The Advanced Approaches to Nutritional and Breadmaking Quality of Wheat, Barley and Rye Flour

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Abstract: This work is focused on the characterization and rapid analytical determination of cereal flour quality with regard to nutritional and breadmaking quality. Starch, protein and non-starch polysaccharides are the main components of cereals. The content and quality of proteins and content of damaged starch is important because of the technological quality of flours. The high content of high molecular weight proteins is substantial for bread technology especially, while soluble protein fractions and non-starch polysaccharides are important for nutrition. The set of wheat, barley and rye flours and their blends were analyzed and their properties and their qualitative parameters were determined. Principal component analysis (PCA) was used on Fourier transform-infrared (FT-IR) spectra in the 1,200-800 cm^{-1} wavenumber region and significant correlations of various nutritional and breadmaking parameters were observed. Results showed that the FT-IR spectroscopy and PCA can serve for rapid screening and classification of cereal flour quality.

Key words: Cereals, flour, quality, FT-IR spectroscopy, PCA.

1. Introduction

Cereals play the main role in the agricultural production of the majority of countries. This fact is connected first of all with the importance of cereals in nutrition. The greatest part of wheat and rye is used for bread and bakery products in Northern, Central and Eastern Europe and they contribute to an important source of protein and dietary fiber in the diet as well as in the Czech Republic. Wheat is the most important crop for breadmaking because of its supreme baking performance in comparison with all other cereals. Only wheat proteins can make the three-dimensional network (gluten structure) of wheat dough during mixing and kneading of dough. However, wheat is lower in dietary fiber content and the nutritive value of bread could be increased by a supplementation of rye and barley or an addition of isolated dietary fiber into

the bread formula [1-3]. However, Hung [4] presented a new kind of whole waxy wheat with higher dietary fiber content than regular whole wheat flour. Among commonly grown cereals, whole grain rye has the highest dietary fiber content [5]. Rye is considered to be a healthy cereal with regard to a number of bioactive compounds such as phenolic acids, lignans and also alkylresorcinols [6]. Recently we have observed a renewed interest in barley for food production [7]. As the latest investigations were accomplished in barley breeding, the new beta-glucan-rich barley for baking was developed [8]. Hull-less cultivars of barley have better nutritional value than hulled ones with regards to higher protein and soluble dietary fiber content. A hull-less barley flour was incorporated into white and wholegrain wheat bread and the bread showed acceptable and desirable nutritional and sensory properties [9].

The main components of cereals are carbohydrates (unavailable: non-starch polysaccharides, dietary fiber

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and available: starch) and proteins. Starch is a major food reserve providing a bulk nutrient and energy source with high glycemic response in the human diet [10]. The content of starch in a cereal grain varies but generally it is between 60% and 75% of the weight of the grain. During cereal processing and grain milling in particular, the content of damaged starch is raised in the flour and can influence the technological quality of flour and handling of dough. Starch granule size distribution is an important factor that affects the quality of final cereal products. The effects of wheat starch granules size distribution were observed on mixing and breadmaking quality [11]. The determination of total or damaged starch can be carried out by chemical end group analysis or modern enzymatic methods. Determination of starch content by hydrochloric acid dissolution consists of break-down of starch in acid and measuring of the optical rotation of formed products in solution. The determination is more exacting and Carrez solutions (one of them contains cyanide) are necessary to protein precipitate. Measurement of total or damaged starch by enzymatic kit is rapid, easy but expensive.

The content of proteins in the fully-grown grains varies from 8% to 15% in dry matter. Dominant part of proteins is stored in endosperm and aleuronic layer of cereal grain. Cereal proteins have a high content of glutamine and proline and a very low content of essential amino acids as lysine and threonine. The earliest classification of cereal storage proteins is based on their solubility properties and it is often called Osborne fractionation [12]. Albumins and globulins belong to water-soluble fractions with low molecular weight and especially have the nutritive importance. Gliadins (prolamins) and glutelins have higher molecular weight (from 30 kDa to 20 MDa) and they are extracted in alcohol-water mixture and alkaline-water mixture, respectively. The ratio between high and low molecular weight gluten fractions is predominant for baking performance. Therefore, it seems that the greater the content of large

molecular weight glutenin subunits, the larger the glutenin polymers and the stronger the flour [13]. Tatham [14] published the up-to-date review of wheat, barley and rye prolamins structure, physical chemistry and their functional properties. Protein in flour can be determined chemically by the Kjeldahl method, but the procedure is very complex. The indirect test by near-infrared spectroscopy (NIR) is very widely used in the milling industry but calibration should be carrying out carefully. The particular protein fractions can be separated by liquid chromatography with reverse phase and UV detection. The baking quality of gluten can be expressed by degree of sedimentation (according to Zeleny) of flour suspended in a lactic acid solution during standard time interval.

