# NEAR-INFRARED METHOD FOR PREDICTING OF BAKERY VARIETY TRAITS

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Prediction of some technological traits by FT-NIR spectroscopy was examined in 151 wheat flour samples. The flours included three groups of samples: 131 calibration samples from variety and commercial wheats (crop years 2002–2004) and a validation set – 20 consumer flours (made in 2004 and 2005). Samples were assessed by protein content, Zeleny sedimentation, fermentographic gas volume, OTG volume and specific loaf volume, which were selected from approximately 40 measured traits. NIR spectra were measured on the spectrometer Bruker IFS66v/S equipped with a Ge/Sn detector and a KBr beamsplitter. Calibration models with cross and independent validation for all mentioned characteristics were computed by force of TQ Analyst using PLS regression. A statistically significant correlation coefficients ($\alpha = 0.01$) were established for all characteristics in case of cross-validation: $r = 0.877, 0.501, 0.762, 0.693, 0.769$ for protein, Zeleny sedimentation, gas volume, OTG volume and specific loaf volume, respectively. Independent validation suggested some possibility of prediction (besides protein and Zeleny sedimentation) for specific loaf volume volume ($r = 0.553$).

FT-NIR; wheat; flour; rheology

## INTRODUCTION

The miller is the first user who is affected by the quality of wheat. It is estimated that 25% of flour quality is determined by the milling technology, mill adjustment, and conditions in the mill, and 75% by the quality of wheat. One of the major contributors to variance in quality is the wheat variety (Petr, 2001). Wheat is cultivated on all continents except Antarctica, and about 1,000 wheat varieties are of commercial significance (Posner, Hibbs, 2005). Czech survey of cultivars (Summary of varieties for 2004, 2005) contains 24 winter and 11 spring wheat varieties divided into several quality classes.

The component in wheat that makes the greatest contributions to the typical flour properties is the protein. Their amount and quality according to Zeleny test are the first sorting traits for wheat quality evaluation. Zeleny sedimentation value (ISO 5529) is not a chemical but empirical method. Wheat flour is shaken with a specified solutions and then allowed to settle. After a standard time, the height of sediment is measured. Generally, Zeleny values lower than 20 ml correspond to feed-quality wheat, and those higher than 40 ml indicate flour of high baking quality. During mixing of flour with water, a protein complex referred to as gluten is formed with gliadins and glutenins as the major components. The present view of the role of these proteins in breadmaking is that the gliadins are more or less responsible for the viscous and extensibility properties, the glutenins for the elastic properties of dough (Sluimer, 2005). Rheological characteristics such as elasticity, viscosity and extensibility and their changes during fermentation are important for bakery industry in prediction of the processing parameters of dough and quality of end products. For quality control of wheat flour, many tests are used to predict baking performance. Fermentation is an important step for the creation of bread volume and crumb grain. Oven spring is the first stage of dough baking and can be simulated in the oil bath of the oven rise recorder. To assess fermented dough properties (the gas volume, the retention capacity) and its behavior during oven spring stage (the dough and bread volume, the oven rise), several rheological apparatuses were developed. Common used are a fermentograph and an oven spring apparatus Brabender. The formula of dough also influences the properties of leavened dough. Full-bread-formula is usually used for fermented dough testing and standard baking test, which can simulate the real bakery process (Švec, Hrušková, 2004).

Because the rheological measurements require a lot of time and equipment to be performed, a fast and reliable test is desired. This problem can be solved by the NIR spectroscopy (Williams, Norris, 2001), which is almost well-established to control basic traits of wheat and flour. Nowadays, NIR is widely applied to the measurement of cereal quality and cereal product composition. NIR filter instruments (such as Inframatic 8600, Perten Inst., Sweden) or dispersion-based ones (such as NIRSystem 6500, Foss NIRSystems, Inc., USA) enable rapid assessment of protein, wet gluten, moisture, ash and with lower reliability also determination of Zeleny sedimentation and water absorption.

However, prediction of dough properties by NIR spectra analysis is influenced by many factors, especially errors of reference methods and results dependence on the protein content of tested flours. Reliability of computed characteristics of dough varies according to calibration sample set, extent and quality range of flour parameters (Hrušková et al., 2005a, b, c, d).

