Dietary Fiber Content and Composition in Six Cereals at Different Extraction Rates

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ABSTRACT

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An enzymatic method was used to examine the relationship between extraction rate and dietary-fiber content in flours from wheat, rye, barley, sorghum, rice, and corn. Results were compared with neutral-detergent, acid-detergent, and crude-fiber determinations. Dietary-fiber content in wheat was constant for extraction rates between 65 and 80%, and then increased linearly with the extraction rate. Dietary fiber content in rice and to some extent in barley had a similar relationship, whereas in rye and sorghum it increased gradually with the extraction rate. Soluble dietary-fiber content was constant in each cereal, regardless of the extraction rate, and constituted almost half of the dietary fiber in wheat, rye, and barley flours of low extraction rate. The relative monomeric composition of the dietary fiber in wheat, rye, and corn was similar at the highest and the lowest rates of extractions, with xylose-arabinose ratios being 0.9–1.5. In barley and rice, the relative xylose content increased with the rate of extraction, whereas in sorghum, both arabinose and xylose content increased. Acid-detergent and crude fiber comprised only a fraction of insoluble and of total dietary fiber. The slope of the regression equation between insoluble dietary fiber and neutral-detergent fiber amylase was 0.8–1.0 for wheat, rye, barley, and rice and 1.2 for corn. Detergent methods applied to sorghum gave obscure results.

Cereal grains are important sources of digestible and unavailable carbohydrates. Cellular areas include the pericarp, testa, germ, aleurone, and endosperm (MacMasters et al 1978, Munck 1981a). Barley and rice kernels are surrounded by a husk (palea and lemma) that, in other cereals, is separated from the kernel during threshing (Barber et al 1981, Munck 1981b). Highly refined white flour consisting mainly of endosperm can be produced if the fibrous outer layers are removed during milling. Thus, dietary-fiber content of flours is determined by rate of extraction.

The analysis of dietary fiber in foods is a controversial subject. Although gas-liquid chromatographic (GLC) and colorimetric methods provide more information, they are too complicated and time-consuming to be used for food labeling and control; thus, for such purposes, gravimetric methods are generally used. In the present study, dietary fiber was measured with a recently developed enzymatic gravimetric method that accounts for both soluble and insoluble components (Asp et al 1983). Soluble dietary-fiber components are quantitatively and qualitatively important in many foods (Asp 1978, Jenkins et al 1976, Stasse-Wolthuis et al 1980). Thus, gel-forming dietary fibers, such as pectins and gums, greatly affect metabolism of carbohydrate and of lipoprotein (Spiller and McPherson-Kay 1980). Cereal fiber has less consistent effects on metabolism (Asp et al 1981, Brodribb and Humphreys 1976, Jenkins et al 1978, McPherson-Kay and Truswell 1980). Soluble pentosans in wheat and rye affect baking properties (Ciacco and D'Appolonia 1982). Water-soluble β-glucans in barley are important quantitatively and influence feed utilization (Burnett 1966).

In the present investigation, variation of insoluble and soluble dietary fiber with the rate of extraction of flour from wheat, rye, barley, sorghum, and rice was studied. Different types of corn flour were also studied. The composition of dietary fiber was determined by GLC in flours having the lowest and the highest extraction rates. Finally, crude fiber, neutral-detergent fiber (NDF) (with amylase), and acid-detergent fiber (ADF) measurements were compared with determinations obtained by the enzymatic-gravimetric method.

MATERIALS AND METHODS

Materials

Hard winter wheat, rye, and spring barley harvested in Denmark in 1980 were used. Sorghum and rough rice from Italy were obtained from commercial sources. The cereals were milled into flours with extraction rates between 100 and 65%. Different types of corn flours were produced from U.S. No. 3 corn.