Dietary fiber is the edible part of the plants or analogous carbohydrates that are resistant to digestion and absorption in the human small intestine with complete or partial fermentation in the large intestine [15]. Detailed medical experiments indicated [16, 17] that dietary fiber can stimulate weakening of hunger, stimulating peristaltic movements, reducing the level of blood glucose, regulating the activity of microbial flora, lowering of cholesterol blood serum level and decreasing of the risk of civilization diseases [18].

Dietary fiber can be classified into water-soluble (SDF) and water-insoluble dietary fiber (IDF) according to their water solubility. The significant components of cereal water-soluble fiber are β -glucans, arabinoxylans, galactomannans, fructans and arabinomannans which can form viscous solutions. Water-insoluble dietary fiber consists of cellulose, hemicelluloses and lignin primarily. Lignin is a lipophilic phenolic polymer that can absorb bile acid [3]. The content of β -glucans and fiber is determined mostly enzymatically. Determination of fibre content is time consuming and expensive (enzyme assay kit, reagents).

Original gravimetric, enzymatic and chemical methods of analysis were not capable to determine reproducibly and quickly the properties and constituents of grain mixtures. On the other hand,

vibrational spectra have recently found their importance in qualitative food analysis [19]. Mid infrared spectroscopy in the wavenumber range 4,000–400 cm^{-1} is a rapid, sensitive and versatile tool for elucidating the structure and physical properties of starch [20] and other carbohydrates [21]. The most important strong absorptions for starch are at 1,150, 1,075 and 1,015 cm^{-1} assigning the bending vibration of (CO) groups, and medium absorptions at 845 cm^{-1} assigning the bending vibration (C₁-H) of β -anomer [20, 22].

Proteins have several strong absorptions in infrared region due to the presence of peptide bond CO-NH. The strongest are stretching vibration of (N-H) groups at wavenumber 3,300–3,200 cm^{-1} (Amide A), stretching vibration of (C = O) at 1,690–1,620 cm^{-1} (Amide I), bending vibration of (N-H) (Amide II) at 1,550–1,500 cm^{-1} and bending vibration of (N-H)/(C-H) at 1,300–1,200 cm^{-1} (Amide III) [23].

Absorbance of specific peaks in the mid infrared spectrum can be correlated with particular cell wall molecules: cellulose 1,162, 1,120, 1,059, 1,033, 930 and 898 cm^{-1} ; β -glucan 1,151, 1,140, 1,076, 1,041, 1,026, 916 and 840 cm^{-1} ; arabinoxylans 1,166 and 998 cm^{-1} ; glucomannan 1,150, 1,092, 1,064, 1,034, 941, 898, 872 and 814 cm^{-1} ; and arabinogalactan 1,139, 1,078, 1,043, 985, 880 and 842 cm^{-1} [24–26]. The characteristic wavenumbers for cell wall non-starch polysaccharides were selected on the basis of the application of chemometric analysis [24]. It was also proved to be useful for a quick evaluation of polysaccharides used as additives in foodstuffs [27].

The aim of this work was to determine the quality of selected flours using several analytical methods to find the relationship among flour quality parameters. The set of Czech and Moravian wheat, barley and rye flours and their blends were analyzed and their qualitative parameters, such as content of moisture, ash, protein and starch, protein fractions, value of sedimentation Zeleny test, solvent retention capacity profile, gluten index, content of soluble, insoluble and total dietary fiber and β -glucans, were determined.

Fourier transform-infrared spectroscopy and principal component analysis were applied to correlate various nutritional and breadmaking parameters.

