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0308-8146(95)00250-2

Food Chemistry, Vol. 57, No. 2, pp. 305–310, 1996

0308-8146/96 $15.00 + .00

Extraction and fractionation of insoluble fiber from five fiber sources[1]

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(Received 15 May 1995; accepted 27 November 1995)

Commercially processed wheat bran (WB), rice bran (RB), oat fiber (OF), tomato fiber (TF), and apple fiber (AF) were analyzed for proximate composition, soluble fiber (SF), insoluble fiber (IF) and total dietary fiber (TDF). IF was further fractionated into four fractions: cellulose, hemicellulose A and B, and lignin. Protein, IF, and TDF values were significantly different among the samples. Protein content ranged from 4.6% in OF to 24.9% in TF. SF values ranged from 1.5% in OF to 13.9% in AF. IF values varied from 46.7% in RB to 73.6% in OF. TDF ranged from 51.4% in RB to 75.1% in OF. Hemicellulose A to B ratio was high in all the samples except rice bran. WB had the highest total hemicellulose (44.0% of TDF) content followed by AF (38.4%), OF (38.3%), TF (36.5%), and RB (31.6%). Cellulose was 32.2, 26.6, 24.4, 20.8, and 19.7% of TDF in WB, OF, RB, AF, and TF, respectively. Lignin levels ranged from 5.2% in WB to 21.4% in OF. Copyright © 1996 Elsevier Science Ltd

INTRODUCTION

The measurement of dietary fiber in foods is a complex issue of not only choice of analytical method, but also the definition of fiber. Dietary fiber is currently defined as the 'sum of polysaccharides plus the phenolic polymer lignin that are not hydrolysed by the endogenous secretions of the human digestive tract' (Southgate *et al.*, 1978; Trowell *et al.*, 1976). Dietary fiber has been reported under various names over the years such as: roughage, bran, plant residue, crude fiber, unavailable carbohydrates, non-starch polysaccharides, and plantix (Spiller & Gates, 1978).

The original concept of dietary fiber focused on the components derived from the plant cell wall which include cellulose, hemicellulose, gums, and pectins. However, this view was expanded after much controversy to include digestion-resistant starch, lignin, and other non-starch polysaccharides, in the definition of fiber (Flint & Camire, 1992; Cummings & Englyst, 1991).

Methods for the determination of dietary fiber are divided into four categories: (1) gravimetric methods, (2) gravimetric–enzymatic methods, (3) calorimetric methods, and (4) chromatographic methods. Gravimetric techniques were the earliest and included crude fiber, acid detergent fiber, and neutral detergent fiber. These methods grossly underestimate dietary fiber content and are being replaced by new and more accurate methods (Dreher, 1987).

Techniques for fractionation of dietary fiber into its individual components are limited in number. Furda (1977, 1981) proposed a fiber extraction technique which included isolation of water-soluble fiber fractions from various food sources. Southgate (1976, 1977) outlined and updated an extraction and fractionation procedure for lignocellulose, crude lignin, and cellulose fractions. The 'Fiber Sparing Analysis' technique (Monte & Maga, 1980) combines methodologies from other analysts in such a way as to produce 13 end products that can be utilized for further evaluation and study.

There is sufficient nutritional evidence to show that total dietary fiber values alone cannot predict the actual physiological properties of dietary fiber (Southgate, 1985; Hall, 1989).

Numerous researchers have determined the cellulose, hemicelluloses, and lignin contents of dietary fiber from food sources, while few studies have attempted to isolate and fractionate fibers into the major components. Whereas it is true that physiological effects of fiber depend on the relative amount of individual fiber components, perhaps the greatest differences have been demonstrated for soluble vs insoluble. Consequently, it seems essential to isolate and fractionate insoluble fiber

[1]Presented in part at the Annual Meeting of the Institute of Food Technologists, Chicago, IL. June 1993.

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from wheat bran, rice bran, oat fiber, apple fiber, and tomato fiber.

MATERIALS AND METHODS

Fiber sources

Sources of fiber for this research were as follows: Hard Red Spring wheat bran (American Association of Cereal Chemists, St. Paul, Minnesota), apple fiber (Tree Top Inc., Selah, Washington), oat fiber (D. D. Williamson Louisville, Kentucky), rice bran (California Natural Products Lathrop, California), and tomato fiber (H. J. Heinz Co., Tracy, California).