Wheat and rye flours were prepared by laboratory roller mills (Quadrumat Junior and Quadrumat Junior II, Brabender GmbH, Duisburg, BRD), and by sifting (Laboratory Plansifter, Bühler-Miag, Braunschweig, BRD), as described by Pedersen and Eggum (1983). Flours of barley were prepared by abrasive milling (Vertical Abrasive Polisher, Schule GmbH, Hamburg, BRD). Sorghum flours were produced by decortication (DVA decorticator, United

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Milling System A/S, Copenhagen, Denmark) and by sifting, as described by Munck et al (1982). Brown rice was prepared by passing rough rice through a Strong Scott Barley Pealer (Seedburo Equipment Co., Chicago, IL). Unshelled rough rice was removed by hand, and the milling yield of brown rice was 82%. Milled rices were prepared by decortication (DVA decorticator, UMS) and by abrasive milling (Vertical Abrasive Polisher, Schule). Milling yields, calculated on basis of rough rice, were 77, 72, 68, and 64%. Various types of corn flour were produced by dehulling (Dehuller DHA 400, UMS) after tempering with 4–5% distilled water for 10 min, disk-milling (MHA 400, UMS), and sifting (Laboratory Plansifter, Bühler-Miag). Corn flours with particle sizes of $<340\ \mu$, $<840\ \mu$, and 500–1,000 μ were prepared in this way. Degerminated corn grits were produced by gravity separation (separator type 51, Cimbria, Thisted, Denmark) of the dehulled corn. The whole grains and the cereal products were finally hammer milled (Laboratory Mill, Christy & Norris Ltd., UK).

Methods

Enzymatic gravimetric method. The total dietary-fiber content was analyzed by a gravimetric method based on digestion of the sample with enzymes, as described by Asp et al (1983). The dietary fiber can be classified into insoluble and soluble components, the sum of which is the total dietary fiber. This method was developed from that of Hellendoorn et al (1975), with the use of the physiological enzymes pepsin (Merck, no. 7190) and pancreatin (Sigma, no. P-1750). Pepsin incubation is performed at pH 1.5 for 1 hr and pancreatin incubation at pH 6.8 for an additional 1 hr. Starch digestion is improved when a heat-resistant α-amylase (Termamyl, Novo, Copenhagen) is added during the initial 15-min gelatinization. Insoluble components are recovered by filtration. Soluble components are recovered by precipitation with four volumes of 95% ethanol and filtration. The results are corrected for undigestible protein (Kjeldahl $N \times 6.25$) and ash (ignition at 525°C for 24 hr) associated with the fiber.

Other gravimetric methods. Crude fiber was determined according to AOAC methods (1980). ADF and NDF were analyzed as described by Goering and Van Soest (1970). The NDF determinations were performed with the following modifications: the cereal was suspended in a phosphate-buffer, pH 7.0, containing 2 g of α-amylase (Merck) per 500 ml of phosphate-buffer. The suspension was incubated for 15 hr at 37°C. To improve the starch digestion further, 100 μl Termamyl (60 L) was added during NDF incubation.

Gas-chromatographic determinations. The dietary-fiber composition was assayed by GLC for the neutral sugars obtained after acid hydrolysis and by a decarboxylation method for the uronic acids, as described by Theander and Åman (1979). The hydrolysis was performed with 72% (12.0 M) sulfuric acid (5 ml per 100 mg) at room temperature for 2 hr, diluted with water to 0.358 M sulphuric acid, and refluxed for 6 hr. The monomeric composition was determined only for the lowest and the highest extraction rates of each cereal. Soluble and insoluble dietary-fiber components were pooled for these analyses. All dietary-fiber components are expressed as polysaccharides, ie, constituting monomers are expressed as anhydroform.

Starch determination. Approximately 20 mg of the dietary fiber was suspended in 1 ml of distilled water and gelatinized for 15 min. Five microliters of amyloglucosidase (Boehringer Mannheim GmbH) was added, and the suspension was incubated at 60°C for 30 min. The released glucose was measured with glucose oxidase reagent (Glox-Novum, Kabi Diagnostica, Stockholm, Sweden).

All values are given in percent on a moisture-free basis and represent means of duplicate analyses.

RESULTS

Wheat

The dietary-fiber content of wheat flours ranged from 2.8 to 12.1% (Fig. 1). The soluble-fiber content was about 1.3%, independent of the degree of extraction. The content of insoluble fiber increased rapidly for extraction rates above 80%. The relative composition of the monosaccharides differed little between the two extraction rates investigated. The main components were glucose, arabinose, and xylose (Table I).

