Prediction of Wheat and Flour Zeleny Sedimentation Value Using NIR Technique

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Abstract

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Analytical quality parameters of wheat flour prepared from variety and commercial wheat samples (wheat harvest 1998, 1999, 2000 and 2001) were assessed by means of filter spectrograph Inframatic 8620 ASH (moisture and protein content) and Sedi-tester (Zeleny sedimentation value). The spectra of all samples were measured on spectrograph NIRSystem 6500. Calibration equations with cross and independent validation for all analytical characteristics were computed by NIR Software ISI Present WINISI II using MPLS and PLS method. The quality of prediction was evaluated by SEP and *r* parameters between the measured and the predicted values from cross and independent validation. In case of Inframatic 8620 ASH, validation was realised by NIRPRG software. A statistically significant dependence between the predicted and the measured values of protein content and Zeleny sedimentation (with probability $P < 0.01$) was determined in both variety and commercial flour sets in the case of cross and independent validation. Better accuracy of prediction was found with NIRSystem 6500. Both important parameters of wheat were successfully predicted by independent validation with nearly the same accuracy.

Keywords: wheat; flour; Zeleny sedimentation value; NIRSystem 6500; Inframatic 8620 ASH; prediction of quality

Wheat and flour quality is expressed by a variety of chemical and physical properties of dough, none of which serves as adequate by itself or is independent of others variables (PYLER 1988). According to FINNEY (1978) "a flour of good quality for breadmaking should have high water absorption, a medium to medium – long mixing requirement, satisfactory mixing tolerance, and bread volume potential (considering protein content), and should yield a loaf with good internal grain and colour". TIPPLES *et al.* (1982) identify the "ideal" bread flour as one that produces good bread over a wide range of processing conditions, that yields doughs with well-balanced handling properties and does not have long mixing requirements.

Wheat's breadmaking potential is derived largely from the quantity and quality of its protein. Protein quantity is influenced by environmental factors, while the quality of the protein is genetically determined. In wheat varieties that are grown under comparable environmental conditions, a high quality wheat will produce good bread over a fairly broad range of protein levels. A poor quality wheat will yield relatively low quality bread even at high protein contents.

When flour and water are mixed into dough and this is kneaded thoroughly under water either by hand or by machine, a cohesive, extensible and rubbery mass is obtained that consists principally of protein and water. When this so-called "crude gluten" is treated with 70% alcohol, the gliadin fraction dissolves or disperses and can be separated in fairly pure form. The remainder of the protein consists essentially of glutenin which is soluble in dilute acid or alkali solution. A number of investigators attempted to determine whether an optimum ratio exists of these wheat proteins with regard to the baking strength. Recent attempts to establish correlations between gluten fractions have shown that loaf volume is controlled by the gliadin fraction and the glutenin fraction is responsible for dough mixing time and dough development (Pyler 1988).

Hydration of gluten proteins, i.e. the ability to absorb water, is for practical purposes synonymous with protein swelling. The capacity of gluten proteins to swell in dilute acids has long been used as a test for the flour quality determination. The application of testing methods based on this principle has shown that a satisfactory correlation exists between the swelling value of protein and the volume of loaf obtained from the corresponding flour. Since the swelling value is a measure of the protein quality, the protein content of the flour must also be taken into account in predicting flour behaviour as the protein level exerts an effect on the ultimate loaf volume (Rubenthaler & Pomeranz 1987).

The sedimentation value according to Zeleny (Zeleny value) describes the degree of sedimentation of flour suspended in a lactic acid solution during a standard time interval and this is taken as a measure of the baking quality. Swelling of the gluten fraction of flour in lactic acid solution affects the rate of sedimentation of a flour suspension. Both a higher gluten content and a better gluten quality give rise to slower sedimentation and higher Zeleny test values. The sedimentation value of flour depends on the wheat protein composition and is mostly correlated to the protein content, the wheat hardness, and the volume of pan and hearth loaves. A stronger correlation between loaf volume and Zeleny sedimentation volume compared to SDS sedimentation volume could be due to the protein content influencing both the volume and Zeleny value (Shewry & Tatham 2000).

