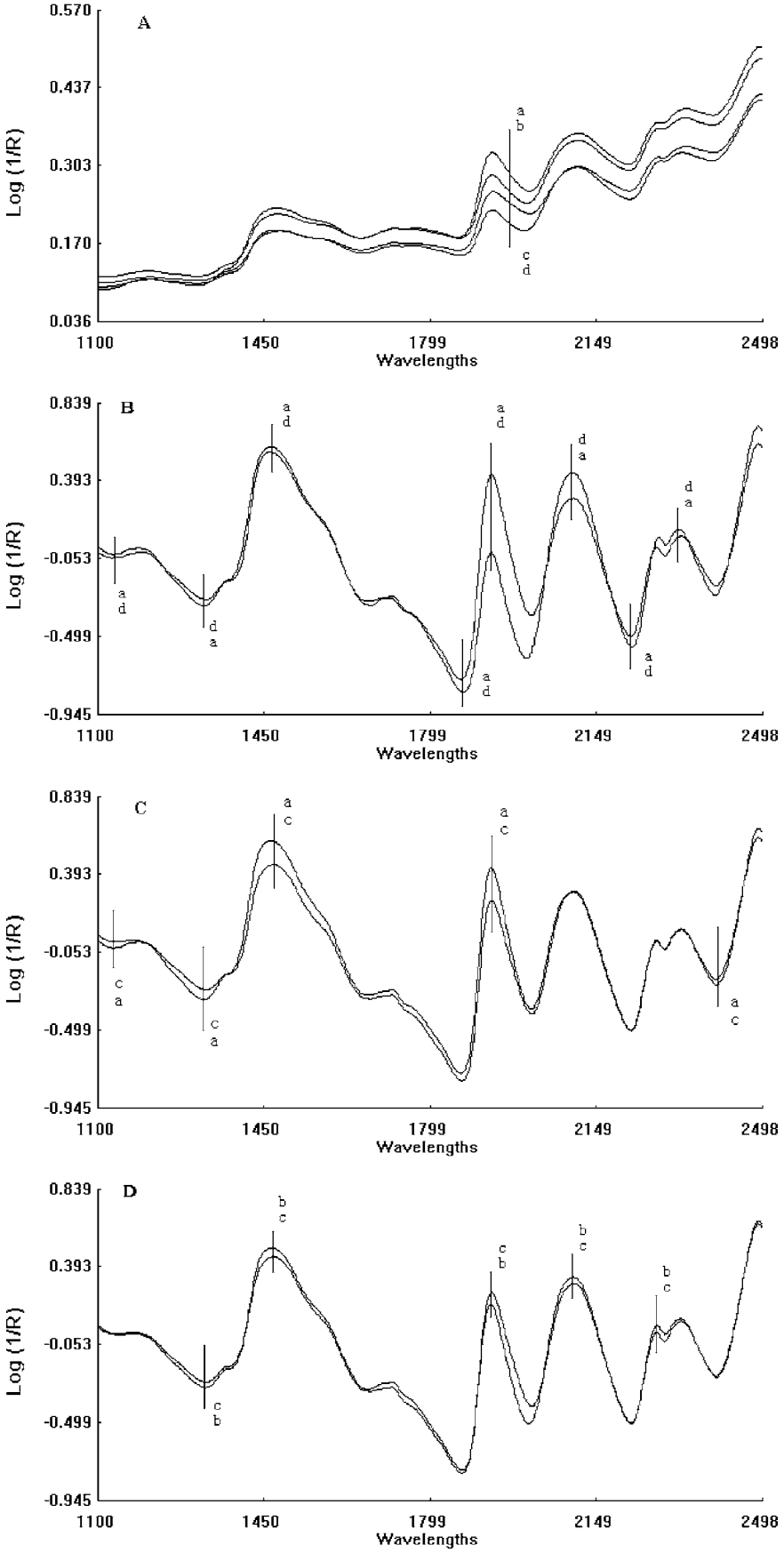

Figure 1. Typical NIR spectra of four rice samples with different combinations of ADF, NDF, and ADL contents (**A**) and comparison of the spectra scatter correction procedure by SNV and D curves (**B**–**D**) attributed to the main difference in ADF (**B**), NDF (**C**), and ADL (**D**). All of the spectra scatter was performed on the whole spectral data ranging from 1100 to 2498 nm. The contents of ADF, NDF, and ADL for the four samples were (a) 41.07, 70.27, and 7.14%; (b) 40.41, 57.48, and 11.60%; (c) 39.07, 57.52, and 6.55%; and (d) 30.90, 70.82, and 5.70%.