By the NDF-amylase method, approximately 70% of the total fiber and 93% of the insoluble fiber obtained with the enzymatic method can be measured (Table II). ADF and crude fiber versus insoluble fiber with the enzymatic method had intercepts at 0.7–0.8.

Rye

Rye contained from 7.5 to 16.1% dietary fiber (Fig. 2). The soluble-fiber content was about 3.8% at all extraction rates. The content of insoluble fiber increased continuously with the degree of extraction. The relative monomeric composition was similar in the flours of highest and lowest extraction. The relative distribution of monomeric sugars was similar to that in wheat (Table I).

Correlation of total fiber in rye with NDF, ADF, and crude fiber values showed intercepts indicating the large amount of soluble fiber (Table II). The NDF-amylase method gave values of about 80% of the insoluble fiber assayed enzymatically.

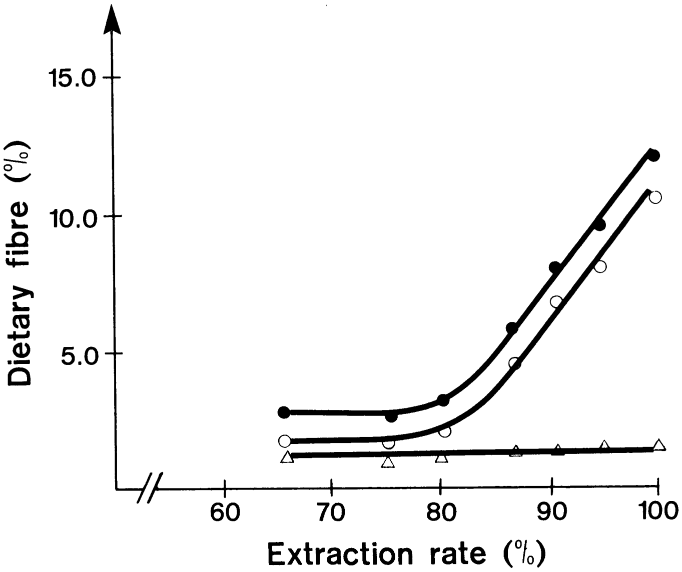

Fig. 1. Dietary fiber content in wheat flours of different extraction rates. • = total dietary fiber; o = insoluble dietary fiber; △ = soluble dietary fiber.

TABLE I
Composition of Dietary Fiber in Wheat, Rye, and Barley

	Extraction Rate (%)					
	Wheat		Rye		Barley	
	66	100	66	100	69	100
Polysaccharides (g/100 g)	2.4	9.5	6.8	12.5	7.9	15.1
Relative composition (%)						
Rhamnose	···	···	···	···	···	···
Arabinose	24	24	25	25	12	14
Xylose	34	36	31	37	14	26
Mannose	4	1	5	3	4	3
Galactose	7	3	3	3	1	1
Glucose	31	33	31	28	69	51
Uronic acids	···	3	5	4	···	5
Klason lignin (g/100 g)	···	2.0	0.3	2.1	···	3.5
Sum of dietary fiber components (g/100 g)	2.4	11.5	7.1	14.6	7.9	18.6
Dietary fiber residue[a]	2.8	12.1	7.5	16.1	8.2	18.8

[a] With the gravimetric method (sum of insoluble and soluble fraction, g/100 g).

TABLE II
Regression Equations and Correlation Coefficients Between Total Dietary Fiber and Insoluble Dietary Fiber Obtained with the Gravimetric Method (X) and Neutral-Detergent, Acid-Detergent, and Crude Fiber (Y)

