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Prediction Chemical Composition and Alveograph Parameters on Wheat by Near-Infrared Transmittance Spectroscopy

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Moisture, protein, wet gluten, dry gluten, and alveograph parameters (W, P, and P/L) of whole wheat grown in different countries around the world were analyzed using near infrared (NIR) transmittance spectroscopy. Modified partial least squares on NIR spectra (850−1048.2 nm) were developed for each constituent or physical property. The best models were obtained for protein, moisture, wet gluten, and dry gluten with $r^2 = 0.99$, 0.99, 0.95, and 0.96, respectively. Initial alveograph NIR models proposed for all wheat samples did not perform well. However, when wheat samples were divided in two groups depending on W (deformation energy) values, NIR models were highly improved, showing enough prediction accuracy for screening wheat at the receiving stage at mills or elevators.

KEYWORDS: NIR; outlier; whole wheat; deformation energy; protein; gluten

INTRODUCTION

The quality of wheat can be defined for several parameters, including milling yield, weight, protein, moisture, gluten, enzyme activity, and rheological properties (alveograph, farinograph, and mixograph). Baking trials are still the only reliable method for determining the bread-making performance of wheat flour; however, analysis time is long. Gluten proteins are essential for bread production because elasticity and extensibility are considered important in the bread-making process. The alveograph is an instrument used for estimating important dough properties such as deformation energy (W), resistance of dough to deformation (P), and extensibility (L), allowing one to predict the baking performance of flours. In many countries, the end use quality of wheat flour is determined by the alveograph method; however, this technique is too time consuming to make decisions at the receiving stage. The near infrared (NIR) method has already been accepted for milling, as it is capable of generating nearly instantaneous results for several whole wheat and wheat flour quality parameters. The use of NIR spectroscopy to determine the protein and moisture content of both wheat and flour is now routine in flour mills worldwide. It is used for testing each delivery of wheat in order to make decisions about acceptance and/or classification into different silos. Thus, NIR would increased the speed and efficiency of wheat analysis while substantially reducing the cost.

Recent advances in NIR spectroscopy have enabled its extension to the prediction of functionality, as well as composition, in wheat (*1*). NIR spectroscopy is widely used to determine protein and moisture content of wheat and wheat flour (*2*, *3*). A number of attempts have been made to obtain a measure of their functionality using this technique, including wheat classes (*4*, *5*), color classes (*6*, *7*), insect damage (*8*, *9*), and physical dough properties (*10*, *11*). Although some rheological calibrations have shown potential, their accuracy has often been insufficient for commercial use. Nearly all research on NIR spectroscopy has been carried out using the reflectance technique, whereas few studies has been made using transmittance procedures. The use of NIR transmittance spectroscopy on whole wheat to measure alveograph parameters has not been studied. This paper describes attempts to predict wheat functionality parameters by NIR transmittance technology using whole wheat grain. The main objective of the present study was to develop robust NIR calibrations for determining whole wheat quality parameters.

MATERIALS AND METHODS

Wheat Samples. Wheat varieties from several countries were selected for NIR transmittance prediction. The varieties selected were from the U.S.A. (hard red winter, soft red winter, and soft white winter), Canada (Canadian western red spring), the European community (soisson, galibier, courtot, anza, bolero, chamorro, marius, commercial French milling wheat, cajeme, yécora, caton, and apache), and the U.K. (commercial U.K. milling wheat).

Milling Process. Clarified wheat grain (6000 g) was conditioned to a moisture level between 14.5 and 15.5% depending on the kernel hardness. Grains were milled on a Chopin Moulin CD mill. The milling process was performed in order to obtain extraction rates of approximately 60−65%.

Analytical Methods. Moisture, protein, wet gluten (WG), and dry gluten (DG) were determined according to the approved AACC methods (*12*). Dough rheological properties were performed using the alveograph test according to the approved AACC methods (*12*). The alveograph parameters registered were P, the configuration ratio (P/L), and W. WG and DG were reported on a 14% moisture basis.

NIR Hardware. A scanning monochromator Infratec 1241 Grain Analyzer (Foss Tecator) was used to measure NIR transmittance spectra from 850 to 1048.2 nm every 2 nm. The NIR spectra were collected from whole wheat samples and measured two times for each sample. The average spectrum of the duplicates was used for calibration, cross-validation, and validation.