Having a few advantages, FT-NIR spectrometers represent another alternative to the above ones. Missbach (1998) has been concerned with their use to assess flour quality in comparison to traditional one. The potential of a partial least squares regression model of multiplicative scatter correction Fourier-transform NIR data as a rapid method for determining important milling and baking properties of wheat flours was examined by Sorvaniemi et al. (1993).

The aim of this work was to create calibration models by FT-NIR technique for five important technological traits of wheat flours and their validation by means of independent set of samples.

## MATERIALS AND METHODS

### Wheat and flour samples

The material used in this work contained wheat flour (fine, bright) made of bakery varieties and commercial wheats and also flours for home consumption. 151 samples including both Czech and international varieties and commercial wheats consisted of four subsets. All wheats were milled at standard conditions on laboratory mill CD1-auto (Chopin, France).

Subset 1 (Selgen Stupice, crop years 2002–2004) included the Czech varieties, 11 winter ones (Alana, Ebi, Meritto, Mladka, Rheia, Samanta, Saskia, Simila, Sulamit, Svitava, Vlasta) and 6 spring ones (Aranka, Leguan, Saxana, Sirael, Vinjet, Zuzana). Wheat samples were grown under the same conditions.

Subset 2 (RICP Prague, crop years 2003, 2004) included 20 varieties from international CIMMYT trials. Samples of year 2003 come from Azerbaijan (Akinci), Bulgaria (Maria), Czech Republic (SG-S1511-99, Samanta, Šárka, Vlasta), Great Britain (Ebi), Hungary (Mv Emese, PRIJMA), Kazakhstan (Oktjabrina), Romania (Flamalb), Russia (Bezostaja), Turkey (WA476/3), Ukraine (Lutescens, Volnyska), USA (Intrada, Jagger, OK97908, TUBBS) and former Yugoslavia (Venera). Samples of year 2004 come from Bulgaria (Demetza, Jubilei I20), Czech Republic (Nela, Samanta, Vlasta), Great Britain (Ebi), Hungary (MV-18-2000, MV-Verbunkos), Mexico (Bonito-27, Seri), Moldova (No Label, Trawaga 54-39-01), Romania (Gloria, Gruia), Russia (Bezostaja 1, Zimorodok), Ukraine (Selyanka, Nikoniya), USA (Arlin/Yuma, Jagger). Wheat samples were grown under the same conditions.

Subset 3 included commercial wheats (40 samples, crop years 2003, 2004) originated from three central Bohemian regions.

Subset 4 included 20 samples of flours bought in retail (crop years 2003 and 2004).

Calibration set including 131 samples was assembled from subsets 1 (51 samples), 2 (40 samples) and 3 (40 samples), remaining 20 samples (subset 4) were intended for validation.

### Methods

Czech standard methods were taken for the determination of protein content (Kjeldahl method, ČSN 56 0512-12) and Zeleny sedimentation (ČSN ISO 5520; SEDI-TEST, ZZN Strakonice).

Rheological behavior of fermented wheat dough was evaluated by SJA Fermentograph (Sweden) and Brabender Oven Spring (Germany). As these test procedures are not included in any international or Czech standard method either, they were performed according to our internal method (Hrušková et al., 2005c). The dough for tests was prepared on Brabender farinograph (Germany) under standard conditions (mixer tempered at 30 °C) using baking test formula and the water amount to reach optimal consistency of 600 ± 20 FU (farinographic units). Only one trait was chosen from each test – fermentografic gas volume and OTG dough volume at 22 min.

Baking test based on fermented dough recipe (internal procedure, see above) was used to assess overall baking flour qualities. The recipe was as follows: flour – 100%, yeast – 4%, salt – 1.7%, sugar – 1.5%, fat – 1% (amount of ingredients in reference to flour) and water needed for dough consistency of 600 ± 20 BU (Hrušková et al., 2005c). Specific loaf volume (as measured by the rapeseed displacement method) was also predicted by NIR technique.

