Wheat Flour Dough Rheological Characteristics Predicted by NIRSystems 6500

MARIE HRUŠKOVÁ[1], *MARTINA BEDNÁŘOVÁ*[1] *and FRANTIŠEK NOVOTNÝ*[2]

[1]*Institute of Chemical Technology – Department of Carbohydrate Chemistry and Technology, Prague;* [2]*Central Institute for Supervising and Testing in Agriculture, Brno, Czech Republic*

Abstract

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Rheological quality of wheat dough prepared from 114 wheat flour samples (wheat harvest 1998 and 1999) was assessed by help of farinograph and extensigraph. Spectra of all samples were measured on spectrograph NIRSystems 6500 NIR. Calibration equations with cross and independent validation for all rheological characteristics were computed by NIR Software ISI Present WINISI II using mPLS and PLS methods. The quality of prediction was evaluated by coefficients of correlation between measured and predicted values from cross and independent validation. A statistically significant dependence between predicted and measured values (with probability higher than 99%) was determined in all mentioned rheological characteristics in the case of cross validation. Only farinograph absorption, time of dough development and mixing tolerance were successfully predicted by independent validation. Predictions of extensigraph characteristics were not found out statistically significant probably due to a small number of tested samples.

Keywords: wheat flour dough; farinograph; extensigraph; NIRSystems 6500; prediction of rheological characteristics

Wheat flour is able to form a cohesive dough with viscoelastic properties and has the ability to retain gas, which is essential for production of baked products with a light texture. The factor responsible for dough formation is gluten protein and its development during mixing, fermentation and handling actions has influence on the rheological properties of dough.

Rheological characteristics such as elasticity, viscosity and extensibility are important for the milling and bakery industries in prediction of the processing parameters of dough and quality of end products. These rheological characteristics change throughout the breadmaking process and are difficult to measure in definitive terms (HRUŠKOVÁ 1993).

To predict the quality of a flour and its dough, a number of physical, chemical and rheological characteristics have to be known. For the quality control of wheat flour dough, many test are used to predict baking performance. Such tests as farinograph (SHUEY 1972) and extensigraph (RASPER & PRESTON 1991) are useful, but only the baking test has a high degree of prediction. Because the baking test and rheological measurements require a lot of time and equipment to be performed, a fast and reliable test is necessary. This problem can be solved by the NIR spectroscopy, which is almost well established to control analytical properties of flour in many industrial mills.

NIR was applied to many predictions of flour components (POMERANZ 1971; OSBORNE *et al.* 1984; DELWICHE & WEAVER 1994), farinograph dough behavior (WILLIAMS *et al.* 1984), mixograph dough parameters (DELWICHE & WEAVER 1994), extensigraph dough characteristics (DELWICHE *et al.* 1998) and alveograph dough properties (RUBENTHALER & POMERANZ 1987; FARAD & RAJDER 1987). Prediction of dough properties by NIR spectra analyses was influenced by many factors, especially errors of rheological methods and result dependence on the protein content of tested flour. Reliability of computed characteristics of dough varies according to calibration sample set, extent and quality range of flour parameters.

The main objective of this work was to predict rheological properties of wheat flour dough, measured on farinograph and extensigraph, by NIR spectroscopy.

MATERIAL AND METHODS

Samples: Wheat flours were milled on an experimental Bühler mill (type MLU-202), with extraction between 70 and 75%, from thirty-nine wheat varieties (harvest 1998) and seventy-five wheat varieties (harvest 1999). Wheat samples came from three breeding farms and were cultivated in the same agricultural conditions. Flours samples were supplied by Central Institute for Supervising and Testing in Agriculture in Brno.

References Analysis: Rheological properties of wheat flour were determined on farinograph (in accordance with ČSN ISO Standard 55 30-1) and on extensigraph (in accordance with ČSN ISO Standard 55 30-2). The results of rheological analysis were expressed by average, minimum and maximum value and by standard deviation of each sample set.