Grain	Total Dietary Fiber: Regression Equations	Total Dietary Fiber: Correlation Coefficient (r)	Insoluble Dietary Fiber: Regression Equations	Insoluble Dietary Fiber: Correlation Coefficient (r)
Wheat	Y = 0.88X − 0.82[a]	0.98	Y = 0.93X + 0.06[a]	0.98
	Y = 0.35X − 0.63[b]	0.99	Y = 0.37X − 0.29[b]	0.99
	Y = 0.25X − 0.42[c]	0.99	Y = 0.26X − 0.18[c]	0.99
Rye	Y = 0.75X − 2.31[a]	0.98	Y = 0.79X + 0.12[a]	0.98
	Y = 0.27X − 1.01[b]	0.98	Y = 0.28X − 0.12[b]	0.98
	Y = 0.15X − 0.15[c]	0.97	Y = 0.16X + 0.36[c]	0.96
Barley	Y = 0.97X − 3.17[a]	0.95	Y = 0.99X + 0.44[a]	0.95
	Y = 0.42X − 2.7[b]	0.99	Y = 0.43X − 1.1[b]	0.98
	Y = 0.39X − 2.6[c]	0.98	Y = 0.39X − 1.1[c]	0.97
Sorghum	Y = 0.13X + 7.2[a]	0.72	Y = 0.13X + 7.23[a]	0.71
	Y = 0.43X + 5.25[b]	0.91	Y = 0.44X + 5.44[b]	0.90
	Y = 0.20X + 0.28[c]	0.99	Y = 0.21X + 0.35[c]	0.99
Rice	Y = 0.94X − 0.59[a]	1.00	Y = 0.95X − 0.03[a]	1.00
	Y = 0.75X − 0.22[b]	1.00	Y = 0.76X + 0.66[b]	1.00
	Y = 0.67X − 0.69[c]	1.00	Y = 0.67X − 0.3[c]	1.00
Corn	Y = 1.29X − 1.52[a]	0.99	Y = 1.21X − 0.62[a]	0.99
	Y = 0.25X − 0.27[b]	0.92	Y = 0.24X − 0.12[b]	0.93
	Y = 0.22X − 0.06[c]	0.88	Y = 0.21X + 0.07[c]	0.90

[a] Obtained with neutral detergent.
[b] Obtained with acid detergent.
[c] Obtained with crude fiber.

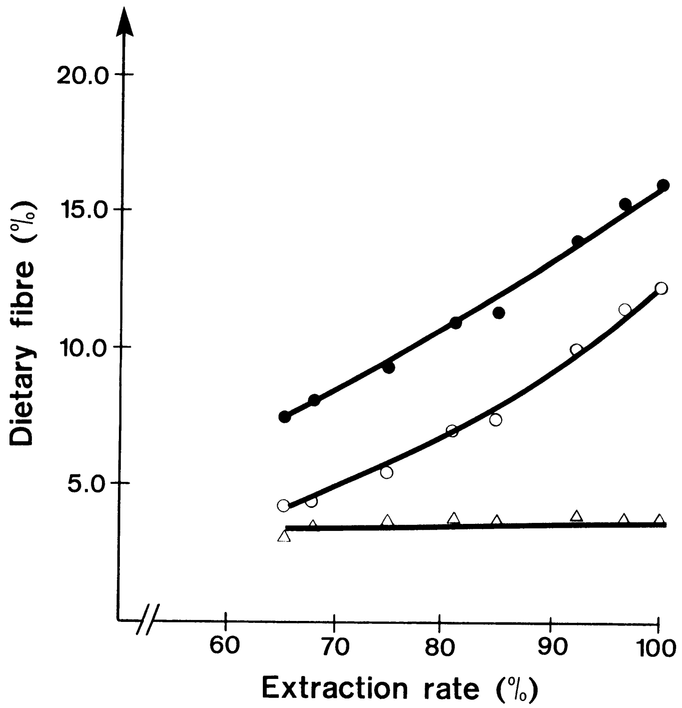

Fig. 2. Dietary fiber content in rye flours of different extraction rates. • = total dietary fiber; o = insoluble dietary fiber; △ = soluble dietary fiber.

