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CHEMICAL AND NUTRITIONAL CHARACTERISTICS OF HIGH-FIBRE RYE MILLING FRACTIONS

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ABSTRACT

Background. Many studies have demonstrated the potential health benefits of consuming more high-fibre cereal-based food products. Therefore, there is a need to discover new ways to improve the overall nutritional balance of refined cereal products and focus on increasing their dietary fibre content, at the expense of readily digestible carbohydrates.
Material and methods. Lab-scale milling and sieving of whole rye grain was used to obtain two fractions rich in dietary fibre. The fractions were analysed and compared, in terms of microstructure, chemical composition and nutritional quality.
Results. The two fractions significantly obtained differed in their particle size and contents of minerals, available saccharides, and nutritional fractions of starch and dietary fibre and its major components. The total dietary fibre concentrations in the coarse and fine fractions were 50.0 and 36.0 g/100 g, respectively, i.e. three and 2.2 times higher than that of wholegrain rye flour. Both fractions also differed in their relative proportions of major fibre components. In the fine fraction, the levels of soluble fibre, as well as soluble arabinoxylans and fructans, were significantly higher than those in the coarse fraction.
Conclusions. It was shown that the application of a simple dry-fractionation method to wholemeal rye flour allows the preparation of two rye products which can serve as concentrated sources of dietary fibre low in available saccharides.

Keywords: rye grain, dry fractionation, high-fibre milling fraction, dietary fibre components, proximate composition, nutritional quality

INTRODUCTION

Many studies have shown the relationship between a high intake of dietary fibre (DF) and the reduced risk of developing many lifestyle-related ailments (Slavin, 2003; Ye et al., 2012). According to the current recommendations of the European Food Safety Authority, the average daily requirement of DF is 25 g per day for an adult (EFSA, 2010). Many nutritionists and diet experts suggest that about 20–30% of this amount should be soluble (SDF) (Elleuch et al., 2011). In a large number of developed Western countries, a deep asymmetry is observed between recommendations for the daily intake of DF, and the low consumption of cereal products rich in DF amongst the general population (Slavin, 2003). This is due to the fact that

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in these countries, including Poland, most cereal-based food products are made with refined kernel endosperm from which the peripheral layers are excluded, despite the fact that these anatomical parts of the kernel have significant nutritional potential and contain most of the DF, micronutrients and phytochemicals of the kernel (Slavin, 2003). Therefore, there is a need to develop new high-fibre cereal products which are attractive to consumers, including cereal-based food ingredients that could be universally recommended as part of a healthful diet.

Among commonly grown cereals, especially in Poland, rye seems to be the best raw material for this type of cereal product, as it has a high DF content in its major components, such as arabinoxylan and fructan (Bach Knudsen and Lærke, 2010; Hansen et al., 2003; Karppinen et al., 2003). Although the β-glucan content of rye is lower than that of barley and oats, it is still almost twice as high as that of wheat (Wood, 2010). Due to the high levels of FRU and the water-soluble fraction of arabinoxylan, rye contains large amounts of SDF. In addition, rye DF components are accompanied by a number of bioactive compounds such as phenolic acids, alkylresorcinols, lignans, phytosterols, minerals and vitamins like tocopherols, tocotrienols and folates etc. (Slavin, 2003; Nyström et al., 2008). It is well documented that the major components of rye DF are mainly located in the outer layers of the kernel, which consists of the outer and inner pericarp, the testa, the hyaline layer and the aleurone and subaleurone layers (Glistø and Bach Knudsen, 1999; Härkönen et al., 1997).