Table 2. Calibration and Cross-Validation Statistics for ADF, NDF, and ADL (Percent Dry Weight) in the Rice Stem Using Different Mathematical Treatments[a]

parameter	mathematical treatments[b]	factors	calibration					cross-validation	
			mean	range	SD	SEC	R^2	SECV	1-VR
ADF	0, 0, 1, 1	7	38.21	26.69–49.72	3.84	0.94	0.9398	0.96	0.9385
	1, 4, 4, 1	7	38.25	26.69–49.81	3.85	0.84	0.9522	0.95	0.9394
	1, 5, 5, 1	7	38.25	26.69–49.81	3.85	0.84	0.9523	0.96	0.9390
	2, 4, 4, 1	7	38.24	26.77–49.71	3.82	0.69	0.9675	0.88	0.9485
	2, 5, 5, 1	7	38.26	26.73–49.79	3.84	0.68	0.9691	0.82	0.9557
	2, 8, 6, 1	7	38.26	26.73–49.79	3.84	0.71	0.9657	0.82	0.9556
NDF	0, 0, 1, 1	5	58.99	44.35–73.63	4.88	2.03	0.8262	2.21	0.7946
	1, 4, 4, 1	3	58.95	44.16–73.73	4.93	2.11	0.8171	2.20	0.8002
	1, 5, 5, 1	3	58.95	44.16–73.73	4.93	2.10	0.8118	2.20	0.8007
	2, 4, 4, 1	4	58.83	44.08–73.58	4.92	1.93	0.8458	2.13	0.8126
	2, 5, 5, 1	3	58.95	44.16–73.73	4.93	2.20	0.8016	2.35	0.7730
	2, 8, 6, 1	3	58.95	44.16–73.73	4.93	2.19	0.8033	2.32	0.7786
ADL	0, 0, 1, 1	9	7.19	3.72–10.66	1.16	0.46	0.8410	0.54	0.7832
	1, 4, 4, 1	8	7.20	3.76–10.63	1.14	0.40	0.8760	0.49	0.8187
	1, 5, 5, 1	8	7.22	3.77–10.66	1.15	0.42	0.8679	0.50	0.8093
	2, 4, 4, 1	7	7.23	3.66–10.80	1.19	0.38	0.8973	0.47	0.8432
	2, 5, 5, 1	7	7.22	3.66–10.79	1.19	0.40	0.8844	0.48	0.8382
	2, 8, 6, 1	7	7.22	3.66–10.79	1.19	0.42	0.8778	0.47	0.8415

[a] R^2, the coefficient of determination for calibration; SD, standard deviation of the reference data; SEC, standard error of calibration; SECV, standard error of cross-validation; 1-VR, coefficient of determination for cross-validation. [b] Mathematical treatment is in the order of derivative, gap, first smoothing, and second smoothing.

Table 3. External Validation Statistics for ADF, NDF, and ADL (Percent Dry Weight) Using the Equations Developed from Different Mathematical Treatments[a]

parameter	mathematical treatments	external validation							
		SD	SEL	SEP	bias	slope	SD/SEP	SEP/SEL	r^2
ADF	0, 0, 1, 1	4.622	0.753	1.164	–0.235	1.046	3.97	1.55	0.938
	1, 4, 4, 1			1.008	–0.170	1.044	4.59	1.34	0.954
	1, 5, 5, 1			1.021	–0.144	1.049	4.53	1.36	0.953
	2, 4, 4, 1			0.951	–0.136	1.011	4.86	1.26	0.958
	2, 5, 5, 1			0.945	–0.136	1.013	4.89	1.25	0.958
	2, 8, 6, 1			0.933	–0.178	1.009	4.95	1.24	0.959
NDF	0, 0, 1, 1	4.497	1.352	2.373	0.549	0.886	1.90	1.76	0.734
	1, 4, 4, 1			2.239	0.437	0.884	2.01	1.66	0.765
	1, 5, 5, 1			2.239	0.434	0.884	2.01	1.66	0.765
	2, 4, 4, 1			2.228	0.624	0.861	2.02	1.65	0.775
	2, 5, 5, 1			2.257	0.388	0.900	1.99	1.67	0.757
	2, 8, 6, 1			2.274	0.416	0.901	1.98	1.68	0.753
ADL	0, 0, 1, 1	1.570	0.326	0.710	0.014	0.975	2.21	2.18	0.796
	1, 4, 4, 1			0.616	–0.036	1.024	2.55	2.02	0.847
	1, 5, 5, 1			0.621	–0.034	1.024	2.53	1.90	0.844
	2, 4, 4, 1			0.645	–0.008	0.981	2.43	1.98	0.832
	2, 5, 5, 1			0.635	–0.030	0.973	2.47	1.95	0.837
	2, 8, 6, 1			0.617	–0.048	0.975	2.54	1.89	0.846