DRIFT spectra of flour samples were recorded on a Bruker IFS66v instrument equipped with a DTGS detector and a KBr beamsplitter. Scanning range from 4000 to 14 500 $cm^{-1}$ and resolution of 16 were used. Diffuse reflectance spectra are reported in Kubelka-Munk Units.

Three spectra records were averaged in OMNIC 6.1a (Thermo Nicolet Corp) software. Calculation of calibration equations was made in programme TQ Analyst 6.0 (Nicolet Instrument Corp). Signal was treated either by MSC or by SNV. First derivative spectra were used, optionally smoothed by Norris derivative filter or Savitzky-Golay filter. Calibration and cross-validation were carried out by Partial Least Square (PLS) regression. The best calibration equations were selected according to their highest coefficient of correlation (*r*) or minimum standard error of cross-validation (SECV). These equations were consequently validated by independent sets.

## RESULTS AND DISCUSSION

The mean values and variencies of examined traits are given in Table 1. Aside from specific loaf volume, the lowest variance can be observed for commercial flours. Year-to-year effect within individual subsets was investigated by means of statistical analysis (ANOVA, 95%).

Table 1. Parameters of flours from variety and commercial wheats

| | Mean | Range | SD | CV (%) |
|---|---|---|---|---|
| Protein (%) | | | | |
| Subset 1 | 11.3 | 8.6–14.4 | 1.05 | 7.3 |
| Subset 2 | 12.7 | 10.8–16.1 | 1.09 | 6.8 |
| Subset 3 | 11.6 | 9.9–12.4 | 0.59 | 4.8 |
| Subset 4 | 13.0 | 11.9–14.7 | 0.84 | 6.4 |
| Zeleny sedimentation (ml) | | | | |
| Subset 1 | 42.6 | 23–64 | 9.37 | 14.6 |
| Subset 2 | 54.5 | 31–72 | 12.77 | 17.7 |
| Subset 3 | 43.7 | 33–53 | 4.85 | 9.1 |
| Subset 4 | 45.6 | 34–62 | 7.47 | 16.4 |
| Gas volume (FeU) | | | | |
| Subset 1 | 117 | 86–140 | 12.71 | 9.1 |
| Subset 2 | 117 | 81–136 | 13.25 | 9.7 |
| Subset 3 | 121 | 108–134 | 5.59 | 4.2 |
| Subset 4 | 127 | 116–145 | 8.59 | 6.8 |
| OTG volume (OU) | | | | |
| Subset 1 | 447 | 270–630 | 97.51 | 15.5 |
| Subset 2 | 523 | 380–685 | 69.52 | 10.1 |
| Subset 3 | 525 | 475–605 | 35.22 | 5.8 |
| Subset 4 | 524 | 450–620 | 48.25 | 9.2 |
| Specific loaf volume (cm$^3$/100 g) | | | | |
| Subset 1 | 273 | 200–394 | 45.46 | 11.5 |
| Subset 2 | 373 | 288–438 | 33.81 | 7.7 |
| Subset 3 | 288 | 211–364 | 42.05 | 11.6 |
| Subset 4 | 307 | 258–360 | 32.36 | 10.6 |

CV – coefficient of variation, SD – standard deviation

Subset 1: Crop years 2003 and 2004 were comparable, the difference was shown for specific loaf volume (higher values in 2004) only. Crop year 2002 differed in lower OTG volume and higher protein and gas volume; specific loaf volume was lower contrary to year 2004. There were no evidential differences among crop years in Zeleny sedimentation. Milling and baking characteristics are more in detail described in Hrušková et al. (2005a, b).

Subset 2: Crop year 2003 deviated in lower protein and gas volume, no evidential differences were proved in other traits. This subset featured high average baking quality (Zeleny sedimentation of 85% samples was over 40 ml).

Subset 3: Crop year 2004 can be characterised by moderate baking quality, featured low protein (average protein content do not satisfied ČSN 46 1100-2) and varying quality as measured by Zeleny test, but 75% samples exceeded 40 ml. OTG volumes were fair for most samples, though varied widely (20–30%). They correlated to maturograph and fermentograph measurements. Specific loaf volume featured higher variance correlated to protein. There was observed relationship between traits and locality. Breadmaking potential of subset 3 samples is more closely described in Hrušková et al. (2005c). Crop year 2003 varied in higher protein and Zeleny sedimentation (85% samples were over 40 ml), but lower specific loaf volume.