To prepare cereal-based products with enhanced levels of DF or its single components (e.g. β-glucan or arabinoxylan), many different wet and dry fractionation technologies have been developed. Wet processes pose many technological problems, and they are expensive (Izydorczyk et al., 2006). Dry fractionation processes are more sustainable and economically more attractive. They are based on progressive grinding of cereal grains, wholegrain flour or bran to decrease the particle size, and then separating the fractions which contain most of the peripheral tissues rich in DF. Many experiments have verified the usefulness and efficiency of various grain pre-treatments before milling or grinding (e.g. tempering, cryogenic pre-treatment, degerming, dehulling, peeling, pearling etc.), different grinders and mills (e.g. roller, impact, hammer, ball, abrasive, cryogenic, ultrasonic etc.) and sorting techniques (e.g. sieving, air-classification, electrostatic separation etc.; Hemery et al., 2007). A combination of wet and dry technologies has also been used (Nordlund et al., 2013b). Most of these studies are related to wheat, barley and oats, but only a few have focused on dry fractionation of rye (Glistø and Bach Knudsen, 1999; Härkönen et al., 1997; Nordlund et al., 2012; 2013a). However, some of these innovative technologies require costly, high-tech specialised equipment, such as superfine milling installations, cryogenic mills or free-fall electrostatic separators.

The purpose of the present study was to evaluate the potential of a simple dry fractionation method using milling and sieving to obtain rye fractions enriched in DF. The chemical composition of the obtained high-fibre products was determined and their nutritional quality was evaluated.

MATERIAL AND METHODS

Materials and sample preparation (grinding and sorting)

The commercial hybrid rye grain cultivar 'Visello', harvested in 2011 in Poland, was used in this study. We chose only one variety, based on three-year pilot studies from our laboratory. Cleaned grains tempered to a moisture content of 14.5% were milled with a laboratory roller mill (Quadrumat Senior, Brabender GmbH, Germany) at the default settings. The obtained wholemeal flour (WF) was additionally passed once through break rollers and then twice through reduction rollers of the mill without sieving. The received fine WF was separated in a sieve shaker (AS 200 Basic, Retsch GmbH, Germany) on a sieve with 50 mm openings to produce a coarse fraction 1B (≥50 mm) and a fine endosperm rich fraction 1A (<50 mm) for 15 min. The fraction 1A was discarded, whereas the fraction 1B was reground in a laboratory ball mill type-6 (LZBM, Poland) for 180 min. The porcelain ball and bowl diameters were 2.4 cm and 22.5 cm respectively, and the internal bowl depth was 22.5 cm. The fraction 1B was sieved again on a sieve with 50 mm openings into two subsequent fractions: 2A (<50 mm) and 2B (≥50 mm). The fraction 2A was removed, whereas the fraction 2B was separated on sieves with openings of

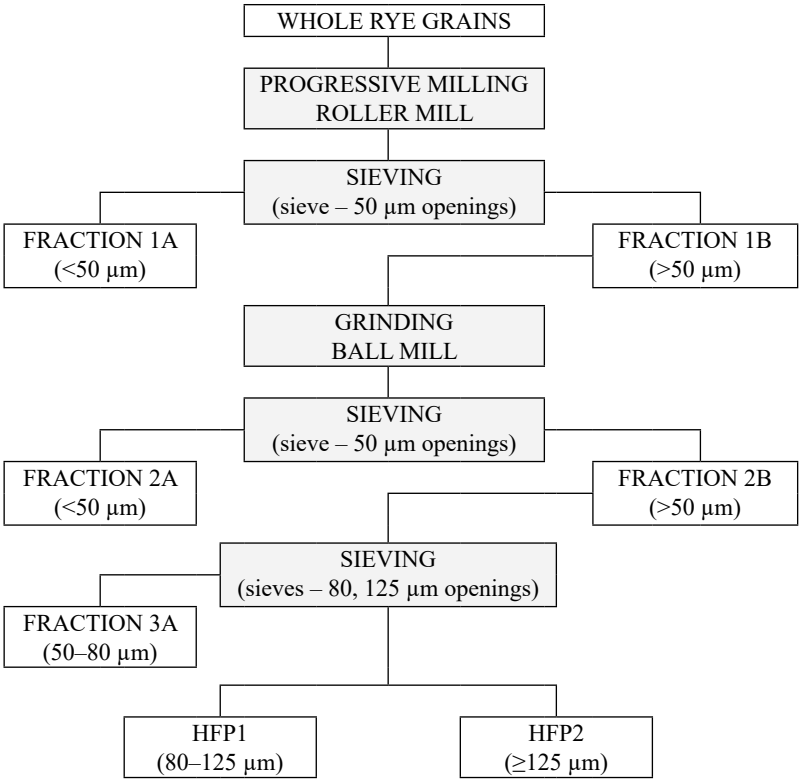

Fig. 1. Flow diagram of rye grain dry fractionation for the preparation of high-fibre products