Apparatus: Spectra were obtained by the wavelength scanning instrument NIRS 6500 (NIRSystems, Inc.) using of 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.

Statistics: NIR Software ISI Present WINSI II (Infrasoft Int.) was used to evaluate the data and to develop chemometric models. Variance correction (v.c.) was performed by Standard Normal Variate Transformation (SNV) and then transformed by the first derivative, which was calculated by using treatment 1, 4, 4, 1 and 1, 8, 8, 1. Calibration was used by Modified Partial Least Square (MPLS) and Partial Least Square (PLS) regression and correlation coefficient (r) was determined. No samples were removed because of their higher deviations. The calibration of dough rheological properties was verified by cross validation (number of selected segments was equal to the number of samples of each set) and independent validation (the calibration for samples – harvest 1999 was verified by an independent set – harvest 1998).The selection of optimum number of PLS terms for the calibration was based on standard error of prediction (SEP) that should be minimised. Two statistical parameters (SEP and r) were used to determine the calibration equation.

Table 1. Flour rheological properties – harvest 1998 and 1999

Parameter	1998 average	1998 range min.	1998 range max.	1998 S.D.	1998 v.c. (%)	1999 average	1999 range min.	1999 range max.	1999 S.D.	1999 v.c. (%)
Farinograph characteristics										
Water absorption (%)	60.6	54.8	67.4	3.5	5.7	57.6	52.2	69.1	6.3	5.4
Time development (min)	4.6	1.6	8.4	1.7	38.1	3.3	1.6	7.2	1.0	38.1
Dough stability (min)	9.3	1.8	18.6	4.8	51.2	5.3	2.7	15.7	2.3	42.9
Mixing tolerance (F.J.)	68	18	154	35.6	52.1	74	31	141	17.9	24.5
Extensigraph characteristics (rest time 45 min)										
Resistance (E.J.)	286	140	450	76	26.5	184	115	360	51	27.6
Max.resistance (E.J.)	412	140	745	148	35.9	243	115	535	83	34.0
Extensibility (mm)	167	131	199	16	9.5	476	124	218	24	13.8
Ratio number	1.7	1	2.5	0.4	25.0	1.1	0.6	2.1	0.3	31.0
Energy (cm^2)	96	26	180	37.0	38.7	68	20	120	19.1	31.8
Extensigraph characteristics (rest time 90 min)										
Resistance (E.J.)	338	125	520	98	29.0	222	125	410	68	27.8
Max. resistance (E.J.)	489	130	900	190	38.9	294	130	645	183	16.7
Extensibility (mm)	162	130	212	16	10.1	178	144	234	17	9.6
Ratio number	2.1	0.7	3.2	0.6	29.2	1.3	0.6	2.6	0.4	33.3
Energy (cm^2)	108	33	198	42.5	39.2	71	29	162	21.3	29.5
Extensigraph characteristics (rest time 135 min)										
Resistance (E.J.)	354	130	560	106	30.1	236	130	490	66	27.5
Max. resistance (E.J.)	507	135	935	202	39.9	320	140	715	112	34.8
Extensibility (mm)	161	109	206	29	18.2	175	145	223	17	9.7
Ratio number	2.3	0.8	3.8	0.7	31.5	1.3	0.7	3.3	0.5	35.8
Energy (cm^2)	109	24	203	43.4	39.8	75	33	160	21.7	28.6