Near-infrared spectroscopy (NIRS) is widely used in the wheat milling industry for measuring moisture and protein contents. NIRS was also used to detect wheat attributes such as class, colour, damage, aflatoxin, and fumonisin (Dowell *et al.* 2002). Suzuki *et al.* (1986) applied NIRS to study bread constituents such as moisture, protein, total sugar, and crude fat. NIRS applications have typically been directed at rapid analysis for quality control.

Previous research has shown that NIRS has the potential to measure Zeleny sedimentation value of wheat and flour. The objective of this study was to investigate the capability of NIRS for measuring wheat and flour quality by the sedimentation value according to Zeleny, and to compare the applicability of spectrometer NIRSystem 6500 with Inframatic 8620 for this reason.

MATERIAL AND METHODS

Samples. The total of 318 wheat samples comprised four groups. Variety flours (39 harvest 1998 and 75 harvest 1999) were obtained from the cultivated varieties from the Central Institute for Supervising and Testing in Agriculture. The others named commercial flours (100 and 104 samples) came from food wheat supplied to industrial mills (harvest 2000 and 2001). The fine (straight-grade) flours were obtained from experimental mills.

References analysis. Basic analytical properties of wheat flour (moisture, protein content) were determined by the filter spectrometer Inframatic 8620 ASH (Perten, Sweden). Flour samples for Zeleny sedimentation value were prepared at experimental mill FQC 109 (Hungary) and the proper measurement was carried out at Sedi-tester (Czech Republic) according to ISO 5529. The results of analysis were expressed by average, minimum and maximum values and by standard deviation and variation coefficients of each sample set.

NIR apparatus. Spectra were obtained by the wavelength scanning instrument NIRS 6500 (NIRSystems, Inc.) using a small ring cup. A scanning range from 400 to 2500 nm and wavelength increments of 2 nm were used. Diffuse reflectance was recorded as log $1/R$. Each sample was scanned twice and the average spectra were used for calibration.

Wheat samples from harvest 2001 were measured – after grinding at experimental mill LM 3100 (Perten, Sweden) – at twenty filter spectrometer Inframatic 8620 ASH (Perten, Sweden). For calibration, the following equation was used:

$$\text{Zeleny value (ml)} = \text{BIAS} - rk \times \log 1/R\ (2310\ \text{nm}) + \\ + rk \times \log 1/R\ (2180\ \text{nm}) - rk \times \log 1/R)\ (1680\ \text{nm})$$

where: BIAS – spectrometer constant
rk – regression coefficient
R (2310 nm) – reflectance for protein
R (2180 nm) – reflectance for water
R (1680 nm) – reflectance for granulation

Validations were realised by NIRPRG software (Perten Inst.)

Statistics. NIR Software ISI Present WINISI II (Infrasoft Int.) was used to evaluate the data and to develop chemometric models. Scatter correction was performed by Standard Normal Variate Transformation (SNV) and then transformed with the first derivative which was calculated using treatment 1,4,4,1 and 1,8,8,1. Calibration was carried out by Modified Partial Least Square (MPLS), and Partial Least Square (PLS) regression and correlation coefficient (r) was determined. No samples were dismissed on account of their higher deviations. The calibration of wheat flour properties was verified by cross validation (the number of selected segments was equal to the number samples of each set) and independent validation. The selection of optimum number of PLS terms for the calibration was based on the standard error of calibration (SEC) or standard error of prediction (SEP) which should be minimised. Two statistical parameters (SEP or SEC and r) were used to determine the calibration equation.

RESULTS AND DISCUSSION

Analytical characteristics. Variety flour quality from harvest 1998 and 1999 satisfied the Czech standard for mill products of fine type (Table 1). The ranges of the analytical data for all 114 samples were: protein 10.8–15.8%, Zeleny value 17–45 ml. The quality of flour proteins seems to be comparable in both sets. Commercial flour quality from harvest 2000 and 2001 differed extremely both in the protein content and Zeleny value. The ranges were from 11.0 to 15.0% and from 7.8 to 13.1% for protein, from 19 to 39 ml and from 20 to 66 ml for Zeleny sedimentation value of the sets tested. Flour samples from harvest 2001 were defined by a low protein content and its good quality; for standard bakery use, however, fine flours were regularly fortified with dry gluten.