Generally, one can conclude that wheat of the year 2003 produced better baking quality and better rheological results than 2004.

Prediction of quality parameters in calibration set is shown in Table 2. For quality assessment of calibration models are crucial validation or cross-validation parameters, but not only calibration. Statistically significant correlation coefficients between calculated and reference values with probability higher than 99% were found for all examined quality parameters even in case of cross-validation ($r_{krit(100)} = 0.254$). However, according to Williams and Norris (2001) these correlations can be classified as applicable to screening (protein), rough screening (gas volume, specific loaf volume) or at least as unsatisfied correlations (Zeleny sedimentation, OTG volume).

It has been well established that NIR technique accurately measures the protein in wheat. Comparing cross-validation results of analytical traits to that of measured by Jirsa et al. (2005) on dispersion-based instrument NIRSystem 6500 (Foss NIRSystems, Inc., USA), who achieved SECV = 0.15%, $r = 0.991$ for protein and SECV = 5.6 cm$^3$, $r = 0.860$ for Zeleny sedimentation, less powerfull models have been established (SECV = 0.54, $r = 0.877$ and SECV = 9.7, $r = 0.501$). Nevertheless, Sorvaniemi et al. (1993) obtained for protein correlations as follows: $R^2 = 0.92$ and SEP = 0.40 g/100 g dry basis (laboratory milled flours); $R^2 = 0.91$ and SEP = 0.63 g/100 g dry basis (commercial milled flours). Less prediction performance of Zeleny sedimentation could be cause of different granulation, because the spectra

Table 2. The best calibration equations selected by cross-validation

| Parameter | Factors | Calibration | | | Cross-validation | |
|---|---|---|---|---|---|---|
| | | $r$ | bias | RMSEC | $r$ | RMSECV |
| Protein (%) | 10 | 0.919 | –0.00165 | 0.442 | 0.877 | 0.540 |
| Zeleny sedimentation (ml) | 7 | 0.753 | 0.0328 | 7.16 | 0.501 | 9.72 |
| Gas volume (FeU) | 8 | 0.833 | –0.00199 | 6.25 | 0.762 | 7.39 |
| OTG volume (OU) | 6 | 0.780 | 0.0481 | 46.2 | 0.693 | 53.4 |
| Specific loaf volume (ml/100 g) | 7 | 0.865 | 0.0519 | 29.9 | 0.769 | 38.3 |

$r$ – correlation coefficient, RMSEC – root mean square error of calibration, RMSECV – root mean square error of cross-validation