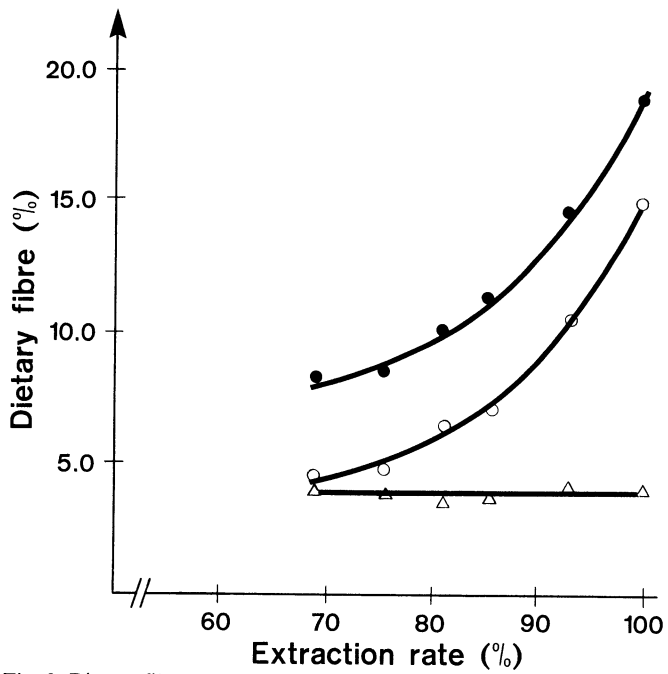

Fig. 3. Dietary fiber content in barley flours of different extraction rates. • = total dietary fiber; o = insoluble dietary fiber; △ = soluble dietary fiber.

Barley

Barley contained 8.2% dietary fiber at the lowest extraction rate and 18.8% in the whole grain (Fig. 3). The soluble fraction was about 3.9% at all extractions. The relative pentosan content was lower and the glucan content higher in barley than in wheat and rye. The monomeric composition between the two extraction rates analyzed also differed (Table I). The main components were glucose, arabinose, and xylose. The relative arabinose content was similar, but the xylose content increased, and the glucose content decreased with increasing extraction rates.

NDF-amylase values correlated well with insoluble fiber. As in rye, ADF and crude fiber values were very similar (Table II).

Sorghum

Sorghum contained only 2.5–9.0% total dietary fiber, and the soluble fraction was about 0.5% in all flours (Fig. 4). The increase in the insoluble fraction was almost proportional to the extraction rate. The dietary fiber in sorghum flour of low extractions consisted mainly of glucans. At high extraction rates, the proportion of pentosans (arabinose and xylose) increased. Uronic acids constituted 11–13% of the dietary fiber in sorghum flours (Table III).

NDF as well as ADF values of sorghum flours were higher than both total and insoluble fiber with the enzymatic method (Table II). The difference between ADF and NDF values and those found by the enzymatic methods were most pronounced in flours of low extraction rates. Crude fiber values correlated well with both total and insoluble fiber determined by the enzymatic method and gave about 1/5 of the total fiber.

Rice

Rice contained from 0.7 to 19.2% total dietary fiber. Removal of the husk, corresponding to about 80% extraction rate, decreased the dietary fiber content to 2.9%. The soluble-fiber fraction was less than 1.0% at all extraction rates (Fig. 5). The main dietary-fiber components in the most refined rice were glucose and uronic acids. In rice flour, including the husk, there were also appreciable amounts of xylose (Table III).

Because rice contains a very small amount of soluble fiber, there was good correlation between NDF-amylase values with both total and insoluble fiber assayed enzymatically (Table II). ADF and crude-fiber values were comparatively high because cellulose is a dominating component in dietary fiber from rice (Table III).

Corn

The total dietary fiber in corn varied from 3.9 to 9.3%. The soluble fraction was small, approximately 0.5%, in all flours (Fig. 6). The chemical composition was similar in whole corn and in the fine corn flour (particle size <340 μ) analyzed. The main components were glucose, arabinose, and xylose (Table III).

NDF-amylase values correlated well with total and insoluble fiber obtained by the enzymatic method, although NDF-values tended to be slightly higher (Table II).

Acid-Insoluble Lignin

Lignin, measured as Klason lignin (acid-insoluble residue), was not detectable or was very low in the most refined flours (Tables I and III). The highest values, 3.5 and 3.9%, were found in whole grain of barley and rice, respectively.

Residual Starch in the Dietary-Fiber Residue

Starch, measured with glucose oxidase after incubation with amyloglucosidase, was not detectable or was very low (≤0.1 g/100 g of flour) in the dietary fiber fractions of all flours investigated.