v.c. = variation coefficient

Table 2. Prediction of flour rheological characteristics – harvest 1998 and 1999

Parameters	1998 n	1998 calibration Term	SEC	r	1998 cross validation segment	SEP	r	1999 n	1999 calibration Term	SEC	r	1999 cross validation segment	SEP	r
Farinograph characteristics														
Water absorption (%)	39	1	3.1	**0.445**	39	3.4	0.277	75	4	1.8	**0.817**	75	2.0	**0.789**
Time of development (min)	39	3	1.0	**0.827**	39	1.3	**0.688**	75	1	0.9	**0.560**	75	0.9	**0.511**
Dough stability (min)	39	3	3.1	**0.765**	39	3.5	**0.701**	75	1	2.4	0.297	75	1.0	0.145
Mixing tolerance (F.J.)	39	4	25	**0.716**	39	28.8	**0.605**	75	1	18.6	0.219	75	20.2	0.000
Extensigraph characteristics (resting time 45 min)														
Resistance (E.J.)	39	4	46.8	**0.785**	39	53.4	**0.717**	74	4	42.8	**0.507**	74	46.4	**0.366**
Max. resistance (E.J.)	39	4	98.3	**0.797**	39	101.0	**0.738**	74	4	69.5	0.550	74	78.9	0.338
Extensibility (mm)	39	2	14.3	**0.433**	39	15.4	0.295	74	1	16.6	0.169	74	17.7	0.000
Ratio number	39	3	0.3	**0.744**	39	0.3	**0.606**	74	1	0.3	*0.240*	74	0.3	0.077
Energy (cm^2)	39	4	23.0	**0.785**	39	25.7	**0.729**	74	3	17.0	**0.506**	74	118.3	**0.383**
Extensigraph characteristics (resting time 90 min)														
Resistance (E.J.)	39	4	61.5	**0.778**	39	70.4	**0.704**	75	43	50.5	**0.554**	75	54.1	**0.464**
Max. resistance (E.J.)	39	4	114.8	**0.797**	39	130.9	**0.733**	75	3	85.7	**0.507**	75	94.0	**0.342**
Extensibility (mm)	39	2	14.4	**0.477**	39	15.5	*0.361*	75	3	14.3	**0.578**	75	17.0	*0.263*
Ratio number	39	4	0.4	**0.695**	39	0.5	**0.600**	75	4	0.4	**0.519**	75	0.4	**0.471**
Energy (cm^2)	39	4	24.8	**0.802**	39	28.6	**0.748**	75	4	20.2	**0.468**	75	22.0	*0.289*
Extensigraph characteristics (resting time135 min)														
Resistance (E.J.)	39	4	66.7	**0.779**	39	76.6	**0.704**	75	4	58.8	**0.509**	75	61.8	**0.439**
Max. resistance (E.J.)	39	4	119.1	**0.808**	39	135.7	**0.749**	75	4	103.3	**0.496**	75	110.3	**0.390**
Extensibility (mm)	39	2	26.0	**0.453**	39	28.4	0.281	75	1	15.9	**0.378**	75	16.6	*0.277*
Ratio number	39	4	0.5	**0.705**	39	0.6	**0.600**	75	1	0.5	**0.421**	75	0.5	**0.361**
Energy (cm^2)	39	4	24.8	**0.821**	39	27.9	**0.773**	75	3	19.9	**0.510**	75	21.7	**0.372**

$r_{crit\ (\alpha = 0.01,\ 75\ samples)} = 0.296$; $r_{crit\ (\alpha = 0.05,\ 75\ samples)} = 0.228$; $r_{crit\ (\alpha = 0.01,\ 74\ samples)} = 0.298$; $r_{crit\ (\alpha = 0.05,\ 74\ samples)} = 0.229$; $r_{crit\ (\alpha = 0.01,\ 39\ samples)} = 0.408$; $r_{crit\ (\alpha = 0.05,\ 39\ samples)} = 0.317$

RESULTS AND DISCUSSION

Analytical Characteristics: Wheat flour quality from harvests 1998 and 1999 corresponded to the Czech standard for bakery products of fine type. The analytical ranges were from 10.8 to 15.8% for protein, from 17 to 45 ml for Zeleny sedimentation value and from 194 to 402 s for Falling number.