2. Materials and Methods

2.1 Sample Characterization

Totally 34 samples of commercial flours from wheat (*Triticum aestivum*) (winter), barley (*Hordeum vulgare*) (2-rowed, spring, hull-less) and rye (*Secale cereale*) were analyzed. 7 samples of wheat flours growing in 2010 in different Czech regions, 12 samples of wheat flours growing in 2010 in different regions of Moravia, 3 samples of barley flours (Czech region, 2010 crop), 1 sample of barley bran (Czech region, 2010 crop), 1 sample of rye flour (Czech region, 2010 crop) and 10 samples of flours blends were used as experimental materials (Table 1). The blends were prepared by mixing in various proportions of cereals (20%, 30%, 40%, 50% and 60% portions of barley to wheat; and blends wheat:rye:barley 30%:10%:60%, 25%:15%:60% and 20%:20%:60%). Samples no. 15 and 16 were wheat flours for biscuits and cookies, samples no. 17 and 18 were blends wheat-rye flours for bread and bakery production. Samples no. 19–34 were wheat flours for bread production. All flours were sieved through a 0.8 mm sieve.

2.2 Chemical Analysis

Moisture content was determined by drying at 105 °C to constant weight [28]. Ash content was determined by burning the samples at 925 °C to constant weight [28]. The soluble, insoluble and total dietary fiber content was determined according to the method AOAC 985.29 [29] combined with total dietary fiber assay kit K-TDFR (Megazyme International Ireland Ltd., Wicklow, Ireland). The content of mixed linkage (1 $\rightarrow$ 3)(1 $\rightarrow$ 4)- β -D-glucans was determined according to the AACC Method 32-23 [30] combined with McCleary method assay procedure K-BGLU (Megazyme International Ireland

Table 1 Summary of analyzed samples of flours and their blends.

Sample No.	Type of flour	Origin location
1	wheat	Czech
2	barley:wheat (20%:80%)	Czech
3	barley:wheat (30%:70%)	Czech
4	barley:wheat (40%:60%)	Czech
5	barley:wheat (50%:50%)	Czech
6	barley:wheat (60%:40%)	Czech
7	wheat:rye:barley (20%:20%:60%)	Czech
8	wheat:rye:barley (25%:15%:60%)	Czech
9	wheat:rye:barley (30%:10%:60%)	Czech
10	barley	Czech
11	barley bran	Czech
12	barley	Czech
13	barley	Czech
14	rye	Czech
15	soft wheat for cookie	Czech
16	soft wheat for cookie	Czech
17	wheat: rye (70:30) for bread	Czech
18	wheat: rye (60:40) for bread	Czech
19	wheat for bread and bakery	Czech
20	wheat for bread and bakery	Czech
21	wheat for bread and bakery	Czech
22	wheat for bread and bakery	Czech
23	wheat for bread and bakery	Moravia
24	wheat for bread and bakery	Moravia
25	wheat for bread and bakery	Moravia
26	wheat for bread and bakery	Moravia
27	wheat for bread and bakery	Moravia
28	wheat for bread and bakery	Moravia
29	wheat for bread and bakery	Moravia
30	wheat for bread and bakery	Moravia
31	wheat for bread and bakery	Moravia
32	wheat for bread and bakery	Moravia
33	wheat for bread and bakery	Moravia
34	wheat for bread and bakery	Moravia

Ltd., Wicklow, Ireland). Total starch content was quantified enzymatically by a total starch assay kit from Megazyme (AACC 76-13) [30]. Nitrogen content was measured using the Kjeldahl method and multiplied by a factor 5.7 to determine the protein content (ICC 105/2, 1996) [28]. Fractionation of proteins was performed by modified procedure [31]. Determination of gluten quality by means of the sedimentation index (Zeleny test) (ISO 5529:2007) [32] and gluten index (GI) (AACC 38-12) [30] was

carried out. Sedimentation test of SRC (solvent retention capacity) profile (AACC 56-11) [30] was based on the reaction of individual components (gluten, damaged starch, arabinoxylans-pentosans) with water and aqueous solvents of sucrose, sodium carbonate or lactic acid.

All the determinations were carried out in duplicate and were expressed on dry weight basis. Fourier transform infrared spectra (FT-IR) of flours and their blends were measured and processed by principal component analysis (PCA).