Correlation between the protein content and Zeleny sedimentation value. The statistical relationships between the protein content of wheat and flour and the Zeleny sedimentation volume were found positive and very strong (with $P < 0.01$).

Table 1. Flour analytical properties

Parameter		Range				
		average	min.	max.	S.D.	v.c. (%)
Variety flours 1998						
Moisture	%	13.9	13.3	14.5	0.3	2.4
Protein	%	13.7	12.0	15.8	0.9	6.5
Zeleny sedimentation value	ml	30	17	40	5	18.2
Variety flours 1999						
Moisture	%	14.1	13.5	14.6	0.2	1.7
Protein	%	12.8	10.8	15.7	0.7	5.6
Zeleny sedimentation value	ml	33	24	45	5	14.2
Commercial flours 2000						
Moisture	%	13.3	13.0	14.1	0.2	1.6
Protein	%	12.8	11.0	15.0	0.9	7.3
Zeleny sedimentation value	ml	31	19	39	5	16.4
Commercial flours 2001						
Moisture	%	14.1	11.7	14.8	0.6	4.2
Protein	%	10.4	7.8	13.1	1.3	12.5
Zeleny sedimentation value	ml	42	20	66	10	23.8

S.D. – standard deviation; v.c. (%) – variation coefficient

Zeleny values were better correlated with wheat protein content ($r = 0.8748$) than with flour protein content ($r = 0.8407$). The difference could be due to the accuracy of measurement of wheat and flour protein content by Inframatic 8620 ASH.

Prediction according cross validation. Prediction of wheat properties from four harvests are shown in Table 2. For the calibration, all single parameters were found significant on 99% statistical level – in the variety sets nearly the same accuracy was found of the protein content (SEC = 0.11 and 0.30%) and Zeleny value (SEC = 3.9 and 3.7 ml). In the case of commercial flour, the accuracy of Zeleny value was more different in both sets. Comparable results were found with cross validation in all four sets of samples. Correlation coefficients were computed logically lower at 99% significant level and their values for Zeleny sedimentation value were lower as compared with those for the protein content.

Prediction according independent validation. The correlation coefficient (r) and standard error of independent prediction (SEP) for analytical parameters are shown in Table 3. The protein contents were independently predicted in all sets, similarly as recently found by Delwiche and Weaver (1994) and Delwiche *et al.* (1998). These authors gave SEP in the range of 0.126% where Dumas method was used as reference. Our results were not computed so accurately (SEP in the range 0.13–0.30%). The accuracy of prediction depends on the range of the flour quality parameters used as many works (Rubenthaler & Pomeranz 1987; Hrušková *et al.* 2001) confirmed. The same result was found in comparison of SEP values of our sets tested. Ze-

Table 2. Calibration and cross validation of flour characteristics

Parameter	n	Calibration			Cross validation		
		term	SEC	r	segment	SEP	r
Variety flours 1998							
Moisture	39	4	0.11	**0.994**	39	0.14	**0.908**
Protein	39	4	0.10	**0.983**	39	0.20	**0.985**
Zeleny sedimentation value	39	2	3.90	**0.687**	39	4.30	**0.625**
				$r_{(P<0.01)}$ = **0.403**		$r_{(P<0.05)}$ = *0.317*	
Variety flours 1999							
Moisture	75	4	0.08	**0.942**	75	0.10	**0.923**
Protein	75	4	0.30	**0.960**	75	0.30	**0.848**
Zeleny sedimentation value	75	3	3.70	**0.631**	75	4.00	**0.534**
				$r_{(P<0.01)}$ = **0.296**		$r_{(P<0.05)}$ = *0.228*	
Commercial flours 2000							
Moisture	100	4	0.18	**0.877**	100	0.19	**0.861**
Protein	100	4	0.21	**0.942**	100	0.26	**0.913**
Zeleny sedimentation value	100	3	3.44	**0.418**	100	3.70	**0.334**
				$r_{(P<0.01)}$ = **0.257**		$r_{(P<0.05)}$ = *0.197*	
Commercial flours 2001							
Moisture	104	4	0.11	**0.968**	104	0.12	**0.961**
Protein	104	4	0.12	**0.990**	104	0.13	**0.989**
Zeleny sedimentation value	104	3	4.88	**0.749**	104	5.32	**0.706**
				$r_{(P<0.01)}$ = **0.254**		$r_{(P<0.05)}$ = *0.195*	