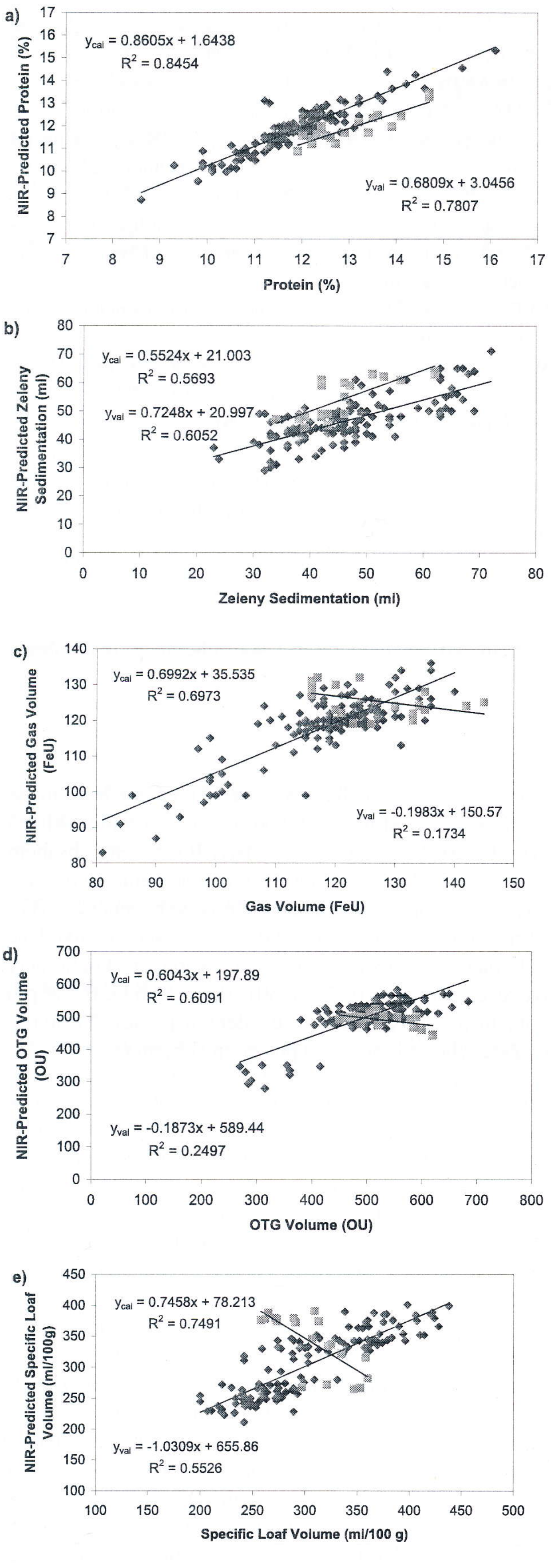

Fig. 1. Comparison of traits determined by prediction model and by reference method: a) protein, b) Zeleny sedimentation, c) gas volume, d) OTG volume, e) specific loaf volume
$y_{cal}$ = calibration model, $y_{val}$ = validation model

were acquired from unsifted flours. This phenomenon showed itself for variety flours, in particular.

As to the rheological parameters, models achieved by presented technique exhibits similar correlation coefficients and standard errors (SECV = 7.4 FeU, $r$ = 0.762 and SECV = 38 $cm^3$/100 g, $r$ = 769) when comparing to Jirsa et al. (2005) – SECV = 6.5 FeU, $r$ = 0.804 for gas volume and SECV = 36 $cm^3$/100 g, $r$ = 0.785 for specific loaf volume. As mentioned above, there was established at least unsatisfied calibration for OTG volume.

Althought the positive and strong relationship between protein and loaf volume in individual wheat cultivars is well known (with usual positive correlations better than 0.90), Rubenthaler and Pomeranz (1987) obtained an evidence that NIR was measuring loaf volume independently from the protein presence.

The selected calibration equations were validated by an independent set. However, the set included quite a low number of samples and they differ from calibration one by nature, which has been shown on validation results. Spectra position of their samples towards calibration ones can be visualised by means of PC-score plot (data not shown). It was revealed that apart from two consumer flours all the validation samples lay within the calibration group. The best prediction was confirmed for protein (Fig. 1a) and Zeleny sedimentation (Fig. 1b), predictions of rheological traits (Fig.1c–e) cannot be considered satisfactory. Further sets examination will be needed to gain more precise calibrations.

## CONCLUSIONS

Cross-validation results confirmed the best prediction accuracy of protein content, but this was less reliable than in case of dispersion-based measurements. On other hand, low predictability was observed for Zeleny sedimentation. Nevertheless, better performance was observed using independent set. Results roughly comparable to reference methods have been developed for rheological parameters – fermentografic gas volume, OTG dough volume at 22 min and specific loaf volume, which is similar to dispersion-based measurements. Especially model for fermentographic gas volume showed usability for screening testing. It seems that close range of validation set of consumer flours and its specific character towards calibration could be influenced by validation results.