2.3 FT-IR Spectroscopy and Statistical Analyses

Fourier transform infrared spectra (FT-IR) of flours and their blends were measured in the wavenumber region of 4,000-400 cm^{-1} (mid infrared region) in KBr tablet, transmittance mode and at the resolution of 2 cm^{-1} . Collected spectra were processed by PCA within the 1,200-800 cm^{-1} region (software package Statistica, version 7.1, StatSoft CR, Czech Republic). The relationships among the different analytical parameters of analyzed flours were tested by a simple correlation (Pearson correlation). Significant differences were declared at $P < 0.05$.

3. Results and Discussions

3.1 Breadmaking Characteristics of Flours

The higher content of ash had barley bran (3.8%) and blends (1.1%) (all values are related to dry matter) (Table 2). The content of total starch was determined from 62% to 73%. The average content of protein in flours was 12.7%, whereas the highest content of protein (15.2%) was found in sample no. 25 (wheat Moravian flour) and the lowest content (7.5%) was in sample no. 17 (wheat: rye Czech flour). The samples of Czech flours (samples no. 19 to 22) had higher content of protein in comparison to samples of Moravian flours (samples no. 23 to 34). The values of GI, Zeleny sedimentation and SRC of lactic acid (expression of glutelins quality) were not significantly distinct for wheat Czech and Moravian flours. An

Table 2 Breadmaking characteristics of studied samples (values are expressed in % in dry matter).

Sample No.	Moisture	Ash	Starch	Protein	GI	Zeleny test	SRC water	SRC lactic acid	SRC sodium carbonate	SRC sucrose
1	12.7	0.4	72	12.6	96	31	64	132	76	100
2	12.1	0.6	69	12.3	92	31	65	104	82	99
3	12.0	0.8	69	12.0	94	26	68	94	88	102
4	11.8	0.9	67	11.8	89	25	71	92	91	105
5	11.6	1.0	66	11.6	90	20	73	88	96	106
6	11.6	1.1	62	11.4	97	17	77	91	100	108
7	11.0	1.1	69	11.1	66	13	83	105	114	124
8	11.3	1.1	70	10.7	72	12	81	102	110	120
9	11.5	1.1	70	10.5	75	14	82	97	108	117
10	13.6	1.0	69	10.9	imp.	25	91	106	120	133
11	11.5	3.8	69	8.9	imp.	18	134	134	148	197
12	12.1	0.9	70	6.7	imp.	25	76	96	98	122
13	10.9	1.9	70	9.4	imp.	43	89	113	129	124
14	11.6	0.7	60	8.0	imp.	23	121	126	155	153
15	14.3	0.5	65	9.8	92	31	86	162	103	105
16	14.0	0.5	65	9.5	84	30	77	177	97	102
17	8.9	0.7	63	7.5	imp.	30	149	141	135	118
18	13.7	0.8	62	11.4	imp.	30	95	169	118	125
19	14.3	0.5	62	13.5	96	44	84	184	108	121
20	14.0	0.5	67	12.2	87	39	80	183	90	82
21	12.5	0.5	66	13.6	85	48	83	182	108	128
22	14.0	0.5	65	13.1	96	40	84	186	111	106
23	14.2	0.5	73	8.3	96	31	87	175	99	136
24	15.1	0.5	73	9.6	97	39	78	182	89	131
25	15.2	0.5	74	9.3	96	41	85	172	100	136
26	13.1	0.5	70	9.7	75	31	86	163	102	122
27	13.5	0.4	70	8.8	93	35	81	169	107	113
28	12.4	0.5	70	10.7	83	30	84	156	105	118
29	13.6	0.5	66	9.5	97	35	80	159	103	115
30	12.6	0.5	67	11.1	80	31	79	163	101	124
31	13.0	0.6	69	8.8	87	30	85	170	105	125
32	13.0	0.5	70	9.9	81	31	82	160	99	122
33	15.0	0.4	68	10.6	95	30	80	166	95	142
34	11.5	0.7	68	10.4	81	31	84	186	120	143

GI: gluten index;

SRC: solvent retention capacity;

imp.: it was impossible to analyze;

Data are averages of duplicates, presented as % of dry weight except for Zeleny test value, which is expressed in mL.

addition of barley and rye to wheat flour caused a decrease of protein content in blends (decrease of value of Zeleny test and gluten index) and pronounced deterioration of breadmaking quality of flours. Wheat flours for bread production showed the higher content of gluten (Zeleny test, SRC of lactic acid) in comparison with blends. Wheat flours and blends did

not show different value of gluten index.