Fiber preparation

Wheat bran was processed with a Wiley mill (Model No. 2) until all passed through a 60 mesh screen size. Tomato fiber (seeds and skin) received fresh from the supplier after extraction of juice was air dried and processed as described above. Apple fiber, rice, bran, and oat fiber were received as fine particles (60–100 mesh) and used as is.

Chemical composition

Two aliquots from original fiber samples were analyzed for moisture and fat contents (AOAC, 1990), while the remaining samples were defatted for 8 h using hexane as a solvent (5 ml/g sample). Two separate samples taken from defatted and dry fiber sources were analyzed in duplicate for protein, ash (AOAC, 1990), and acid detergent fiber (Robertson & Van Soest, 1981).

Dietary fiber determination

Duplicate fat free dry samples were analyzed for soluble and insoluble fiber using the method of Prosky *et al.* (1988). After enzyme incubation of the samples was completed, the mixture was filtered. The insoluble fiber (IF) residue was dried and weighed while the filtrate was transferred into a 600 ml beaker and mixed with 4 volumes of 95% ethanol. The mixture was filtered and the residue (soluble fiber) was dried and weighed. After corrections for ash and protein were made, combined values for SF and IF gave a figure for TDF.

Fractionation procedure

The fiber extraction and fractionation was conducted as outlined by Southgate (1976) with some modifications (Fig. 1). In order to optimize yield of components, cold and hot water extraction of the fibers was used to remove partially soluble polysaccharides and proteins before enzyme treatment. The procedure has been found to reduce these components by about 20% (Monte & Maga, 1980; Anderson & Clydesdale, 1980). Delignification and autoclaving steps were omitted. Potassium hydroxide (under nitrogen atmosphere) was used in place of the detergent technique for extracting lignocellulose. Hemicellulose A and B were extracted according to the procedure described by Monte & Maga (1980). Duplicate extractions were made and sample sizes increased for more accuracy as suggested by Rasper (1981).

Removal of soluble complex carbohydrates and proteins

The method employed was that described by Anderson & Clydesdale (1980). Defatted fiber samples were extracted for 2 h at 20°C using slightly alkaline water (1:10 w/v ratio, pH 7.0–7.5). The samples were then centrifuged at 1500 ***g*** for 10 min. The supernatants were discarded and the procedure repeated three times.

Residues were extracted with 0.01 M EDTA solution for 2 h to bind cations and solubilize more pectic substances (Furda, 1977). The mixtures were filtered and the extraction repeated twice. After extraction the residue was washed twice with 80% ethanol and three times with distilled deionized water to remove the alcohol. The washed residue was lyophilized and saved for further analysis. This residue was called nonpurified insoluble residue (NPIR).

Enzymatic treatment of non-purified insoluble residue

The NPIR of each fiber was treated enzymatically using the method of Southgate (1976). Twenty-five grams of NPIR were weighed into a 4 liter glass beaker to which 0.1 M acetate buffer pH 4.8 was added (50 ml/g fiber). Duplicate treatments were conducted. The pH was adjusted if it did not equal pH 4.8 ± 0.1. An amyloglucosidase solution was added (0.15 ml/g fiber), the beaker covered with foil and incubated for 3 h at 55°C, with continuous agitation. After cooling, the pH was adjusted to pH 8 ± 0.1 by adding 0.275 N NaOH solution. Trypsin was added (5 mg/g fiber) and incubated for 18 h at 37°C, stirring slowly. Finally, the mixture was filtered and washed three times with 80% ethanol, once with 95% ethanol, three times with DD water, then freeze dried. The residue obtained was considered as pure insoluble residue (PIR). The presence of starch in the enzyme treated fiber fractions was checked using an iodine solution.

Removal of insoluble pectic substances

Duplicate 1 g PIR samples were extracted three times using 10 ml of 0.5% (w/v) ammonium oxalate solution at 85°C for 2 h (Monte & Maga, 1980). The fiber residue was filtered and washed with ethanol, DD water, and then dried. The residue was defined as depectinated insoluble residue (DIR). The loss in weight on drying was the yield of insoluble pectin present in the sample.