Comparison of Gravimetric and Gas-Chromatographic Dietary Fiber Assay

As shown in Tables I and III, the sum of the analyzed polysaccharides (measured with GLC) and Klason lignin correlated

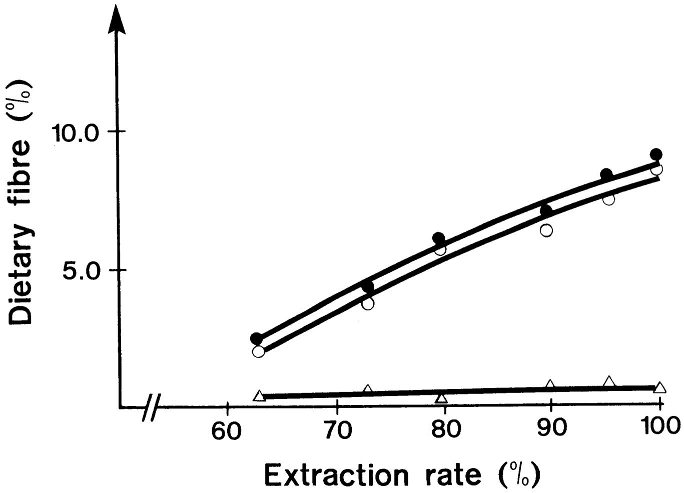

Fig. 4. Dietary fiber content in sorghum flours of different extraction rates. • = total dietary fiber; o = insoluble dietary fiber; △ = soluble dietary fiber.

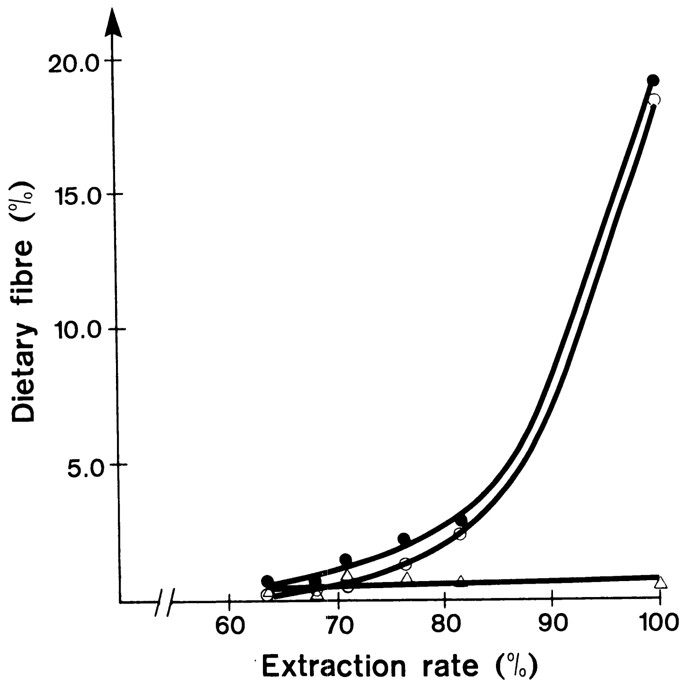

Fig. 5. Dietary fiber content in rice flours of different extraction rates. • = total dietary fiber; o = insoluble dietary fiber; △ = soluble dietary fiber.

TABLE III
Composition of Dietary Fiber in Sorghum, Rice, and Corn

	Extraction Rate (%) of					
	Sorghum		Rice		Corn	
	63	100	64	100	Fine	Whole
Polysaccharides (g/100 g)	2.3	6.7	1.1	13.0	4.1	8.0
Relative composition (%)						
Rhamnose	···	···	···	···	···	···
Arabinose	3	18	4	6	26	24
Xylose	5	15	4	25	24	30
Mannose	9	3	5	1	2	2
Galactose	9	2	5	2	5	5
Glucose	61	51	72	57	30	32
Uronic acids	13	11	10	9	13	7
Klason lignin (g/100 g)	0.4	2.5	···	3.9	0.8	1.4
Sum of dietary fiber components (g/100 g)	2.7	9.2	1.1	16.9	4.9	9.4
Dietary fiber residue[a]	2.5	9.0	0.7	19.2	3.9	9.3

[a]With the gravimetric method (sum of insoluble and soluble fractions, g/100 g).