Rheological Characteristics: The results of farinograph and extensigraph measurements of dough are summarised in Table 1 (harvest 1998 and 1999). According to these characteristics, flours from harvest 1998 were evaluated as stronger with higher water absorption, longer time of development and dough resistance. Doughs prepared from these flours had an optimal viscoelastic behaviour described by a well-balanced ratio of gluten extensibility and elasticity, which improved after resting time. On the other hand, wheat flours from harvest 1999 were marked as bakery weaker, but typical of local conditions.

Prediction according to Cross Validation: Prediction of flour rheological properties from harvest 1998 (set of thirty-nine samples), from harvest 1999 (set of seventy-four samples) and both sets together (all samples) are shown in Tables 2 and 3, respectively. For the calibration of farinograph characteristics all parameters were found significant on 99% statistical level in the set of all samples but in some of them (harvest 1998 or 1999) only time of dough development was predicted successfully. Extensigraph parameters were computed with good accuracy in all samples.

Prediction according to Independent Validation: The correlation coefficient *r* and standard error of independent prediction SEP for analytical parameters are shown in Table 4. Flour moisture, ash content, protein content and Zeleny sedimentation value were successfully predicted in agreement with published literature (MC DONALD 1986). A higher correlation coefficient ($r = –0.68$) mentioned in Zeleny value in comparison with the result obtained in this experiment could be explained by a different range (sedimentation value 31–54 ml) of calibration set.

The correlation coefficient *r* and standard error of independent prediction SEP for farinograph and extensi-

Table 3. Prediction of flour rheological properties – harvests 1998 and 1999

Parameters	*n*	Calibration			Cross validation		
		Term	SEC	*r*	segment	SEP	*r*
Farinograph characteristics							
Water absorption (%)	114	3	2.3	**0.724**	114	2.5	**0.655**
Time of development (min)	114	1	1.1	**0.623**	114	1.2	**0.592**
Dough stability (min)	114	1	3.3	**0.559**	114	3.4	**0.494**
Mixing tolerance (F.J.)	114	3	22.9	**0.466**	114	24.5	**0.335**
Extensigraph characteristics (resting time 45 min)							
Resistance (E.J.)	113	1	50.1	**0.750**	113	57.7	**0.651**
Max. resistance (E.J.)	113	1	59.3	**0.749**	113	102.4	**0.654**
Extensibility (mm)	113	1	14.7	**0.536**	113	16.1	**0.389**
Ratio number	113	1	0.3	**0.738**	113	0.4	**0.624**
Energy (cm^2)	113	1	20.9	**0.748**	113	24.1	**0.648**
Extensigraph characteristics (resting time 90 min)							
Resistance (E.J.)	114	2	66.9	**0.700**	114	73.7	**0.622**
Max. resistance (E.J.)	114	1	110.1	**0.748**	114	128.4	**0.637**
Extensibility (mm)	114	1	15.4	**0.573**	114	16.0	**0.529**
Ratio number	114	1	0.5	**0.649**	114	0.5	**0.607**
Energy (cm^2)	114	1	25.3	**0.701**	114	28.2	**0.609**
Extensigraph characteristics (resting time 135 min)							
Resistance (E.J.)	114	1	73.3	**0.681**	114	79.1	**0.616**
Max. resistance (E.J.)	114	1	131.4	**0.664**	114	145.2	**0.568**
Extensibility(mm)	114	1	19.8	**0.508**	114	21.0	**0.418**
Ratio number	114	1	0.5	**0.653**	114	0.6	**0.610**
Energy (cm^2)	114	1	25.2	**0.702**	114	28.3	**0.602**

$r_{crit\ (\alpha = 0.01,\ 100\ samples)} = 0.254$; $r_{crit\ (\alpha = 0.05,\ 100\ samples)} = 0.195$

Table 4. Independent validation of calibration equations – analytical characteristic