Hemicelluloses A and B extraction

The method of Monte & Maga (1980) was used to extract hemicellulose A and B. Five grams of depectinated IF

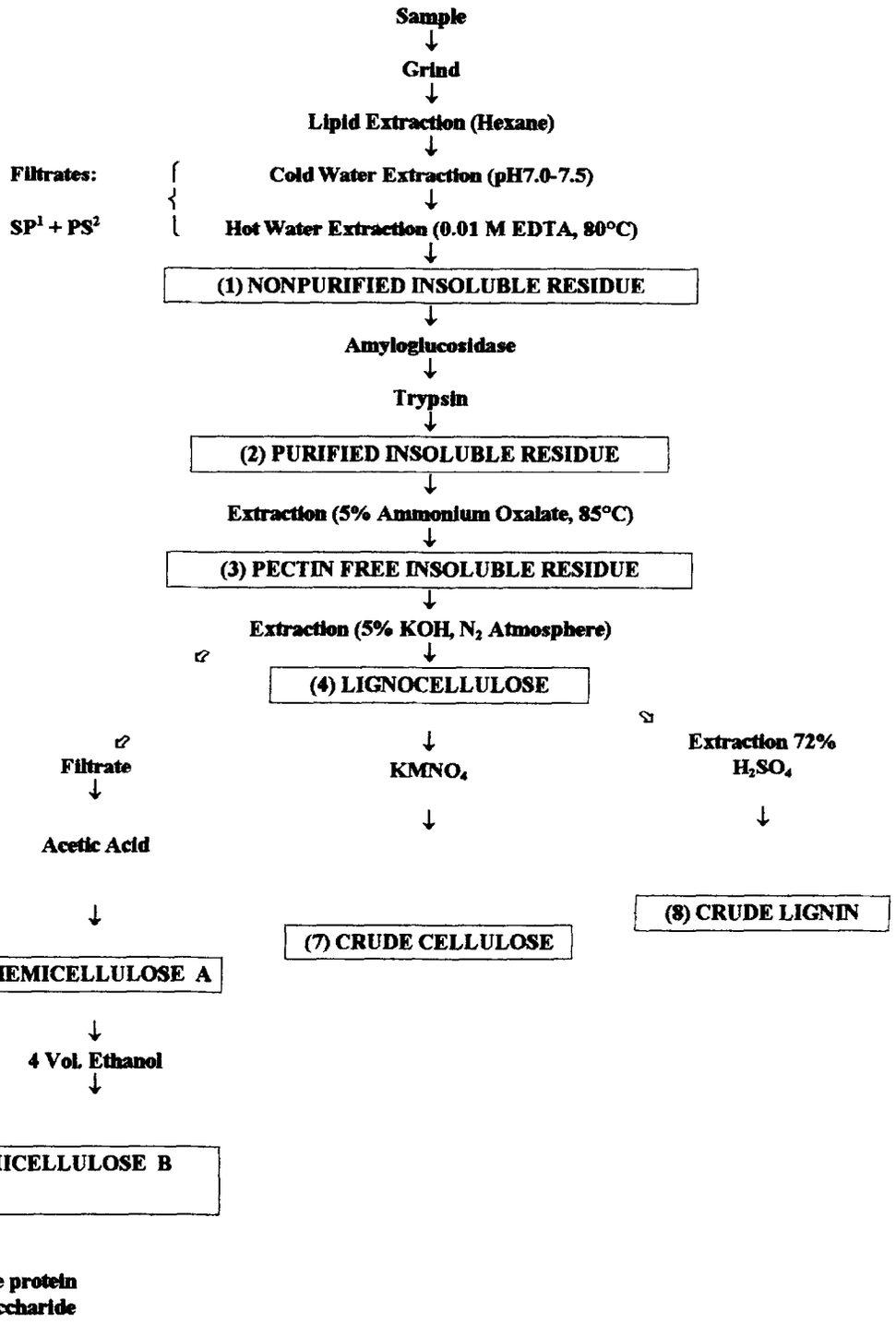

Fig. 1. Extraction and fractionation of fiber sources.

were weighed in duplicate into 250 ml plastic stoppered centrifuge bottles and 100 ml of 5% (w/v) potassium hydroxide solution was added. The bottles were flushed with nitrogen and shaken for 24 h, then centrifuged at 1500 *g* for 10 min. The supernatant was decanted and saved, while the residue was further extracted two more times. The residue described as lignocellulose was washed, dried, and saved for further analysis. Filtrates were combined with 50% acetic acid, adjusted to pH 5.0–5.5 and centrifuged. The hemicellulose A (HCA) fraction was washed and freeze dried while the supernatant was diluted further with 4 volumes of 95% ethanol to produce a second precipitate, hemicellulose B (HCB).