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Table 1. Summary of Attributes of Wheat Samples

attribute	*n*[a]	mean	SD	range
protein (%)	443	13.2	1.63	10.38–18.03
moisture (%)	443	14.12	1.89	9.32–17.58
WG (%, 14% mb)	443	24.01	4.66	15.6–39.3
DG (%, 14% mb)	443	8.53	1.63	5.8–13.67
P (mm)	600	59	18.3	23–116
P/L	600	0.68	0.32	0.17–1.7
W (10^{-4} J)	600	204	79.7	69–427

[a] *n*, number of wheat samples selected for each attribute.

Table 2. Means, Ranges, and SDs of Wheat Samples of Calibration and Validation Sets

attribute		no. term[a]	*n*[b]	mean	SD	range
protein (%)	Cal	10	334	13.16	1.61	10.38–18.03
	Val		109	13.34	1.68	11.31–17.97
moisture (%)	Cal	11	334	14.11	1.89	9.32–17.58
	Val		109	13.34	1.68	10.72–17.45
WG (%, 14% mb)	Cal	7	334	23.88	4.68	15.6–38.86
	Val		109	24.42	4.59	18.1–39.3
DG (%, 14% mb)	Cal	8	334	8.48	1.62	5.8–13.5
	Val		109	8.68	1.65	6.21–13.67
P (mm)	Cal	12	500	56	18.6	23–116
	Val		100	58	19.4	30–107
P/L	Cal	13	500	0.7	0.36	0.19–1.71
	Val		100	0.71	0.37	0.2–1.5
W (10^{-4} J)	Cal	10	500	220	80	69–427
	Val		100	218	83	80–390

[a] No. term, number of terms determined by cross-validation of calibration samples. [b] *n*, number of wheat samples selected for each attribute.

Calibration and Validation. A WinISI III (ver. 1.50e) software program was employed for spectra data analysis and development chemometric models. Calibrations were performed from whole wheat spectra.

Prior to calibration, log(1/*T*) spectra were transformed using the standard normal variant and detrending (SNV + D) procedures and first derivate mathematical treatment (derivative = 1, gap = 4, smooth = 4, second smooth = 1). Calibration was performed using modified partial least squares regression by WinISI. Cross-validation was applied to optimize calibration models and detect outliers. The outliers with a large residual (*T* value > 2.5 or *H* value > 10) were removed, and the calibration was performed again. The cycle of cross-validation to eliminate outliers was done a maximum of two times. The optimal number of terms was determined by cross-validation of calibration samples.

The samples used in validation were selected from the total population to represent the full range of composition. Following completion of the calibration, the model was validated using an independent set of wheat samples. The assignment of wheat samples was grouped to ensure that both high and low values were included in both tests. The assignment to one or the other of the sets was at random. The statistics of the most interest were the following: standard error of calibration (SEC), standard error of cross-validation (SECV), standard error of performance (SEP), coefficient of determination (R^2), and linear correlation coefficient (r^2) between reference methods and estimated by prediction models.

RESULTS AND DISCUSSION

The means, ranges, and standard deviations (SDs) of wheat quality parameters are summarized in **Table 1**. Among the samples analyzed for the different parameters, some of them were selected for calibration by WinISI software, and the remaining samples were used for the validation set (**Table 2**).

Table 3. Results of NIR Calibration and Validation Sets for Wheat Samples

	calibration			validation	
attribute	SEC	SECV	R^2	SEP	r^2
protein (%)	0.12	0.13	0.99	0.16	0.99
moisture (%)	0.11	0.11	0.99	0.14	0.99
WG (%, 14% mb)	1.01	0.95	0.95	1.00	0.95
DG (%, 14% mb)	0.29	0.29	0.96	0.32	0.97
P (mm)	9.2	9.2	0.72	12	0.67
P/L	0.15	0.16	0.61	0.21	0.54
W (10^{-4} J)	30	31	0.85	41	0.84

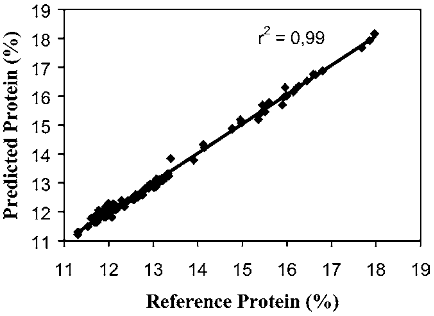

Figure 1. Comparison of protein content determined by prediction model and by reference method.

