

Characterization and Estimation of Barley Polysaccharides by Near-Infrared Spectroscopy. I. Barleys, Starches, and β -D-Glucans

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ABSTRACT

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Large and small starch granules were isolated from regular amylose (about 25%), high-amylose (44-49%), and high-amylopectin (traces of amylose) barleys. Their near-infrared reflectance (NIR) spectra were compared with those of relatively pure (1-3),(1-4)- β -D-glucans from barley and oats and with NIR spectra of ground barleys—naked and covered, regular, high-amylose, and high-amylopectin. The objective was to obtain detailed information on NIR spectra of barleys, isolated starches, and isolated β -D-glucans to serve as a basis for development of a near-infrared spectroscopy method for β -glucan assay in barleys. There was little difference in NIR spectra of hull-less and covered barleys; whole meals of regular, high-amylose, and high-amylopectin barleys showed

similar spectra. Small differences were recorded between spectra from high-amylose and regular or high-amylopectin starches. Spectra of starches and whole meals from which the starches were obtained differed widely. Major differences were recorded in three areas over the whole range of 1,600 to 2,400 nm for whole meals, isolated starches, and isolated β -glucans. They included 1) wavelengths 1,702, 1,707, 1,772, and 1,773 nm, principal bands of starches, cellulose, and hemicellulose; 2) 2,060, 2,096, and 2,132 nm, bands associated with the interaction between starch and protein; and, foremost, 3) 2,268, 2,282, and 2,335 nm, bands that typify starch, β -glucans, and cellulose.

Barley is mainly used as an animal feed, but a significant and economically important amount is used in the malting and brewing industries (Munck 1981).

The α -glucan starch and the mixed-linkage β -glucan compose the bulk of polysaccharides present in the barley grain. Starch occurs in the form of large lenticular and small spherical granules and can vary from regular (75% amylopectin, 25% amylose) to high-amylose (35-70% amylose) to waxy (up to 100% amylopectin) (Hoseney 1986, Pomeranz 1987). During the isolation of barley starch, small granules associate with the protein fraction (brown layer), which appears on top of centrifuged suspensions of crude starch. The lower, white layer obtained after centrifugation contains mostly large starch granules (McDonald and Stark 1988).

Mixed-linked (1-3),(1-4)- β -D-glucans, referred to hereafter as β -glucan, are nonstarchy polysaccharides distributed largely in the endosperm cell walls and also in the aleurone cell layer of barley grains (Bacic and Stone 1981, Dixon 1985). The β -glucan constitutes a relatively minor fraction (2-10% by weight) of the total barley carbohydrates. However, as a high molecular weight water-soluble polymer, it has unique properties of both nutritional and technological significance. β -Glucan is undigested by monogastric animals and has been reported to be responsible for the low use of barley as a poultry feed (Wood 1984). On the other hand, as a soluble dietary fiber, β -glucan may be an effective hypoglycemic and hypocholesterolemic agent in the human diet (Newman et al 1989). Much attention has been directed to barley β -glucans in the brewing industry. Several problems have been associated with increased levels of β -glucan, such as retardation of malting, increased viscosity of wort and beer, or formation

of precipitates in the final beer (Bamforth 1982). Thus, a simple and reliable method for the estimation of β -glucan would be of value in predicting the suitability of barley for malting and feed purposes and for breeding programs aimed at lowering the β -glucan and/or increasing the starch content of barley. In many situations, the speed of such estimation is of major importance, and a reduction in analytical accuracy may be acceptable if short analysis times can be achieved.

Current methods for the determination of barley β -glucan include both physicochemical and enzymatic analyses (for a review see Jorgensen and Aastrup 1988). As screening procedures, these tests are expensive and time-consuming and often require specialized equipment.

Near-infrared reflectance spectroscopy (NIRS) is an established, economical, and nondestructive technique widely used in the food and feed industry (Williams and Norris 1987). The method is fast (normally less than 1 min per sample), and often more than one component can be determined at the same time. In barley, determinations were made, among others, for moisture (Downey 1985), protein (Donhauser et al 1983), starch and energy (Kim and Williams 1990), amino acids (Williams et al 1984), hot water extract (Morgan and Gothard 1979), crude fiber (Gerstenkorn 1985), disease resistance (Asher et al 1982), and aleurone color and endosperm modification (Henry 1985a). We know of no work on characterization by NIRS of barley starches. Similarly, little work has been reported on the assay of barley β -glucan, mainly because of the occurrence of small differences between recorded spectra of different polysaccharides. Henry (1985b) and Law (1985) attempted to determine total β -glucan content in barley by NIRS but were unable to obtain a satisfactory prediction (the multiple correlation coefficient was 0.69). Better results ($P = 0.87$) were claimed by Allison et al (1978), who used six selected wavelengths for the estimation of the soluble β -glucan content in barley.