Crude cellulose extraction

Crude cellulose was obtained by the method of Robertson & Van Soest (1981). Duplicate 2 g portions of lignocellulose were extracted using about 20 ml of combined reagent ($KMnO_4$ + lignin buffer, 2:1 ratio) in sintered glass crucibles, and allowed to stand for

Table 1. Chemical composition of five fiber sources[a]

Fiber sources	Moisture %	Crude fat %	Ash %	Protein %	Total carbohydrate ADF[b] %	Total carbohydrate CHO[c] %
Wheat bran	4.8 ± 0.01[ab]	1.7 ± 0.05[c]	6.9 ± 0.11[c]	17.2 ± 0.27[c]	15.2 ± 0.14[d]	59.0 ± 0.47[a]
Oat fiber	4.7 ± 0.26[b]	0.9 ± 0.01[a]	5.9 ± 0.01[d]	4.6 ± 0.14[d]	40.5 ± 0.13[c]	48.1 ± 0.04[c]
Rice bran	5.1 ± 0.11[a]	1.3 ± 0.08[d]	11.9 ± 0.02[a]	19.3 ± 0.12[b]	15.1 ± 0.01[d]	52.5 ± 0.01[b]
Apple fiber	2.8 ± 0.01[c]	2.5 ± 0.03[b]	10.9 ± 0.11[b]	4.7 ± 0.01[d]	56.1 ± 0.12[a]	25.8 ± 0.04[d]
Tomato fiber	2.0 ± 0.01[d]	3.2 ± 0.11[a]	5.1 ± 0.02[e]	24.9 ± 0.33[a]	47.2 ± 0.29[b]	19.7 ± 0.84[e]

[a]Determined in duplicate dry samples (mean ± SD). Mean values having the same superscript within columns are not significantly different ($P < 0.05$).[b]ADF = Acid Detergent Fiber.[c]CHO = Carbohydrate (calculated by difference): 100 − (protein + fat + ash + acid detergent fiber).

90 ± 15 min, at 22°C, with periodic stirring. The reagent in the crucibles was made to remain purple by changing frequently for the duration of the extraction process. The combined reagent was drawn out by suction and the crucibles transferred to a clean pan. Demineralizing solution (20 ml) was added to each crucible, allowed to stand for 5 min, refilling as necessary, then removed by suction. The completion of demineralization was indicated by the removal of black manganese from the white/gray cellulose. The extraction lasted an average of 30 min. Crude cellulose (CR) was washed with 80% alcohol and DD water then lyophilized and stored in a freezer.

Lignin extraction

The Klason lignin method (Robertson & Van Soest, 1981) was used in this study. Duplicate samples of lignocellulose (5 g) were extracted with cold 72% sulfuric acid solution (1 g/ml, w/v) at 4°C for 30 h. Cold distilled deionized water was added (150 ml) and the residue allowed to precipitate. The residue was washed with warm DD water until no acid was detectable. The crude lignin residue (CLR) was then air dried and kept in a freezer for later studies.

Statistical analysis

Data, expressed as mean ± SD, obtained from two separate fiber samples and their respective isolated fractions were statistically analyzed using one way analysis of variance with means separated and a least significance set at $P < 0.05$ (Steel & Torrie, 1960).