Table 3. Independent validation of calibration equations

Parameter	*n*	Calibration			Independent validation		
		term	SEC	*r*	segment	SEP	*r*
Flours 1998, 1999							
Moisture	75	4	0.08	**0.942**	39	0.20	**0.863**
Protein	75	4	0.30	**0.960**	39	0.40	**0.963**
Zeleny sedimentation value	75	3	3.70	**0.631**	39	4.60	**0.548**
				$r_{(P<0.01)}$ = **0.296**		$r_{(P<0.05)}$ = *0.317*	
Flours 2000							
Moisture	70	4	0.18	**0.912**	30	0.19	**0.642**
Protein	70	4	0.15	**0.965**	30	0.31	**0.928**
Zeleny sedimentation value	70	3	2.61	**0.591**	30	1.93	**0.687**
				$r_{(P<0.01)}$ = **0.306**		$r_{(P<0.05)}$ = *0.236*	
Flours 2001							
Moisture	104	4	0.11	**0.968**	26	0.11	**0.983**
Protein	104	4	0.12	**0.990**	26	0.13	**0.992**
Zeleny sedimentation value	104	3	4.88	**0.749**	26	4.05	**0.833**
				$r_{(P<0.01)}$ = **0.254**		$r_{(P<0.05)}$ = *0.195*	

leny sedimentation value was predicted with the accuracy 3.7–5.3 ml at statistical level 99%. The results point to the wheat quality effect on NIR spectra and could be improved by adjustment on a year-to-year basis. Validation of Zeleny value from measured and computed data obtained at Inframatic 8620 for the value range of 29–64 ml was showed SEP 5 ml, r = 0.872. The accuracy from both NIR spectrometers was considered adequate for a rapid screening in wheat harvest.

Conclusions

NIR spectroscopy was used for the quantitative screening of moisture, ash, wet gluten, and protein content of wheat and flour at mills and bakeries. For qualitative properties of wheat (water absorption, Zeleny sedimentation value), greater differences were mostly found between the measured and the computed data. Zeleny sedimentation value from 318 Czech wheat samples from four harvests (1998 to 2001) could be predicted with slightly lower accuracy as a reference method. The accuracy of prediction was found to be higher in the case of NIRSystem 6500 in comparison to Inframatic 8620 ASH. The results point to an important effect of the wheat quality on NIR spectra. The accuracy of prediction could be improved on a year-to-year basis.

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Souhrn

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Analytické vlastnosti pšeničné mouky vyrobené z odrůd potravinářské pšenice – 114 vzorků (sklizeň 1998 a 1999) a 204 vzorků (sklizeň 2000 a 2001) – byly zjištěny pomocí filtrového spektroskopu Inframatic 8620 (vlhkost, obsah bílkovin) a Sedi-testeru ZZN Strakonice (Zelenyho test). Na spektrofotometru NIRSystem 6500 byla u všech vzorků naměřena NIR spektra. Pro analytické parametry byla programem NIR Software ISI Present WINISI II metodami MPLS a PLS provedena kalibrace, křížová a nezávislá validace. Kvalita předpovědi byla posuzována podle hodnot směrodatné odchylky predikce (SEP) a korelačního koeficientu (*r*) z výsledků křížové a nezávislé validace. Statisticky významná závislost mezi předpověděnými a naměřenými hodnotami s pravděpodobností vyšší než 99 % byla zjištěna u všech sledovaných ukazatelů při cross i nezávislé validaci na disperzním spektrometru NIRSystem 6500. Nižší přesnost stanovení Zelenyho testu po kalibraci přístroje Inframatic 8620 ASH ještě vyhovuje pro screeningové určení jakosti potravinářské pšenice při nákupu, kde je preferovaná rychlost měření.

Klíčová slova: pšenice; pšeničná mouka; NIRSystem 6500; Inframatic 8620 ASH; predikce kvality

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