The objective of this investigation was to determine whether NIRS can be used to estimate (measure) the total β -glucan content of barley. To this end, first we determined factors that govern

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and affect the NIRS assay. Those factors included naked versus covered barleys, small and large starch granules, and starches isolated from barleys that varied in amylose content.

MATERIALS AND METHODS

Barleys, Starches, and β -Glucans

Barley starches (regular, high-amylose, and waxy) were isolated and purified from Glacier (regular, high-amylose) and Wanubet (waxy) cultivars. Those barley genotypes, from the 1989 crop, were provided by S. E. Ullrich, Agronomy Department, Washington State University. Starch was separated into white and brown layers, which in some experiments were mixed to obtain representative starch granule preparations. In addition, whole-grain meals prepared from covered and hull-less barley cultivars (regular or high-amylose Glacier and waxy Wanubet) were included in the examination. A commercially available pure barley β -glucan was from Novo Industri A/S, Copenhagen, Denmark. A crude preparation was obtained from J. D. Mullen (General Mills, Minneapolis, MN). A preparation of purified β -glucan from oats (cv. Hinoat) was supplied by P. Wood, Agriculture Canada, Ottawa. All barley samples used for analyses were ground in a grinder (Udy Corp., Fort Collins, CO) fitted with round, 0.5-mm sieve openings.

Analytical Reagents

Amyloglucosidase, A-3042, from *Aspergillus niger*; protease, P-5147, type XIV, from *Streptomyces griseus*; protease, P-5380, type VIII, from *Bacillus licheniformis*; β -glucanase, C-0901, from *Penicillium funiculosum*; glucose oxidase, G-6766, from *Aspergillus niger*; peroxidase, P-6782, type VI-A, from horseradish; *o*-dianisidine, D-3252; amylose, A-0512, from potato; and amylopectin, A-515, from potato were purchased from Sigma Chemical Co., St. Louis, MO. Heat-stable α -amylase, Takalite L-340 from *Bacillus licheniformis*, was obtained from Miles Laboratories, Elkhart, IN.

Isolation of Barley Starches

Starch was isolated from barleys by a modification of the method of McDonald and Stark (1988). Barley kernels were cracked lightly by passing through a Tecator Cemotec 1090 sample mill (Tecator, Herndon, VA) and dehulled in a Kice-DT4 air-aspirated grain cleaner (Kice Metal Products Co., Wichita, KS). After steeping at 4°C in 0.02M HCl for 17 hr and neutralizing with 0.2M NaOH, the pearled material was rubbed gently in a mortar with water, and the resulting slurry was successively sieved through 130- and 73- μ m polypropylene screens. The residue was homogenized in a blender (Waring, New Hartford, CT) with water, the mixture was screened, and the process repeated three to four times. The lower, white layer obtained on centrifugation (1,700 \times g for 20 min) of barley starch suspensions was purified six times by the toluene shaking procedure (McDonald and Stark 1988). The pigmented fractions (proteinaceous brown layer and tailings) on top of the starch were pooled and purified three times by protease XIV (5 mg/g of starch in 30 ml of incubation mixture) and six times by the toluene shaking procedure. The isolated and purified starches from the barley contained (dry matter basis) 97.0–98.5% starch, of which 0.0–49.3% was amylose, 0.4–0.9% crude protein (N \times 6.25), 0.2–0.3% ash, and 0.3–0.6% lipids. For additional description of the starches, see Szczodrak and Pomeranz (1991).

Analytical Methods

Protein was determined by Kjeldahl nitrogen (N \times 6.25), Method 46-11A; ash by dry combustion, Method 08-01; moisture by oven drying for 1 hr at 130°C, Method 44-15A; and free lipids by exhaustive extraction with petroleum ether, followed by evaporation to constant weight under vacuum, Method 30-25 (AACC 1983). The content of total β -glucan was measured enzymatically as described by Ahluwalia and Ellis (1984). β -Glucan was extracted from barley flour by 50 mM perchloric acid at 96°C for 3 min. The extract was incubated with *Penicillium*

funiculosum β -glucanase preliminarily heated at 70°C for 1 hr (pH 4.0) to inactivate the starch-degrading enzymes (Bamforth 1983). Appropriate substrate and enzyme blanks were included to correct for any free glucose not emanating from β -glucan. Starch was determined essentially as described for dietary fiber (Prosky et al 1988). The starch was converted to glucose by successive treatments with bacterial α -amylase, followed by protease and fungal amyloglucosidase. The glucose released after β -glucan and starch hydrolysis was measured with a glucose oxidase-peroxidase reagent (Lloyd and Whelan 1969), and results were expressed on a polysaccharide basis (glucose \times 0.9). Quantification of amylose (as percentage of starch) was made according to Hovenkamp-Hermelink et al (1988). All analyses were done at least in duplicate; average results of all analyses were expressed on a dry matter basis.