80 mm and 125 mm into three fractions: 3A, HFP1 and HFP2. The fraction 3A (50–80 mm) was eliminated, while the HFP1 fraction (80–125 mm) enriched in SDF and the HFP2 fraction (≥125 mm) enriched in insoluble fibre (IDF) comprised the final products (Fig. 1).

Analytical methods

The main chemical components were determined according to AACC Approved Methods and ICC Standards: dry matter (ICC-Standard No. 110), ash (ICC-Standard No. 104), protein (AACC Approved Method 46–10), lipids (AACC Approved Method 30–10) and total starch (TS) (AACC Approved Method 76–13) (AACC, 2000; ICC, 1998). The contents of free glucose (FG), rapidly and slowly digestible starch (RDS and SDS respectively) and resistant starch (RS) were determined with the method described by Englyst et al. (1992), while total reducing saccharides, after two-step 80% methanol extraction, were colorimetrically determined using 3,5-dinitrosalicylic acid, according to the method described by the same authors. The AACC procedure was carried out to determine soluble fibre (SDF) and insoluble fibre (IDF) (AACC Approved Method 32-07) using the Megazyme total dietary fibre assay kit K-TDFR-200A (Megazyme International Ireland Ltd., Wicklow, Ireland). The total dietary fibre (TDF) content was calculated as a sum of SDF and IDF. The contents of DF components were determined as follows: total arabinoxylan (AX) and its water-soluble fraction (WE-AX) according to the method of Hashimoto et al. (1987), total β-glucan (BG) and its water-insoluble fraction (WUE-BG) according to the ICC Standard No. 168 and fructan (FRU) with the AACC Approved Method 32-32. The content of water-insoluble arabinoxylan (WUE-AX) was calculated as the difference between the contents of total AX and WE-AX, while the content of water-soluble β-glucan (WE-BG) was calculated as the difference between the total BG and WUE-BG contents. The BG

and FRU contents were analysed using the Megazyme β-glucan mixed linkage and fructan assay kits, K-BG -LU and K-FRUC, respectively. The amount of total phenolic compounds (TPC) in the methanol extracts was determined using the Folin-Ciocalteu method according to Singleton et al. (1999). All analyses were run in triplicate.

Microscopy

The microstructure of HFP1 and HPF2 was examined by using the Q Imaging-Go3 camera and the Image--Pro Plus software Media Cybernetics.

Statistical analysis

The results are presented as mean ±standard deviations. For multiple comparisons, one-way analysis of variance (ANOVA) was used. Differences between means were tested for significance using Duncan's multiple range test (MRT). Significance was set at $p \leq 0.05$. The statistical analysis was performed using the statistical software Statistica 8.0 (StatSoft Inc., USA).