Solvent retention profile of selected flours and their blends is showed in Fig. 1. The value of SRC of lactic acid characterized the high content of glutenins (high molecular fraction of gluten) in wheat flours and the low content of hordenins in barley flours (Table 2). The highest content of protein, glutenin (determined by

Fig. 1 Solvent retention capacity profile of selected flours and their blends.

SRC of lactic acid) and glutenin fraction was determined in wheat Czech flour (sample no. 1). The content of glutenin decreased both in wheat and barley blends (samples no. 1 to 5) and in wheat, barley and rye blends (samples no. 7, 8 and 9). It corresponded to the decrease of protein content determined by Kjeldahl. The mentioned tendency corresponds to the results of fractionation (especially the content of glutelins).

The increased value of SRC of sucrose indicated higher content of arabinoxylans in rye (sample no. 14) and barley flour (sample no. 12). An addition of barley and rye to wheat flour resulted in increase of nutrition value of blends. The rye flour had higher value of SRC of sodium carbonate; it can mean more damaged starch and higher water absorption. An addition of barley and rye to wheat flours increased the value of SRC of sodium carbonate in blends. The rye flour had the highest value of SRC of water. The highest of water absorption was caused by the present of water-soluble components (β -glucans and arabinoxylans). On the contrary wheat flours had the

lowest water absorption (low value of SRC of water).

3.2 Nutritional Characteristics of Flours

Generally, wheat flours had the lowest content of dietary fiber and β -glucans (Table 3). An addition of barley or rye increased the content of fiber and β -glucans in blends. Brennan [5] and Rakha [18] described that the content of β -glucans is about 1% in wheat grains, 1%-2% in rye and 5%-11% in barley. In our case, barley flour contained from 2.5% of β -glucan (sample no. 10) to 4.2% (sample no. 13). The highest content of total dietary fiber had blended flours (samples no. 6-9), barley flour (sample no. 13) and rye flour (sample no. 14). The insoluble dietary fiber involved a predominant part of total fiber.

The portion of protein fractions of Czech flours and blends were not significantly different (Table 3). Only, rye flour (sample no. 14) had evidently higher content of albumins and globulins in comparison with other cereals. The most often published wheat and barley protein fraction proportions are 20%-25%, 40%-50%,

Table 3 Nutritional characteristics of studied samples (values are expressed in % in dry matter).

Sample No.	β -glucan	IDF	SDF	TDF	Albumins and globulins	Gliadins	Glutenins
1	0.2	1.9	1.4	3.0	19.8	31.7	48.4
2	1.0	3.2	1.4	4.7	20.7	33.9	45.5
3	1.6	3.0	1.9	6.3	22.1	33.8	44.1
4	1.8	2.1	2.4	6.0	22.6	34.0	43.4
5	2.3	5.4	2.2	7.1	23.5	33.9	42.6
6	2.7	5.2	3.1	8.5	24.8	33.9	41.3
7	2.9	4.9	2.9	9.2	28.4	32.9	38.7
8	2.7	5.5	3.1	9.0	27.4	33.1	39.5
9	3.1	5.3	3.2	9.1	26.1	33.3	40.5
10	2.5	2.4	1.5	4.1	27.2	26.3	46.5
11	3.0	6.5	3.7	9.8	26.1	26.9	47.0
12	2.7	2.1	1.8	4.2	26.4	26.8	46.8
13	4.2	4.0	2.6	7.0	26.1	26.6	47.3
14	1.3	4.0	2.9	7.9	43.9	25.8	30.3
15	0.1	0.9	0.4	1.5	19.1	35.5	45.4
16	0.1	1.0	0.4	1.5	19.3	36.2	44.5
17	1.1	1.8	1.3	3.1	34.1	24.4	41.5
18	1.3	1.7	1.4	3.2	21.2	30.7	48.1
19	0.5	1.7	1.4	3.2	21.8	33.1	45.2
20	0.5	1.8	1.5	3.2	23.2	33.0	43.8
21	0.6	1.6	1.3	3.2	22.5	30.0	47.5
22	0.5	1.9	1.2	3.2	23.1	33.3	43.6
23	1.1	1.6	1.3	3.2	19.4	35.0	45.6
24	1.2	1.7	1.5	3.1	21.5	36.8	41.7
25	1.1	1.8	1.3	3.0	23.3	39.7	37.0
26	1.1	1.7	1.0	3.0	22.0	28.2	49.8
27	1.1	1.7	1.2	3.0	22.2	37.0	40.7
28	1.0	1.6	1.3	2.9	27.0	30.8	42.2
29	1.2	1.8	1.5	3.0	24.2	15.4	60.4
30	1.1	1.8	1.4	3.1	24.9	14.3	60.8
31	1.0	1.8	1.5	3.1	27.1	12.9	60.0
32	1.1	1.6	1.3	3.0	20.9	32.7	46.4
33	1.0	1.7	1.2	2.9	21.9	35.1	43.0
34	1.2	1.9	1.7	3.2	21.2	40.7	38.1