NIRS

The samples were scanned by NIRS on a Technicon 500 InfraAlyzer (Technicon, Tarrytown, NY). The readings were taken with reference to the ceramic standard throughout the range from 1,100 to 2,500 nm, unless stated otherwise. Statistical analyses were made with the software furnished with the Technicon instrument. The techniques of scanning and the mathematical treatment are described below. The materials were scanned as follows.

All samples were scanned from 1,100 to 2,500 nm at 1-nm intervals that gave 1,400 data points for each sample, but in this case all samples were recorded in four files, each containing 350 of the 1,400 data points. The files represented the following NIRS regions: 1,100–1,449, 1,450–1,799, 1,800–2,149, and 2,150–2,499 nm. Subsequently, all samples were scanned from 1,600 to 2,400 nm at 2-nm intervals, which gave 400 data points per sample, and finally, all samples were scanned from 1,100 to 2,500 nm at 4-nm intervals, which gave 350 data points for each sample. A review of the 1-nm scans in the four files was used to point to important regions for more detailed examination in the single 400-point file.

The samples differed in particle size, density, and color (from bright white for starches and pure β -glucans to brown for crude β -glucan and whole-grain meals). The first two properties, as stated by Norris and Williams (1984), influence penetration of radiation into the samples and reflectance from the samples. To reduce adverse effects of those factors on the accuracy of NIR spectra, the raw spectra recorded as log (1/R) were subjected to mathematical transformation to obtain the first and second derivatives. The derivatives were calculated by keeping 8.0 nm per derivative segment and 14.0 nm between derivative segments. The original data and the first and second derivatives of the various regions of the spectrum were used to establish differences (if present) among whole-grain meals, between covered and hull-less barleys, among barleys differing in amylose-amylopectin ratios, between barley starches and whole meal, between white starch layer and brown layer, between pure barley β -glucan and pure oat β -glucan, between pure barley β -glucan and crude barley β -glucan, and between barley β -glucan and barley starches and whole-grain meal.

RESULTS AND DISCUSSION

The chemical composition of whole-grain meals and isolated and purified starches from three barley genotypes, as well as the analysis of β -glucan preparations, are shown in Table I. The hull-less barleys were higher than the covered barleys in starch, total β -glucan, crude protein, and free lipids. The content of ash, as expected, was consistently higher in covered than in hull-less barleys. Starch was 98.5 and 97.0% in the large- and small-granule preparations, respectively. The purified starch contained less than 1% protein and up to 0.3% ash and 0.6% free lipids. No β -glucan was detected in the purified starch preparations. The amylose content in the starch varied from 0% in waxy barley to 26% in the regular cultivar and up to 49.3% in the high-amylose cultivar. In high-amylose barley, there was a significant difference in amylose content between the large and small granules; the latter

TABLE I
Chemical Composition of Starchy and β -Glucan Samples^a

Source	Material	Starch	Amylose (% of Starch)	β -Glucan	Crude Protein	Free Lipids	Ash
Barley (Glacier, regular)	Covered (whole-grain meal)	62.1	26.0	5.8	15.0	2.9	2.8
	Hull-less (whole-grain meal)	65.7	25.9	6.5	15.8	3.2	1.9
	Purified white starch layer ^b	97.8	24.7	0.0	0.5	0.5	0.2
	Purified brown starch layer ^c	97.0	25.5	0.0	0.6	0.6	0.3
	Mixture ^d	97.4	26.0	0.0	0.5	0.5	0.2
Barley (Glacier, high-amylase)	Covered (whole-grain meal)	54.5	42.3	6.2	13.8	3.4	3.2
	Hull-less (whole-grain meal)	59.7	42.6	7.0	14.6	3.7	2.1
	Purified white starch layer	98.5	43.5	0.0	0.4	0.3	0.2
	Purified brown starch layer	97.8	49.3	0.0	0.5	0.4	0.3
	Mixture	98.0	45.0	0.0	0.4	0.3	0.2
Barley (Wanubet, waxy)	Covered (whole-grain meal)	60.3	0.0	7.6	12.8	4.1	2.6
	Hull-less (whole-grain meal)	64.6	0.0	8.4	13.8	4.5	1.8
	Purified white starch layer	97.9	0.0	0.0	0.8	0.5	0.2
	Purified brown starch layer	97.1	0.0	Trace	0.9	0.6	0.3
	Mixture	97.8	0.0	0.0	0.8	0.5	0.2
Barley	Pure β -glucan	ND ^e	ND	96.8	0.7	ND	ND
Barley	Crude β -glucan	ND	ND	48.7	23.2	ND	4.2
Oat	Purified β -glucan	ND	ND	92.0	6.2	ND	1.9