RESULTS AND DISCUSSION

Grinding and sieving

The developed procedure made it possible to gradually increase the ratio of particles originating from the outer kernel tissues rich in DF (fractions 1B and 2B) to particles from the inner tissues rich in starch (fractions 1A, 2A and 3A). The aim of progressive grinding in roller and ball mills was the fragmentation of the cellular structures, to release the maximum possible amount of starch granules. Next, the purpose of sieving on sieve with 50 mm openings was to remove starch (fractions 1A and 2A). To increase concentration of outer tissues in the final fractions HFP1 and HFP2, sieves with openings of 80 and 125 mm were used. The 50 mm mesh screen was chosen because it allowed most of the rye starch granules to pass through. Verwimp et al. (2004) found that rye starch granules ranged from 10 to 40 mm in diameter with average particle size of 31 mm. The best biomarkers for the presence of peripheral kernel tissues are phenolic acids, alkylresorcinols and phytic acid, while the marker of the presence of endosperm tissues is starch (Hemery et al., 2009). The authors concluded that the contents of ferulic acid dehydrotrimer and p-cumaric acid can be considered markers of the outer pericarp and aleurone layer respectively, and are therefore efficient tools for the quantification of kernel tissue proportions in wheat milling fractions. Furthermore, Zieliński et al. (2007) demonstrated that the total phenolic compound content in the pericarp with testa of rye kernel is almost double that in the endosperm. Based on these observations, the usefulness of the applied dry fractionation procedure was verified by the determination of the TS and TPC contents in all fractions and WF. The ratios of TS to TPC content were also calculated (Table 1). Exclusion of the endosperm rich fraction 1A (<50 mm) meant that the fraction 1B (≥50 mm) contained 31% less TS than the initial material, and the ratio of TPC to TS increased by approximately 50% compared to WF. A relatively large amount of TS in the coarse fraction 1B was the reason for regrinding it in a ball mill to release the rest of the starch granules from the cellular structures, and then sorting into two consecutive fractions; 2A (removed) and 2B. To further increase the concentration of outer kernel tissues, fraction 2B was separated into three fractions: 3A (50–80 mm), HFP1 (80–125 mm) and HFP2 (≥125 mm). Fraction 3A was

Table 1. Yield, content of total starch (TS) and phenolics compounds (TPC) and the ratios of TPC to TS in wholemeal flour and different fractions from dry fractionation process of rye grain

Products	Yield, %	Total starch, % DM	Total phenolics FAE, mg%	Ratio of TPC/TS
1	2	3	4	5
Roller mill				
Wholemeal flour (WF)	100.0	59.8^{e} ±0.5	132^{c} ±5	1 : 453
1A (<50 μm)	19.2 ±1.4	88.8^{h} ±1.0	39^{a} ±2	1 : 2278
1B (≥50 μm)	80.8 ±1.4	50.7^{d} ±0.6	168^{d} ±16	1 : 302

Table 1 – cont.

1	2	3	4	5
Ball mill				
2A (<50 µm)	16.6 ±0.8	80.3^{g} ±1.2	87^{b} ±3	1 : 923
2B (≥50 µm)	64.2 ±0.8	40.3^{c} ±0.2	213^{e} ±9	1 : 189
3A (50–80 µm)	21.8 ±0.9	67.1^{f} ±0.1	122^{c} ±7	1 : 550
HFP1 (80–125 µm)	16.8 ±1.0	38.1^{b} ±0.5	176^{d} ±13	1 : 217
HFP2 (≥125 µm)	25.6 ±0.6	20.0^{a} ±0.1	287^{f} ±11	1 : 70

Mean value ±standard deviation.
FAE – ferulic acid equivalents.
Values in the same columns followed with the different letters are significantly different ($P \leq 0.05$).