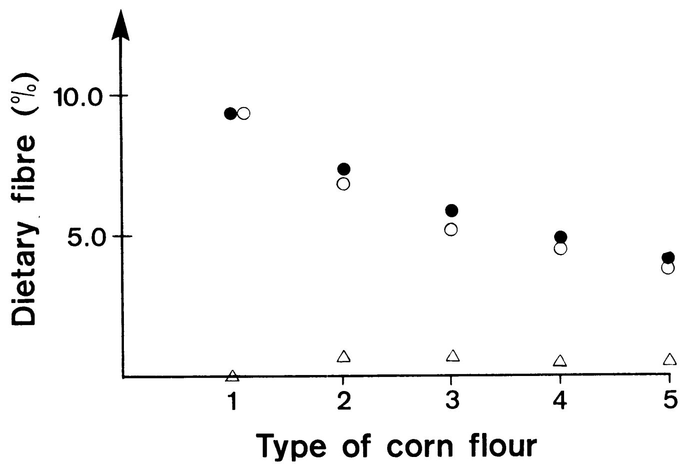

Fig. 6. Dietary fiber content in different corn products. **1,** whole corn; **2,** 500–1,000 μ corn flour; **3,** <840 μ corn flour; **4,** <340 μ corn flour; **5,** dehulled and degerminated corn flour. • = total dietary fiber; o = insoluble dietary fiber; △ = soluble dietary fiber.

well with the total dietary fiber (corrected for ash and undigestible protein) determined by the enzymatic method. The correlation coefficient was 0.99. The lower values obtained with GLC in some samples may have resulted from undetermined minor components such as phenolic acids (Smart and O'Brien 1979) or from losses during hydrolysis, before GLC. The higher values in other samples might be caused by overlapping of the undigestible protein corrected for in the enzymatic method and by Klason lignin in the GLC method (Robertson and Van Soest 1981, Theander and Åman 1981).

DISCUSSION

As expected, the cereals analyzed showed increased dietary fiber content with increasing extraction rate. The slope and the start and endpoints of the curves differed, however. These differences are explained by differences in histological constitution of the whole grains, as well as by differences in the degree of separation of the various cell layers obtained in the dry-milling process. Thus, the constant dietary-fiber level below 80% extraction rate and the sharp rise in insoluble dietary fiber above 80% extraction rate in wheat shows that the pericarp, testa, and aleurone are better separated from the endosperm in this cereal than in rye. The same tendency is seen in barley and rice compared to sorghum when all three cereals are subjected to milling. Furthermore, barley and rice contain husks that are rich in insoluble dietary fiber and lignin (Barber et al 1981, Munck 1981a, Salomonsson et al 1980). This explains the higher concentration of nonstarchy polysaccharides and total dietary fiber in these two cereals.

A critical step in all dietary-fiber methods is the removal of starch. Starch residues add to the dietary fiber and appear as glucose in gas-chromatographic determination of dietary-fiber composition. The combination of Termamyl and pancreatin in the enzymatic gravimetric method used here leaves almost no starch residues in unprocessed flours (Asp et al 1983). All dietary-fiber residues obtained were also checked for residual starch by amyloglucosidase incubation, and the highest value found was 0.1% (dry basis). Resistant starch (ie, starch available to amylase only after solubilization with 2*M* KOH) has been reported to occur in the dietary-fiber fraction of processed foods (Englyst et al 1982). Resistant starch has not been demonstrated in unprocessed flours, nor did we find measurable amounts of it when analyzing wheat and rye flours by the enzymatic gravimetric method (*unpublished results*). Further evidence for the efficiency in starch removal is the agreement between dietary-fiber glucan values in the present study and those reported by Englyst et al (1982) for wheat, rye, and barley.

The amount of soluble fiber was higher in rye, barley, and wheat than in the other cereals. The water-soluble fraction in wheat and rye contains mainly arabinoxylans (Geissmann and Neukom 1973, Holas et al 1972) whereas in barley it is dominated by β-glucans (Fincher 1975). In all the cereals we investigated, the content of soluble fiber was independent of the extraction rate, showing that soluble polysaccharides arise essentially from the endosperm. Our results for wheat are in accordance with those reported by Neukom et al (1977), who found 0.8–1.2% soluble, ethanol precipitable, nonstarchy polysaccharides in wheat flours of various extraction rates. Only traces of nonstarchy polysaccharides (0.02–0.05%) were not precipitated by ethanol. The total nonstarchy polysaccharide content was 2.2–10.3%, similar to the results reported in Table I. The higher pentosan content of wheat and rye is probably important for its baking properties, especially certain soluble pentosan fractions (Ciacco and D'Appolonia 1982).