Parameters	*n*	Calibration			Independent validation		
		Term	SEC	*r*	segment	SEP	*r*
Analytical characteristics							
Moisture (%)	75	4	0.08	**0.942**	39	0.20	**0.863**
Falling number (s)	75	4	46.8	**0.722**	39	60.3	0.219
Ash content (% D.M.)	75	4	0.05	**0.827**	39	0.05	**0.687**
Protein content (% D.M.)	75	4	0.3	**0.960**	39	0.4	**0.963**
Zeleny sedimentation value (ml)	75	3	3.7	**0.631**	39	4.6	**0.548**

$r_{crit(a=0.01,\ 75\ samples)} = 0.296$; $r_{crit\ (a = 0.01,\ 39\ samples)} = 0.408$; $r_{crit\ (a = 0.05,\ 39\ samples)} = 0.317$

graph dough characteristics are summarised in Table 5. The time of dough development and dough resistance against overmixing were independently predicted as was found by DELWICHE and WEAVER (1994). These authors give SEP for dough stability 1.1–3.2 min and $r = 0.8$. With lower probability (on 95% level) farinograph flour water absorption was determined as Delwiche reported in Canadian flours (DELWICHE *et al.* 1998).

Independent validation of the calibration equation were not found significant for any extensigraph parameters at

Table 5. Independent validation of calibration equations – rheological characteristic

Parameters	*n*	Calibration			Independent validation		
		Term	SEC	*r*	segment	SEP	*r*
Farinograph characteristics							
Water absorption (%)	75	4	1.8	**0.817**	39	3.4	*0.401*
Time of development (min)	75	1	0.9	**0.560**	39	1.4	**0.619**
Dough stability (min)	75	1	2.4	**0.297**	39	4.7	0.202
Mixing tolerance (F.J.)	75	1	18.6	0.219	39	34.3	**0.415**
Extensigraph characteristics (resting time 45 min)							
Resistance (E.J.)	74	4	42.8	**0.507**	39	85.5	0.071
Max. resistance (E.J.)	74	4	69.5	**0.550**	39	160.7	0.095
Extensibility (mm)	74	1	16.6	0.169	39	16.4	0.071
Ratio number	74	1	0.3	*0.240*	39	0.4	0.200
Energy (cm^2)	74	3	17.0	**0.506**	39	39.5	0.100
Extensigraph characteristics (resting time 90 min)							
Resistance (E.J.)	75	4	50.5	**0.554**	39	100.1	0.118
Max. resistance (E.J.)	75	3	85.7	**0.507**	39	194.6	0.063
Extensibility (mm)	75	3	14.3	**0.578**	39	18.1	*0.330*
Ratio number	75	4	0.4	**0.519**	39	0.6	0.228
Energy (cm^2)	75	4	20.2	**0.468**	39	44.7	0.045
Extensigraph characteristics (resting time 135 min)							
Resistance (E.J.)	75	4	58.8	**0.509**	39	102.8	0.276
Max. resistance (E.J.)	75	4	103.3	**0.496**	39	205.5	0.095
Extensibility (mm)	75	1	15.9	**0.378**	39	27.1	*0.394*
Ratio number	75	1	0.5	**0.421**	39	0.7	*0.373*
Energy (cm^2)	75	3	19.9	**0.510**	39	45.6	0.032

$r_{crit\ (\alpha = 0.01,\ 75\ samples)} = 0.296$; $r_{crit\ (\alpha = 0.01,\ 39\ samples)} = 0.408$; $r_{crit\ (\alpha = 0.05,\ 75\ samples)} = 0.228$; $r_{crit\ (\alpha = 0.05,\ 39\ samples)} = 0.317$; $r_{crit\ (\alpha = 0.01,\ 74\ samples)} = 0.298$; $r_{crit\ (\alpha = 0.05,\ 74\ samples)} = 0.229$

three resting times and probability level 95% despite the fact that WILLIAMS *et al.* (1984) results were positive (SEP for extensigraph energy was 29 cm^2). The accuracy of NIR methods depends on the used range of quality parameters and on the accuracy of reference methods.