SDF: soluble dietary fibre;

IDF: insoluble dietary fibre;

TDF: total dietary fibre.

and 35%-40% for albumins and globulins, gliadins and glutenins, respectively [12]. Different fraction proportion is showed in rye: albumins 44%, globulins 10%, gliadins 21% and glutelins 25%. Some Moravian flours came from same place of origin (e.g. samples no. 23-25, or samples no. 29-31) and they had comparable contents of protein fractions. The higher content of glutenins and the lowest content of gliadins were found in samples no. 29, 30 and 31. The content of glutelins decreased in blends in accordance with a decrease of total protein content determined by Kjeldahl.

Statistically significant positive correlations were

observed between β -glucan and protein ($r = 0.85$, $P < 0.05$), β -glucan and SRC of sucrose ($r = 0.93$, $P < 0.05$), between β -glucan and soluble dietary fiber ($r = 0.88$, $P < 0.05$), and β -glucan and gluten value of Zeleny test ($r = 0.81$, $P < 0.05$). A strong positive correlation between the content of β -glucan and protein and between β -glucan and soluble fiber was confirmed with the results obtained by Holtekjølén [33]. Positive correlations were found between soluble dietary fiber and SRC of water ($r = 0.95$, $P < 0.05$), between proteins and total dietary fiber ($r = 0.88$, $P < 0.05$), and SRC of lactic acid and gluten value of

Fig. 2 PCA score scatter plot of the FT-IR spectra of flours in the 1,200-800 cm^{-1} wavenumber region. The clusters included the individual wheat flours, barley, rye flours and blends on the basis of starch and non-starch polysaccharides content.

Zeleny test ($r = 0.70$, $P < 0.05$). Significant negative correlations were observed between content of β -glucan and starch ($r = -0.69$, $P < 0.05$) and between proteins and starch ($r = -0.83$, $P < 0.05$).

3.3 FT-IR Spectroscopy and Principal Component Analysis

The transmission FT-IR spectra of flours and blends were recorded. The characteristic region of starch, non-starch polysaccharides and protein absorption was observed. PCA was used on IR data and PCA score allowed to separate the flours and their blends according to the content of starch and non-starch polysaccharides, especially β -glucan and fiber content. The flours were separated to three clusters (wheat, blends, barley and rye) (Fig. 2).

The application of a PCA to the FT-IR spectra in the mid-infrared region was effective for fast distinction of various different types of flours on the basis of nutritional and breadmaking flour parameters.

4. Conclusions

An addition of barley or rye to wheat flour influenced the quality of flour blends significantly. The content of nutritive and healthy components

(soluble proteins, fiber, β -glucans and pentosans) was increased in blends, but the breadmaking quality decreased (higher content of damaged starch, higher water capacity and sticky dough).

FT-IR spectroscopy and chemometric analysis (PCA analysis) can be used to a fast screening of the cereal flour quality and to classification of flours on the basis the specific purpose of their uses.

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