[a] Bias, difference of means (laboratory minus predicated by NIRS); r^2, coefficient of determination for external validation; SD, standard deviation of the reference data; SEL, standard error of laboratory; SEP, standard error of performance; SEP/SEL, ratio of standard error of performance to standard error of laboratory.

the whole spectral region of 1100−2498 nm was applied to develop calibration equations.

Calibration and Validation. Obviously, the calibration and cross-validation statistics were affected by mathematical treatments (**Table 2**). It seemed that the second derivative of spectral data gave better results than the first derivative and the original spectra because the standard error of calibration (SEC) decreased and the coefficient of determination for calibration (R^2) increased, as did the standard error of cross-validation (SECV) and coefficient of determination for cross-validation (1-VR) (**Table 2**). For ADF, the calibration equation (R^2 and 1-VR, 0.93) was good enough if the original spectral data were used, but the R^2 increased 3% if the second derivative treatment was applied. However, for NDF, the best calibration equation came from the math treatment of 2, 4, 4, 1 (R^2, 0.846; 1-VR, 0.813); all other treatments gave lower R^2 and 1-VR (**Table 2**). For ADL, second-derivative treatment increased R^2 and 1-VR, but 2, 4, 4, 1 treatment gave the highest R^2 (0.897) and 1-VR (0.843). Generally, the equations from the second derivative produced lower SEC and SECV and higher R^2 and 1-VR values, especially for the mathematical treatment 2, 4, 4, 1. Other studies (*15*, *16*) obtained similar R^2 and 1-VR for ADF and ADL for pigeon pea and for cereal crops, respectively, but R^2 and 1-VR for NDF were much higher than the present results (**Table 2**). Using fecal NIR spectral data, the ADF and NDF of forage grasses could be predicted with acceptable accuracy (*37*), but the R^2 and 1-VR for ADF was smaller than the present result (**Table 2**). The calibration equation established to determine ADF of oilseed Brassica based on NIR spectra of either intact or ground seed was predicted less accurately than in this study (*24*).

The statistics of external validation (**Table 3**) were calculated using the equations developed from the different mathematical treatments.The statistical values of r^2 and SD/SEP in the external validation for the second derivative were still higher, whereas the SEP/SEL was lower, than first-derivative treatment and