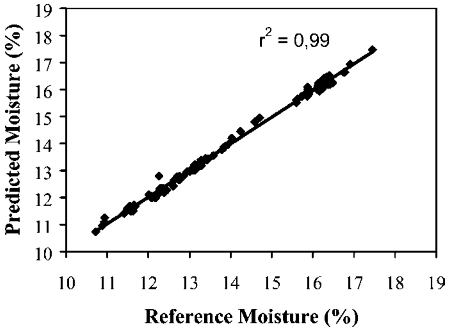

Figure 2. Comparison of moisture content determined by prediction model and by reference method.

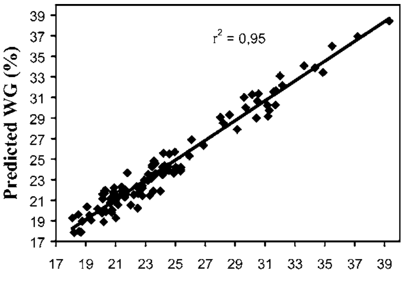

Figure 3. Comparison of WG content determined by prediction model and by reference method.

Chemical Composition. The statistical evaluation of calibration and validation of wheat parameters is summarized in **Table 3**. The models' performances for protein and moisture were excellent, showing r^2 = 0.99 and SEP = 0.16 (**Figure 1**) and r^2 = 0.99 and SEP = 0.14 (**Figure 2**), respectively.

WG and DG show good validation performance with r^2 = 0.95 and SEP = 1.00 (**Figure 3**) and r^2 = 0.97 and SEP = 0.32 (**Figure 4**), respectively. It was reasonable to find good NIR calibration and validation sets for WG and DG because of the strong correlation with protein content.

Alveograph Parameters. The NIR models for calibration and validation for each physical dough properties are summarized in **Table 3**. Alveograph NIR models for P and P/L

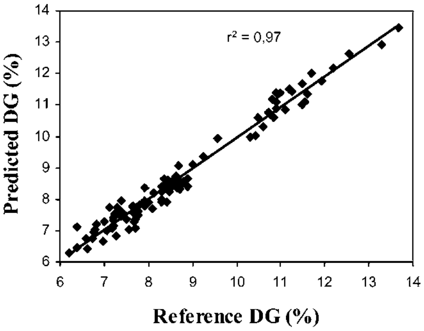

Figure 4. Comparison of DG content determined by prediction model and by reference method.

show low validation performance with $r^2 = 0.67$ and SEP = 12 and $r^2 = 0.54$ and SEP = 0.21, respectively. The W NIR model showed a relatively high correlation, $r^2 = 0.84$; however, the SEP value (41) was higher for screening purposes (**Figure 5**).

The scatter plot for the W for all samples together showed two groups of wheat (**Figure 5**). As shown in the graphic, two differentiable groups of samples appeared. One of the groups had low values, and the other samples covered a much wider range. The SEP value of 41 was very high when we intended to predict W values between 40 and 140. For higher W values, however, these SEP values could be acceptable. For this reason and to minimize the SEP value, two sample groups were selected, one with low W values and one with high ones. This separation of samples allowed me to obtain higher *r* values and smaller SEP values, giving greater precision in measurements.

To obtain better NIR calibration and validation sets for alveograph parameters, wheat samples were divided in two groups depending on the W value. It is generally well-accepted that the more the protein content, the higher the W value. A simple linear Person correlation overall sample set showed high correlation between W and protein, with $r^2 = 0.86$. However, some samples from the sample set showed high protein content while W values were lower than expected for protein content. Wheat samples with the same protein content showed large differences in the W value. Thus, because of this fact, the wheat varieties selected for each group's purposes were the following: (i) weak wheat: soft red winter, soft white winter, marius, anza, chamorro, bolero, and commercial U.K. milling wheat; and medium−strong wheat: hard red winter, galibier, cajeme, yécora, soisson, commercial French milling wheat, Canadian western red spring, and apache.