RESULTS AND DISCUSSION

Chemical analysis

The chemical composition of wheat bran, oat fiber, rice bran, tomato fiber, and apple fiber is presented in Table 1. Rice bran, tomato fiber, and wheat bran contained significantly higher ($P < 0.05$) protein than apple and oat fibers. Protein values were in the same range as those previously reported for rice bran and wheat bran (Dreher, 1987; Saunders, 1990). Values for apple and tomato fiber were also in agreement with those reported by Idouraine *et al.* (1995) and Chang & Morris (1990). Crude fat content was significantly different ($P < 0.05$) among the samples. Values varied from 0.9% in oat fiber to 3.2% in tomato fiber. Rice bran and apple fiber showed significantly higher ($P < 0.05$) amounts of ash than wheat bran, oat fiber, and tomato fiber, respectively. Moisture was about 5% or below. Acid detergent fiber (ADF) content, mainly lignin and cellulose, was significantly higher in tomato, oat, and apple fibers than wheat and rice bran. ADF content of all the fiber samples except oat were within the ranges of those reported by Idouraine *et al.* (1995) and Schweizer & Wursch (1978).

Differences could also be related to milling/separation procedures and/or varietal/environmental conditions of the fiber sources. Total carbohydrate content was generally high for all the fiber samples.

Dietary fiber

Soluble fiber (SF), insoluble fiber (IF), and total dietary fiber (TDF) values are indicated in Table 2. SF values varied significantly ($P < 0.05$), and ranged from 1.5% for oat fiber to 13.9% for apple fiber. IF values ranged from 46.7% for rice bran to 73.6% for oat fiber, indicating that IF was a major component of the fiber material obtained from suppliers. The high ADF content of oat, apple, and tomato fibers (Table 1) was

Table 2. Soluble, insoluble, and total dietary fiber values for five food sources[a]

Fiber sources	Soluble fiber g/100 g	Insoluble fiber g/100 g	Total dietary fiber g/100 g
Wheat bran	4.6 ± 0.27[c]	49.6 ± 0.59[c]	54.2 ± 0.30[d]
Oat fiber	1.5 ± 0.01[d]	73.6 ± 0.85[a]	75.1 ± 0.86[a]
Rice bran	4.7 ± 0.12[c]	46.7 ± 0.01[d]	51.4 ± 0.11[e]
Apple fiber	13.9 ± 0.14[a]	48.7 ± 0.13[c]	62.6 ± 0.26[c]
Tomato fiber	8.3 ± 0.11[b]	57.6 ± 0.43[b]	65.9 ± 0.54[b]

[a]Determined in duplicate fat free dry samples (mean ± SD). Mean values having the same superscript within columns are not significantly different ($P < 0.05$).

Table 3. Cellulose, hemicellulose, and lignin content of five fiber sources[a]

Fiber sources	Hemicellulose A	Hemicellulose B	Cellulose	Lignin	Insoluble pectin
			%		
Wheat bran	28.0 ± 0.72[a]	16.0 ± 0.09[b]	32.2 ± 0.04[a]	5.2 ± 0.25[e]	3.9 ± 0.01[d]
Oat fiber	21.3 ± 0.25[c]	17.0 ± 0.27[b]	26.6 ± 0.05[b]	21.4 ± 0.45[a]	8.9 ± 0.20[b]
Rice bran	7.6 ± 0.03[d]	24.0 ± 0.09[a]	24.4 ± 0.29[c]	18.4 ± 0.29[b]	7.4 ± 0.10[c]
Apple fiber	28.4 ± 0.76[a]	10.0 ± 0.1[d]	20.8 ± 0.01[d]	12.1 ± 0.29[d]	8.9 ± 0.03[b]
Tomato fiber	23.3 ± 0.23[b]	13.2 ± 0.29[c]	19.7 ± 0.22[e]	13.8 ± 0.28[c]	9.7 ± 0.15[a]

[a]Expressed as percent total dietary fiber. Determined on duplicate fat-free dry samples (mean ± SD). Mean values having the same superscript within columns are not significantly different ($P < 0.05$).

reflected in the high IF values for those samples. Renard & Thibault (1991) studying SF, IF, and TDF of apple fiber reported values of 59.7, 46.3, and 13.4%, respectively. Their values are similar to those reported in our study. Values obtained for wheat bran, oat fiber, and tomato fiber are within the range of values reported by Weber *et al.* (1993). The TDF content of rice bran indicated in this study was within the range provided by the company.