In wheat and rye, the monomeric composition of the dietary fiber was similar at high and low extraction rates. This suggests that the xylose–arabinose ratio in the endosperm and outer layers is similar. Our results for wheat are in agreement with those of Englyst et al (1982). The monomeric composition of nonstarchy polysaccharides in rye and barley reported by these authors also agrees with our results. Anderson and Clydesdale (1980), however, found a higher xylose–arabinose ratio in wheat. According to Medcalf et al (1968) and Wilson Lee and Stenvert (1973), the proportions between arabinose and xylose vary in different wheat varieties.

In barley and rice, the relative proportion of xylans increased with increasing extraction rate, whereas that of glucans decreased. The kernels of these cereals are surrounded by a husk, which, in barley, is rich in xylans (Salomonsson et al 1980). This explains the dramatic rise in xylose content in the flours containing the husk (100% extraction rate). The increase in xylose content at high extraction rate in barley and rice is in contrast to that found in wheat, rye, and corn. These cereals had relatively uniform arabinose–xylose ratio at the various extraction rates. According to Fincher (1975) endosperm cell walls of barley contain up to 75% of mixed β-glucans. This, together with the removal of the aleurone cells consisting mainly of arabinoxylan carbohydrates (McNeil et al 1975), explains the relative increase in glucose-based dietary fiber constituents at the low extraction rate.

In sorghum, both the arabinose and xylose content was higher in whole grain than in refined flour. This is in agreement with Woolard et al (1977), who found that the outer layers contained an arabinoxylan.

The fine corn flour had a xylose–arabinose ratio around 1. The tendency to higher xylan and glucan content in the whole grain flour is in agreement with the high cellulose and xylan content in corn bran (Schimberni et al 1982).

Appreciable amounts of uronic acids were found only in sorghum, rice, and corn. The presence of pectic substances in rice endosperm cell walls has been reported (Shibuya and Iwasaki 1978). The absence of pectins in wheat and barley endosperm is in agreement with results from studies by Fincher (1975) and Mares and Stone (1973).

The degree of lignification differed considerably with the rate of extraction, reflecting the fact that only the pericarp and husk are lignified. This may be important for physiological properties, such as resistance to bacterial fermentation in the large intestine, and fecal bulking (Cummings 1981).

The general appearance of the variation in the dietary fiber in relation to extraction rate is probably typical. Analyses of another variety of, for example, wheat, might change the start and end points of the curves slightly. The milling procedure used may also affect the slope of the curves. Nevertheless, the determination of dietary fiber by gravimetric analyses can probably be useful for prediction of extraction rate of flours used in cereal products. In processed products to which salt has been added, ash determination cannot be used for estimation of extraction rates.

Comparisons of different gravimetric dietary-fiber methods show that the NDF-amylase procedure correlates differently with total dietary fiber in different cereals. This is mainly because the content of soluble dietary-fiber components varies. In wheat, rye, and barley of low extractions, almost half of the total dietary fiber was soluble, clearly demonstrating that this fraction should be included in a proper assay of dietary fiber.

The NDF amylase method seems to solubilize some rye components that are insoluble with an enzymatic method. ADF values and crude-fiber values correlate differently with total and insoluble fiber in different cereals. Earlier investigations of the relationship between extraction rate and fiber content used the crude-fiber method (Ziegler and Greer 1978). By this procedure, which measures only a small and variable part of dietary fiber, fiber traces of 0.1% have been found in white wheat flour and of 2.2% in whole grain wheat flour. Thus, the underestimation of dietary fiber by this method is most pronounced in flour of low extraction, and therefore a comparison of crude fiber values in various flours would be misleading.

NDF and ADF determination in sorghum implies special problems related to proteins that are difficult to solubilize (Bach Knudsen and Munck 1983). Because a low-tannin sorghum was used in the present study, tannin is unlikely to have interfered.

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