Table 4. Means, Ranges, and SDs of Wheat Groups

	weak wheat			medium–strong wheat		
attribute	mean	SD	range	mean	SD	range
P (mm)	34	6.06	23–52	64	14	37–116
P/L	0.36	0.16	0.19–0.93	0.74	0.24	0.36–1.71
W (10^{-4} J)	98	10.83	69–137	228	71.1	140–427

Table 5. Means, Ranges, and SD of Wheat Groups of Validation and Calibration Sets

attribute		no. term[a]	*n*[b]	mean	SD	range
			weak wheat			
P (mm)	Cal	11	180	34	6.06	23–52
	Val		64	34	5.85	23–50
P/L	Cal	11	180	0.38	0.15	0.19–0.81
	Val		64	0.36	0.16	0.19–0.93
W (10^{-4} J)	Cal	9	180	98.8	10.8	69–137
	Val		64	99.8	10.4	77–125
			medium–strong wheat			
P (mm)	Cal	9	236	64.8	14.8	37–116
	Val		120	66.0	14.11	46–116
P/L	Cal	12	236	0.73	0.24	0.36–1.71
	Val		120	0.78	0.25	0.37–1.64
W (10^{-4} J)	Cal	9	236	227	68.9	140–427
	Val		120	238	82.2	142–417

[a] No. term, number of terms determined by cross-validation of calibration samples. [b] *n*, number of wheat samples selected for each attribute.

The range selected for the W value was as follows: weak wheat (69−137 × 10^{-4} J) and medium−strong wheat (140−427). The mean, range, and SD of them are summarized in **Table 4**. The mean, SD, and range of validation and calibration of alveograph parameters are shown in **Table 5**.

With this selection, NIR calibration and validation models for all parameters were improved (**Table 6**). Among the models for alveograph parameters, only the W demonstrated a predictive ability. The model developed for W of medium−strong wheat was more accurate than the model for W of weak wheat, showing $r^2 = 0.95$ and SEP = 24 (**Figure 6**) and with $r^2 = 0.87$ and SEP = 6.1 (**Figure 7**) for the validation sets, respectively. The SEP values obtained for each one were accurate enough for screening purposes at the receiving stage at mills or elevators, allowing one to make decisions very quickly.

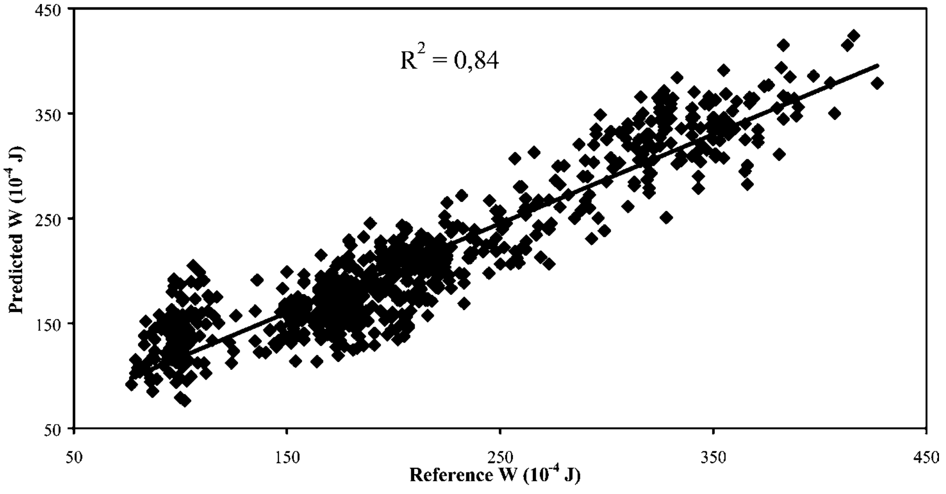

Figure 5. Comparison of W for all wheat samples by prediction model and by reference method.

Table 6. Results of NIR Calibration and Validation Sets for Wheat Groups

	calibration			validation	
attribute	SEC	SECV	R^2	SEP	r^2
		weak wheat			
P (mm)	3	3.7	0.79	4.8	0.80
P/L	0.05	0.06	0.68	0.08	0.76
W (10^{-4} J)	4.7	4.7	0.81	6.1	0.87
		medium−strong wheat			
P (mm)	5.47	5.43	0.847	6.7	0.86
P/L	0.12	0.12	0.71	0.16	0.66
W (10^{-4} J)	18.25	18.73	0.93	24	0.95

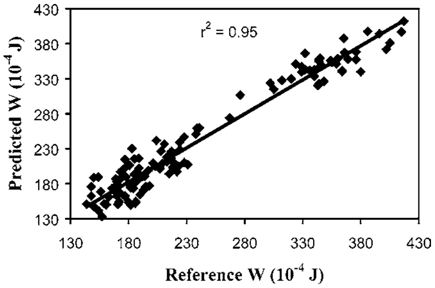

Figure 6. Comparison of W for medium−strong wheat determined by prediction model and by reference method.