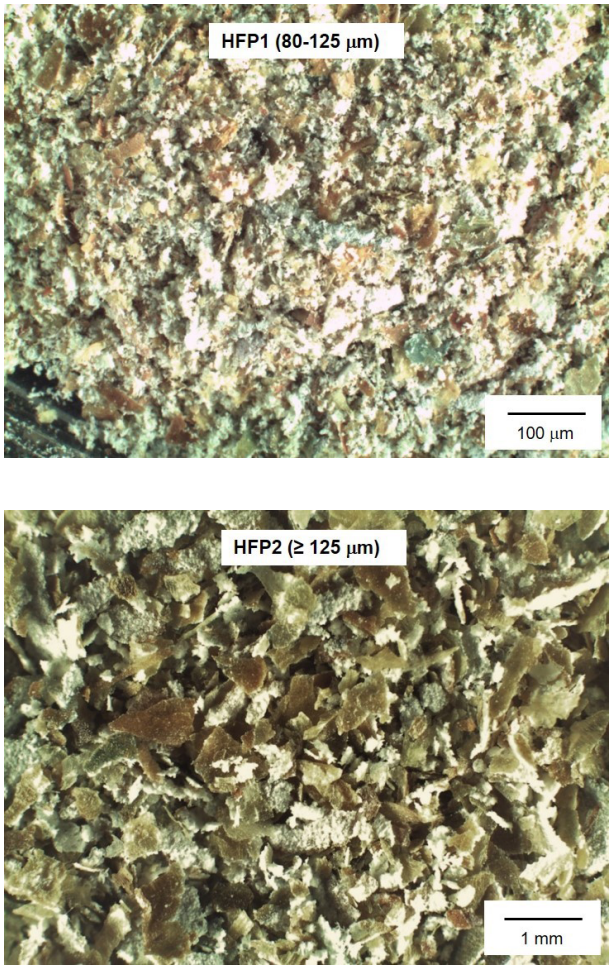

Fig. 2. Microstructure of high-fibre products (HFP1 and HFP2) imaged by stereomicroscope

discarded, whereas the two other fractions, HFP1 and HFP2, yielded 16.8 and 25.6% of grain initial weight respectively, and had significantly lower TS contents and increased proportions of outer to inner tissue content when compared to WF. The residual TS contents in the HFP1 and HFP2 fractions, as a proportion of those originally present in the WF, were 10 and 8% respectively, whereas the ratios of TPC to TS in HFP1 and HFP2 were respectively double and 6.5-times higher than that in the raw material. The average particle sizes were 102 µm and 427 µm for the fine fraction HFP1 and the coarse HFP2 respectively.

Microstructure

High-fibre products (HFP1 and HFP2) were examined by stereomicroscope (Fig. 2). A comparison of the micrographs showed that both fractions differed in the composition of their particles. It was observed that HFP2 contained more lignified fragments of pericarp/testa cell walls rich in DF components, and fewer particles consisting of starch-rich endosperm, in comparison to HFP1. The coarse fraction was similar to rye bran, while the fine fraction looked like the products of the final reduction passages of rye industrial milling.

Dietary fibre and its components

As expected, HFP1 and HFP2 differed significantly in their contents of TDF and its major components (Table 2). The coarse fraction and the fine fraction were respectively 2.7 and 1.8 times richer in TDF than the WF. However, the content of SDF in the HPF1 was twice as high as in HPF2. In both fractions, the AX

Table 2. Content and composition of dietary fibre in wholemeal rye flour (WF) and high-fibre products (HFP1 and HFP2), % of DM

Dietary fibre and its components	WF	HFP1	HFP2
Total dietary fibre (TDF)	18.4^{a} ±0.4	33.1^{b} ±0.8	49.5^{c} ±0.3
– soluble (SDF)	3.9^{a} ±0.1	12.4^{c} ±0.5	5.8^{b} ±0.3
– insoluble (IDF)	14.5^{a} ±0.4	20.7^{b} ±0.6	43.7^{c} ±0.4
Total arabinoxylan (AX)	9.3^{a} ±0.5	15.4^{b} ±0.9	19.8^{c} ±0.6
– water-soluble (WE-AX)	4.0^{a} ±0.1	7.4^{b} ±0.3	3.7^{a} ±0.3
– water-insoluble (WUE-AX)	5.3^{a} ±0.3	8.0^{b} ±0.5	16.1^{c} ±0.6
Total β-glucan (BG)	2.1^{a} ±0.1	3.8^{b} ±0.2	4.1^{b} ±0.2
– water-soluble (WE-BG)	1.1^{a} ±0.0	1.5^{b} ±0.1	1.2^{a} ±0.1
– water-insoluble (WUE-BG)	1.0^{a} ±0.1	2.3^{b} ±0.2	2.9^{c} ±0.1
Fructan (FRU)	4.5^{a} ±0.2	7.2^{c} ±0.1	6.5^{b} ±0.3

Mean value ±standard deviation.
Values in the same columns followed with the different letters are significantly different ($P \leq 0.05$).