Removal of insoluble pectin

Insoluble pectin removed from the fiber fractions varied from 3.9% for wheat bran to 8.9% for apple fiber and oat fiber (Table 3). Tomato, apple, and oat fibers were significantly the highest ($P < 0.05$) followed by rice and wheat brans. The residue obtained after insoluble pectin extraction was regarded as pure IF. Sabir *et al.* (1975) and Aspinall & Jiang (1974) have reported virtually complete extraction of pectins from various fibers using dilute ammonium oxalate.

Hemicellulose A and B extraction

The amounts of hemicelluloses A and B extracted are indicated in Table 3. Hemicellulose A ranged from 7.6% for rice bran to 28% for wheat bran. Hemicellulose B varied from 10% for apple fiber to 24% for rice bran. The hemicellulose A to B ratio was high in all samples except rice bran. It can be calculated from the data published by Dreher (1987) that wheat bran and rice bran contain about 52 and 39% total hemicellulose, respectively. These values were slightly lower than those indicated in this study. Anderson & Clydesdale (1980) and Mongeau & Brassard (1982) reported a hemicellulose content of 65% which is higher than that reported in our study. Southgate (1976) reported a value of 50.4% for oat fiber, higher than that found in this study. These differences may be due to the procedures used to isolate the fiber fractions. Hemicellulose data, particularly in wheat bran, might be an important factor since it can affect the quality of baked products. Jeltema & Zabik (1979) and Jeltema *et al.* (1983) showed that insoluble hemicellulose had the greatest effect on the baking quality of cakes, positively correlated with increased cake volume, tenderness, and thinner cell walls.

Crude cellulose

Cellulose values varied from 19.7% in tomato fiber to 32.2% in wheat bran (Table 3). Wheat bran showed significantly the highest ($P < 0.05$) crude cellulose content followed by oat fiber, rice bran, apple fiber, and tomato fiber, respectively. The cellulose value of wheat bran reported in this study was similar to that calculated from data reported by Dreher (1987). Cellulose values of the other fibers reported in the literature were not consistent. This might be explained by differences in the methods used for determination. Southgate (1976), using the permanganate procedure, reported crude cellulose values of 33% for oat fiber and 26% for rice bran. On the other hand, Dreher (1987) reported a value of 14% for rice bran. No cellulose data was available for tomato fiber for comparison.

Crude lignin

Crude lignin content was significantly different ($P < 0.05$) among the fibers, varying from 5.2% in wheat bran to 21.4% in oat fiber (Table 3). An appropriate length of time for acid hydrolysis is still a controversy. Idouraine *et al.* (1995) reported crude lignin values of 4.7, 15.0, and 23.6% for rice bran, apple fiber, and tomato fiber, respectively. These results as well as others in the literature are not consistent, probably because of the differences in methodology and length of time of hydrolysis. The lignin values obtained for wheat in this study agree with previous results reported by Dreher (1987) and Flint & Camire (1992).

The recovery of these fractions, expressed as percentage total dietary fiber, was good and varied from 79.7% in wheat bran to 95.2% in apple fiber. The difference may be due to some carbohydrate monomer loss during the fractionation.

A major advantage of developing a fractionation procedure is the additional information that the major components of various fiber sources may provide for further studies such as physiological effects (mineral binding) and physicochemical properties of foods. The amounts of fiber components, as shown in this study, vary from one fiber source to another. Similarly, previous works indicate that various fibers have different physical properties associated with physiological responses and food applications (Schneeman, 1986;

Holloway & Greig, 1984; Wen *et al.*, 1988). Dietary fiber has been implicated in the binding of bile acids and reduced availability of minerals. Fractionation of dietary fiber to cellulose, hemicellulose, and lignin may elucidate which fraction affects mineral bioavaliability. Further studies are therefore needed on these fractions.

CONCLUSION

The present scheme permits separation of the fibers into their major components. The insoluble fiber composition of the five samples studied varied from one fiber source to another. Hemicellulose was found to be the major component in all the samples followed by cellulose and lignin, respectively. The results presented in this study for wheat bran were in agreement with those found in previous studies. This would suggest that the methods provide reliable fractions for further characterization and nutritional studies. Standard procedures for fractionation are required to study the numerous conventional and non-conventional fiber sources. There is good potential for hemicelluloses and cellulose of various fiber sources as food ingredients, e.g. bulking agent, in food product formulations. Finally, the market demand for high-fiber foods will continue to increase as more supporting data becomes available on their disease prevention ability.

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