CONCLUSIONS

NIR filter spectroscopy was used for a screening of analytical properties of wheat flours in mills and bakeries. The results of evaluation of reliability and accuracy of moisture, ash and protein content and Zeleny sedimentation value were proved by independent validation of the calibration equations in the set of samples from wheat harvests 1998 and 1999. Larger differences were found between measured and computed data in rheological properties of wheat dough. Farinograph water absorption, time of dough development and dough stability could be predicted with comparable accuracy as reference methods. Extensigraph characteristics were not successfully predicted according to the results obtained in this experiment, probably due to a small size of wheat flour sample set and their qualitative differences.

References

DELWICHE S.R.,WEAVER G. (1994): Bread quality of wheat flour by NIR spectroscopy: Feasibility of modeling. J. Food Sci., **59**: 410–415.

DELWICHE S.R., GRAYBOSH R.A., PETERSON C.J. (1998): Predicting protein and biological properties of HRW wheat by NIR. Cereal Chem., **75:** 412–416.

FARADI H., RASPER V.F. (1987): The Alveograph Handbook. AACC, Inc. St. Paul, Minnesota, US: 17–22.

HRUŠKOVÁ M. (1993): Některé aspekty pekařské technologie s ohledem na kvalitativní znaky mouky a jejich vliv na jakost hotových výrobků. In: Sbor. QALIMA 93, Hradec Králové: 21–23.

MC DONALD C. (1986): NIR spectroscopy in the milling and baking quality cereal laboratory. Cereal Food World, **31**: 410–411.

OSBORNE B.G., BARET G.M., CAUVAIN S.P., FEARN T. (1984): Determination of protein, fat and moisture in bread by NIR spectroscopy. J. Sci. Food Agric., **35**: 940–945.

POMERANZ Y. (1971): Wheat Chemistry and Technology. AACC, Inc. St. Paul, Minnesota, US: 536–543.

RASPER V.F., PRESTON K.R. (1991): The Extensigraph Handbook. AACC, Inc. St. Paul, Minnesota, US: 13–18.

RUBENTHALER G.L., POMERANZ Y. (1987): NIR Spectra of HRW wheat varying widely in protein content and breadmaking potential. Cereal Chem., **64**: 407–411.

SHUEY W.C. (1972): The Farinograph Handbook. AACC, Inc. St. Paul, Minnesota, US: 20–33.

WILLIAMS P.C., EL-HARAMEN F.J., ORTIS-FERIRA G., SRIVASTA J.P. (1984): Preliminary observations of the determination of wheat strength by NIR. Cereal Chem., **65:** 109–114.

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Souhrn

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Reologické vlastnosti pšeničného těsta, vyrobeného ze 114 vzorků pšeničných mouk z odrůd potravinářské pšenice sklizně 1998 a 1999, byly hodnoceny pomocí farinografu a extenzografu. Na spektrofotometru NIRSystems 6500 byla u všech vzorků naměřena NIR spektra. Pro uvedené reologické charakteristiky byla programem NIR Software ISI Present WINISI II metodami mPLS a PLS provedena kalibrace, křížová a nezávislá validace. Kvalita předpovědi byla posuzována podle hodnot korelačního koeficientu 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 křížové validaci. Při nezávislé validaci v daném souboru lze úspěšně predikovat farinografickou vaznost, dobu vývinu a stabilitu těsta. S pravděpodobností vyšší než 99 % nebyla nalezena statisticky významná závislost žádného extenzografického ukazatele, pravděpodobně vlivem malého rozsahu testovaného souboru a rozsahu jejich jakostních znaků.

Klíčová slova: pšeničné těsto; farinograf; extenzograf; NIRSystems 6500; predikce reologických ukazatelů

Corresponding author:

Ing. MARIE HRUŠKOVÁ, CSc., Vysoká škola chemicko-technologická, Ústav chemie a technologie sacharidů, Technická 5, 166 28 Praha 6, Česká Republika

tel.: + 420 2 24 35 32 06, fax: + 420 2 33 33 99 90, e-mail: hruskovm@vscht.cz.