were the quantitatively most significant DF components. The amounts of AX in HFP2 and HFP1 were greater than in the raw material, i.e. 2.1 and 1.7 times higher respectively. Moreover, HFP2 contained twice as much WUE-AX as HFP1, whereas the fine fraction was evidently richer in WE-AX. It is well known that WUE-AX is the primary structural cell wall component of the rye kernel pericarp/testa tissues, whereas WE-AX are mainly located in endosperm cell walls (Bach Knudsen and Lærke, 2010). The contents of BG did not differ significantly between the two fractions and were about twice as high as in WF. However, the difference between HFP1 and HFP2 was evident in the WE-BG contents. HFP1 was 20% richer in WE--BG than HFP2. Moreover, the fine fraction contained 10% more FRU than the coarse fraction. These significant differences may result from a dissimilar FRU to AX and BG distribution in the peripheral tissues of the rye kernel. Kuhlmann et al. (2002), analyzing rye milling products, showed that the fine bran obtained from reduction passages contained almost twice as much FRU as the raw material, while its content in the coarse bran obtained from break passages remained at a similar level to in the whole grain.

Nutritional quality of the high-fibre products

The contents of macronutrients in both fractions are summarised in Table 3. Both products were a good source of minerals and protein. The contents of minerals in HFP1 and HFP2, compared to the WF, were higher slightly; more than 1.5- and about 2.5-times respectively. It is expected that their addition to the human diet will significantly contribute to increased intake of the recommended macro- and microelements The protein contents in both fractions were similar, but about 70% higher compared to WF. Rye protein does not have a high biological value; however, the amount of protein originating from the peripheral layers of the kernel is several times higher than that of the endosperm. It is known that functional proteins are present in the external tissues of the rye kernel, whereas the majority of proteins in the inner endosperm are storage proteins. Similarly, there were no significant differences in the lipid contents in HFP1 and HFP2, but these were about 50% higher than in WF. This increase in fat content may be disadvantageous, as it reduces the shelf life of the product and raises its caloric value. On the other hand, it is preferable due to the higher content of unsaturated fatty acids, tocotrienols

Table 3. Content of macronutrients in high-fibre products (HFP1 and HFP2), g/100 g of product as eaten

Component	HFP1	HFP2
Water	10.6^{a} ±0.1	10.7^{a} ±0.1
Ash	3.1^{a} ±0.1	4.9^{b} ±0.2
Fat	2.6^{a} ±0.4	2.7^{a} ±0.2
Protein (N×6.25)	16.1^{a} ±0.4	16.8^{b} ±0.2
Total saccharides	30.4^{b} ±0.7	21.0^{a} ±0.4
– available saccharides	29.2^{b} ±0.5	19.1^{a} ±0.1
Total starch (TS)	27.6^{b} ±0.6	17.9^{a} ±0.1
– rapidly digestible starch (RDS)	17.7^{b} ±0.3	10.7^{a} ±0.2
– slowly digestible starch (SDS)	8.7^{b} ±0.3	5.3^{a} ±0.1
– resistance starch (RS)	1.2^{a} ±0.4	1.9^{a} ±0.2
Total reducing saccharides	2.8^{a} ±0.1	3.1^{b} ±0.1
– free glucose (FG)	1.5^{a} ±0.2	1.6^{a} ±0.0
Total dietary fibre (TDF)*	36.0^{a} ±0.4	50.0^{b} ±0.3
– soluble (SDF)*	17.5^{b} ±0.2	11.0^{a} ±0.3

Mean value ±standard deviation.
Values in the same columns followed with the different letters are significantly different ($P \leq 0.05$).
*The amounts of TDF and SDF include amounts of FRU.

and phytosterols accompanying these lipids in rye milling products (Slavin, 2003).