^a Percent dry matter.

^b Purified six times using toluene extraction.

^c Purified three times using protease and six times by toluene extraction.

^d White and brown layer mixture.

^e Not determined.

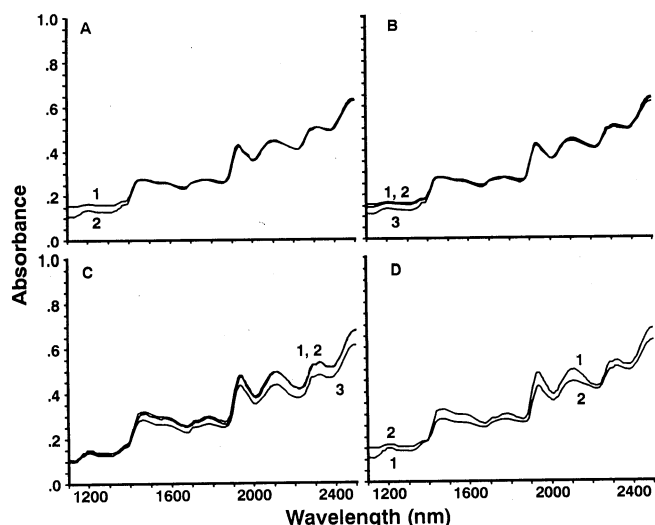


Fig. 1. Near-infrared reflectance spectra, $\log (1/R)$, of barley meals and barley starches. **A**, Covered (1) and hull-less (2) barley meals; **B**, covered regular (1), high-amylase (2), and waxy (3) barley meals; **C**, starches (large granules) from regular (1), waxy (2), and high-amylase (3) covered barleys; **D**, starch (1) (large granules) and meal (2) from regular covered barley.

contained 6% more amylose. A higher amylose content in small starch granules was found in a previous study on starches isolated and purified from covered high-amylase Glacier barley (Szczo drak and Pomeranz 1991). Our results on amylose distribution in barley starch granules differ from those of Stark and Yin (1986), who reported that small granules (brown layer) of regular (25% amylose) barley contained 4% less amylose than the large granules (white layer). The preparations of β -glucan from barley contained from 49% (crude material) to 97% (pure commercial preparation) of this polysaccharide. In purified material from oat, β -D-glucan was 92%.

The $\log (1/R)$ spectra of covered and hull-less regular barley samples (Fig. 1A) show that the covered sample has a higher absorption between 1,100 and 1,500 nm; the samples did not differ, however, in the higher spectral regions. The same pattern

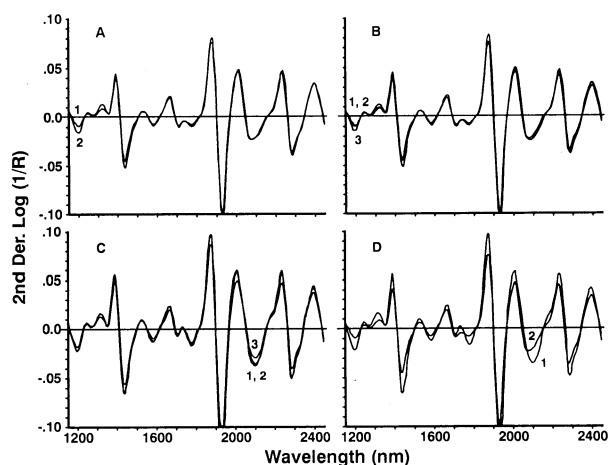


Fig. 2. Plots of second derivatives of near-infrared reflectance spectra, $\log (1/R)$, of barley meals and barley starches. **A**, Covered (1) and hull-less (2) barley meals; **B**, covered regular (1), high-amylase (2), and waxy (3) barley meals; **C**, starches (large granules) from regular (1), waxy (2), and high-amylase (3) covered barleys; **D**, starch (1) (large granules) and meal (2) from regular covered barley.

was obtained for high-amylase and waxy samples (data not shown). Figure 1B illustrates three spectra of covered whole meals from regular, high-amylase, and waxy barley samples. Spectra of starches obtained from those three genotypes are shown in Figure 1C. The spectra of a meal from covered regular barley and of the white starch layer (large granules, mainly) separated from the same samples are compared in Figure 1D.