## REFERENCES

HRUŠKOVÁ, M. – ŠVEC, I. – JIRSA, O.: Milling and baking characteristics of selected winter wheat varieties. Mlynářské noviny, *16*, 2005a (3/4): 7–10. (In Czech)

HRUŠKOVÁ, M. – ŠVEC, I. – JIRSA, O.: Milling and baking characteristics of selected spring wheat varieties. Mlynářské noviny, *16*, 2005b (5/6): 7–9. (In Czech)

HRUŠKOVÁ, M. – ŠVEC, I. – JIRSA, O.: Baking potential in commercial wheat. Pekař cukrář, *5*, 2005c: 20–23. (In Czech)

HRUŠKOVÁ, M. – JIRSA, O. – ŠVEC, I.: Wheat dough rheological characteristics predicted by NIR technique. In: Proc. ICC – Jubilee Conf., Vienna, Austria, 2005d.

JIRSA, O. – HRUŠKOVÁ, M. – ŠVEC, I.: Prediction of Fermented Wheat Dough Behaviour. Getreidetechnologie, 2005.

MISSBACH, M.: Performance comparison of low price NIR diode array apparatus with traditional NIR spectrometers. Lebensm. Biotechnol., *15*, 1998: 72.

PETR, J.: Wheat cultivation according top utility trends. Zeměd. Inform., 2001 (20): 9–11. (In Czech)

POSNER, E. S. – HIBBS, A. N.: Wheat Flour Milling. 2nd ed. St. Paul Minnesota, USA, AACC, Inc. 2005: 6–47.

RUBENTHALER, G. L. – POMERANZ, Y.: Near-infrared reflectance spectra of hard red winter wheats varying widely in protein content and breadmaking potential. Cereal Chem., *64*, 1987: 407–411.

SLUIMER, P.: Principles of Breadmaking. St. Paul, Minnesota, USA, AACC, Inc. 2005: 27–41.

SORVANIEMI, J. – KINNUNEN, A. – TSADOS, A. – MAELKKI Y.: Using partial least squares regression and multiplicative scatter correction for FT-NIR data evaluation of wheat flours. Lebensm.-Wiss. Technol., *26*, 1993: 251–258.

Summary of varieties for 2004 (2005): Central Institute for Supervising and Testing in Agriculture, Brno, Division of Variety Testing. (In Czech)

ŠVEC, I. – HRUŠKOVÁ, M.: Wheat flour fermentation study. Czech J. Food Sci., *22*, 2004: 17–23.

WILLIAMS, P. C. – NORRIS, K. H.: Near-Infrared Technology in the Agricultural and Food Industries. 2nd ed. St. Paul, Minnesota, USA, AACC, Inc. 2001.

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JIRSA, O. – HRUŠKOVÁ, M. – ŠVEC, I. (Vysoká škola chemicko-technologická, Ústav chemie a technologie sacharidů, Praha, Česká republika):

**NIR metoda pro predikci znaků pekařských odrůd.**

Scientia Agric. Bohem., *37*, 2006: 146–150.

Predikce vybraných technologických ukazatelů FT-NIR spektroskopií byla sledována na 151 vzorcích pšeničné mouky. Mouky zahrnovaly tři skupiny vzorků: 131 kalibračních vzorků z odrůdových a komerčních pšenic (sklizeň 2002–2004) a validační soubor – 20 konzumních mouk (rok výroby 2004 a 2005). Vzorky byly hodnoceny obsahem bílkovin, Zelenyho testem, fermentografickým objemem plynů, OTG objemem a měrným objemem, které byly vybrány z cca 40 hodnocených znaků. NIR spektra byla měřena na přístroji Bruker IFS66v vybaveném DTGS detektorem a KBr děličem paprsků. Kalibrační modely s křížovou a nezávislou validací pro všechny uvedené charakteristiky byly počítány PLS regresí prostřednictvím TQ Analyst. Statisticky významné korelační koeficienty ($\alpha = 0{,}01$) byly nalezeny pro všechny charakteristiky při křížové validaci: $r = 0{,}877$, 0,501, 0,762, 0.693, 0,769 pro obsah bílkovin, Zelenyho sedimentaci, objem plynů, OTG objem a měrný objem, v uvedeném pořadí. Nezávislá validace naznačila možnost predikce (kromě obsahu bílkovin a Zelenyho sedimentace) pro měrný objem ($r = 0{,}553$).

FT-NIR; pšenice; mouka; reologie

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