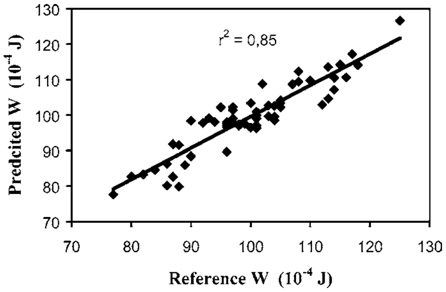

Figure 7. Comparison of W for weak wheat determined by prediction model and by reference method.

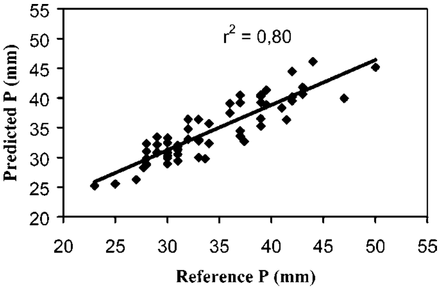

Figure 8. Comparison of resistance of dough to deformation for weak wheat determined by prediction model and by reference method.

The models developed for P of weak and medium−strong wheat showed a relatively high performance with $r^2 = 0.80$ and SEP = 4.8 (**Figure 7**) and with $r^2 = 0.86$ and SEP = 6.7 (**Figure 8**).

The worst NIR models validation was obtained for P/L of weak wheat with $r^2 = 0.76$ and SEP = 0.08 (**Figure 9**) and medium−strong wheat with $r^2 = 0.66$ and SEP = 0.16 (**Figure 10**).

CONCLUSION

Until recently, criteria for control at the receiving stage in the milling industry were variety, moisture, and protein. Better calibration techniques for gluten and other parameters have gradually emerged and have been incorporated as controls upon delivery. However, determining the W of whole wheat requires a good deal of time. It must be tempered and milled; then, the alveographic test of the flour must be done. Wet chemistry entails too much time to make a quick decision on whether to reject or reclassify the wheat into a specific silo.

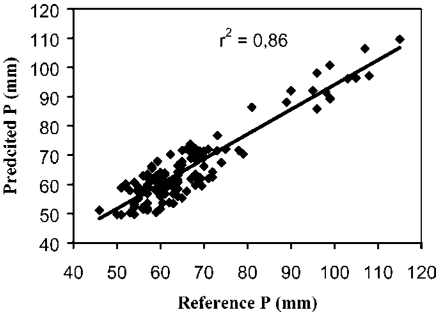

Figure 9. Comparison of P for medium−strong wheat determined by prediction model and by reference method.

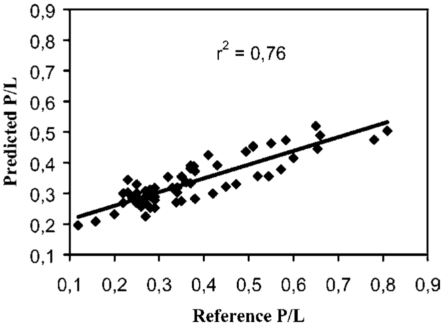

Figure 10. Comparison of P/L for weak wheat determined by prediction model and by reference method.

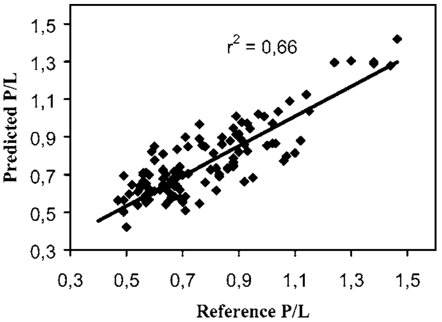

Figure 11. Comparison of P/L for medium−strong wheat determined by prediction model and by reference method.

It was investigated whether NIR transmittance could be applied to quality evaluation of whole wheat from different origins. The results indicate that this technique could be considered a valuable tool for whole wheat quality prediction, demonstrating a high level of accuracy for protein, moisture, and WG and DG contents. This method provides repeatable measurements, which may be related to a number of traditional measures of wheat quality. Physical dough characteristics (P and W) can be predicted with reasonable success when the wheat variety sample is applied to an specific calibration model. Thus, from a single NIR test, several wheat quality parameters could be obtained for screening purposes. NIR models from whole wheat spectra could be very helpful for breeding programs and industry communities where there is a need to analyze many wheat samples in a short time and make decisions very quickly. More research is needed to confirm these results.

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