The main advantage of HFP1 and HFP2 was the low content of available saccharides, defined as the sum of available starches (RDS and SDS) and total reducing sugars, with a concurrent high TDF content. In 100 g of HFP2 as eaten, only 19.1 g of available saccharides were present, of which 56% was RDS. A significantly higher content of available saccharides was found in HFP1. It contained 29.2 g/100 g, of which 60% was RDS. The available starch content and the proportions of RDS in TS contents in HFP2 presented here were lower than those found in cereal bran. Englyst et al. (1996) showed that the contents of available starch in cereal brans ranged from 21.1 to 44.8 g/100 g of product as eaten, whereas the proportions of RDS in the total available glucose contents of wheat, rye and oat bran was about 65%. Moreover, the same authors analyzing the nutritionally important starch fraction contents of 39 starchy foods and glycaemic indices (GI) have reported a highly significant positive correlation between GI and both RDS and rapidly available glucose (RAG). A high RAG content in cereal-based products causes a rapid increase in plasma glucose and insulin levels after ingestion, which might be associated with several health complications, including diabetes, cardiovascular disease and obesity. For many people with diabetes, carbohydrate exchange is the most important element of meal planning. According to the Polish definition, one carbohydrate exchange (1 CE) corresponds to 10 g of available carbohydrates. One serving (assumed to be 15 g, i.e. one cup) of HFP1 delivers 0.5 CE, whereas HFP2 delivers only 0.3 CE. In comparison, one serving of wholegrain product, generally recommended in diets for overweight individuals and diabetics, contains about 1–1.5 CE. Moreover, Ragaee et al. (2011) found strong correlations between contents of both IDF and SDF and contents of both RDS and SDS in wheat breads enriched in fibre. The TDF content in 100 g of HFP2 as eaten was 50.0 g, of which 11.0 g was the SDF, whereas in 100 g of HFP1 – 36.0 and 17.5 g respectively. This means that approximately 3.5 cups of HFP2 or 5 cups of HFP1 fully cover the recommended daily intake of TDF, as well as 75% and 170% of the required daily intake of SDF respectively. The amounts of the TDF and the SDF presented here in the both products include amounts of FRU, which is currently classified as a component of DF. The TDF content in HFP2 as well as the SDF content in HFP1 were significantly higher than those found in a previous study on rye bran (Nordlund et al., 2012; 2013a). Rye bran, eliminated during industrial milling of grains, is generally considered a concentrated source of DF and is often recommended by doctors and diet experts as part of a healthful diet. Kołodziejczyk and Michniewicz (2018) demonstrated that TDF contents in Polish commercial rye bran ranged from 23.2 to 36.2 g/100 g of product as eaten, of which 7.5–8.2 g was SDF when FRU content was included.

Potential application

According to current EU regulations concerning high-fibre products, to claim that a food product is "high in fibre" there should be at least 6 g of fibre per 100 g

(or 3 g of fibre per 100 kcal; European Commission, 2006). The TDF contents in the both obtained rye fractions exceeded those required quantities several times over, which is why the fine fraction was called "high-fibre product 1" and the coarse fraction "high--fibre product 2". The coarse fraction, as concentrated source of DF, especially of IDF, can be used as a potential additive to dairy products (e.g. milk, kefir, yogurt, buttermilk, etc.), as well as to soups, salads or muesli-type products. The fine fraction, with a very high SDF content, can be used as an additive to bread, pasta, muffins/cakes, extruded snacks, confectionery, etc. Moreover, HPF1 may be mixed with HFP2 with different quantitative ratios, while the obtained blend may be added to many food products as a concentrated source of IDF and/or SDF.

CONCLUSIONS

It was demonstrated that the application of a simple dry-fractionation method, using repeated milling and sieving of the wholemeal rye flour, allows the preparation of two fractions substantially enriched in DF. The coarse fraction, with an average particle size of 427 μm, and the fine fraction, with the average particle size of 102 μm, gave a satisfactory yield and respectively contained 50.0 and 36.0 g of TDF in 100 g of products as eaten, i.e. 3.0 and 2.2 times higher than that of wholegrain rye flour. Both fractions were characterized by various quantitative and qualitative compositions of the major components of DF. The TDF content in the coarse fraction, which had a higher concentration of outer kernel tissues, was accompanied by a rise in the amount of insoluble DF components. However, the fine fraction, which contained fewer pericarp/testa particles, had larger amounts of soluble DF. The high content of DF in the both fractions allows them to be considered high-fibre cereal-based products. Both high-fibre products, due to their advantageous nutrient composition, may be a valuable addition to the human diet, not only in terms of dietary fibre but also in minerals and protein. They may be useful in the prophylaxis and/or treatment of the so-called 'diseases of civilization'.

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