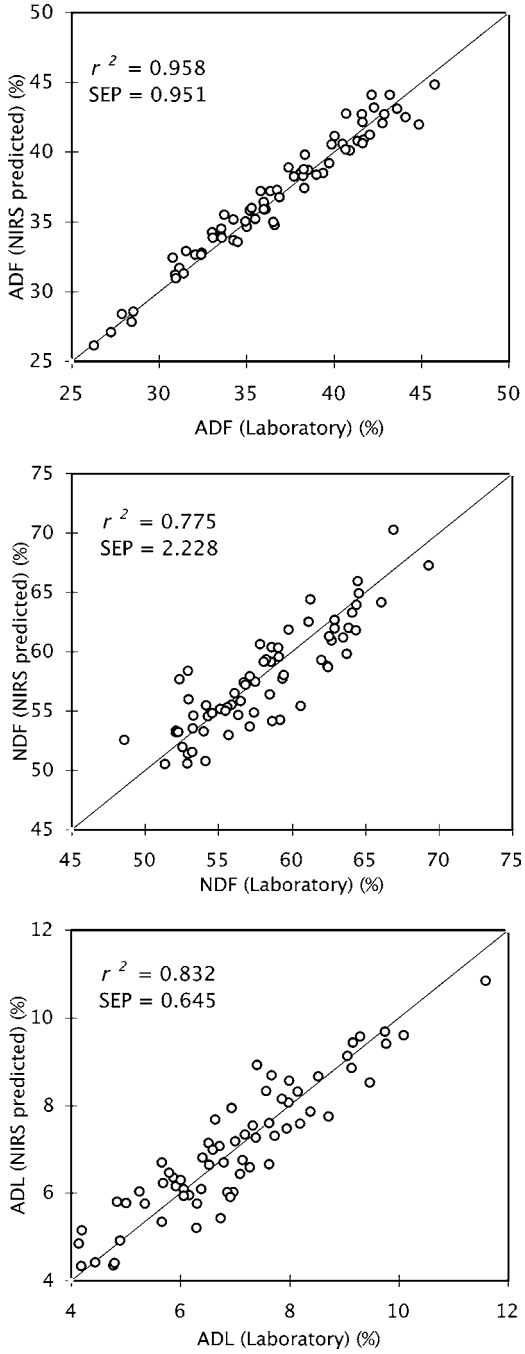

Figure 2. External validation scatter plot for ADF, NDF, and ADL contents (percent) with the 2, 4, 4, 1 mathematical treatment.

original spectral data. As could be expected from calibration (**Table 2**), the statistics of external validation confirmed that the ADF content could be accurately predicted by NIR because the r^2 was higher than 0.95 and the SD/SEP was larger than 4.5 with the first- and second-derivative treatments. For NDF, the highest r^2 (0.775) and SD/SEP (2.091) still came from 2, 4, 4,1 treatment. However, these statistics showed that the prediction was not so accurate, but the equation would still be useful to differentiate high and low NDF. For ADL, the equations established with mathematical treatments of 2, 4, 4, 1 and 2, 5, 5,1 gave similarly accurate predictions (r^2, 0.83; SD/SEP, 2.5). For all of the validation results, the bias was small and close to 0, and the slope was close to 1 (**Table 3**). The laboratory-measured ADF, NDF, and ADL versus predicted results obtained by the mathematical treatment 2, 4, 4, 1 (**Figure 2**) showed that the predictive values were close to the measured values. No independent external validation was carried out in other studies (*15*, *16*, *37*). Font et al. (*24*) carried out external validation for ADF of oilseed Brassica; even though the r^2 (0.83) and SD/SEP (2.40) were small, they believed that ADF of ground seed could be accurately predicted because the ratio of the range to SEP was larger than 10 (*24*). The present study gave the ratios of range to SEP of 19.5 for ADF and 12.4 for ADL, proving the predictions were accurate enough on this basis. However, the ratio of range to SEP was 9.4 for NDF, indicating a less favorable predictive ability. The entire set of samples was analyzed in triplicate (ADF) or in duplicate (NDF and ADL), giving standard error of laboratory (SEL) values of 0.753, 1.352, and 0.326% for ADF, NDF, and ADL, respectively (**Table 3**). The SEP/SEL is another useful statistic to evaluate the precision of an NIR equation. The SEP/SEL values of 1.3 for ADF, 1.7 for NDF, and 2 for ADL (**Table 3**) were indicative of high accuracy in the prediction abilities in the external set of samples.

In conclusion, ADF and ADL contents of rice stem materials can be predicted with sufficient accuracy by NIRS, facilitating its application to selection in early generations in rice-breeding programs. NDF can be predicted with less accuracy, but NIRS screening for NDF can still offer enough information to select or discard a breeding line.

ABBREVIATIONS USED

ADF, acid detergent fiber; ADL, acid detergent lignin; MPLS, modified partial least squares; NDF, neutral detergent fiber; NIRS, near-infrared spectroscopy; R^2, coefficient of determination for calibration; r^2, coefficient of determination for external validation; SD, standard deviation; SD/SEP, the ratio of standard deviation to standard error of performance; SEC, standard error of calibration; SECV, standard error of cross-validation; SEL, standard error of laboratory; SEP, standard error of performance; SNV-D, standard normal variate and detrend; SEP/SEL, the ratio of standard error of performance to standard error of laboratory; 1-VR, coefficient of determination for cross-validation.

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