The nature of the differences among the spectra is difficult to establish because offsetting of curves can be due to particle size and bulk density. As stated by Norris and Williams (1984), coarser samples have higher absorption and higher $\log (1/R)$ values. To reduce the possible effect of the above properties on the spectra, the first and the second derivatives were calculated. Figure 2 shows the second derivatives of the corresponding $\log (1/R)$ curves presented in Figure 1. Second derivatives of the covered and hull-less barleys showed no differences (Fig. 2A). The whole meals of the three barley genotypes also showed very

similar second derivative patterns (Fig. 2B).

Differences were noticed, however, among the white layer starches (mainly large granules) isolated from barleys that varied in amylose content (Fig. 2C). The second derivative of starch from the high-amylose barley differed somewhat from the second derivatives of the other two starches at 2,335 nm. Large differences in the second derivatives were noted between the starch and the whole meal of regular barley (Fig. 2D) as well as between the

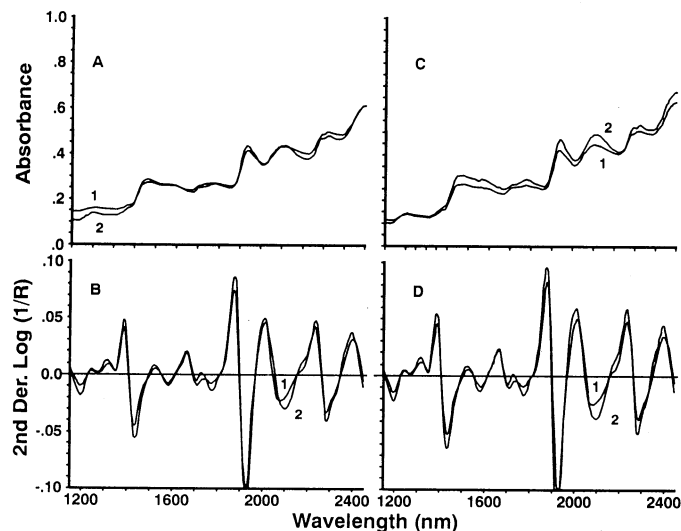


Fig. 3. Near-infrared reflectance spectra, $\log(1/R)$, and second derivatives of covered barley meals and starches. A and B, Spectra and corresponding second derivatives, respectively, of high-amylose barley meal (1) and starch (2). C and D, Spectra and corresponding second derivatives, respectively, of waxy barley meal (1) and starch (2).

starches and meals of high-amylose and waxy genotypes, for which results are shown in Figure 3. The second derivative curves clearly point to differences in the regions of 1,700–1,780 and 1,900–1,970 nm and most pronounced differences in the regions of 2,090–2,200 and 2,270–2,370 nm. It should be pointed out, however, that the 1,900- to 1,970-nm region is dominated by a strong water band.

Figure 4 presents the $\log(1/R)$ values of the white starch layer (mainly large granules) and the brown layer (mainly small granules) for regular, high-amylose, and waxy barleys. Within each genotype a substantial offsetting of curves is recorded along the $\log(1/R)$ axis. This is probably because of the different physical properties of the samples. The white layers of the starches gave higher $\log(1/R)$ values than the brown layers or the mixtures. As stated before, properties such as mean particle size, particle size distribution, bulk density, or nature of the surface influence the penetration of radiation into the samples. Consequently, they also influence the reflectance of light from a sample (Williams 1975, Williams and Thompson 1978).

Figure 5 illustrates the second derivatives of the $\log(1/R)$ curves (for the white and brown layers only) presented in Figure 4. Throughout most of the spectral region, the second derivative curves differ little. In each genotype, however, the brown starch layer differed at 2,300 nm from the mixture (not shown) and especially from the white layer. Whether the differences are because of the high purity of the large granules or the structural-compositional differences between large and small barley granules remains to be determined.

The differences shown in Figure 2C and D and Figure 5 among the second derivatives likely resulted to a greater extent from differences in chemical composition than from differences in physical properties. Kim and Williams (1990) reported that first or second derivatives of $\log(1/R)$ provided better information on composition of feed grains than examination of the raw $\log(1/R)$.

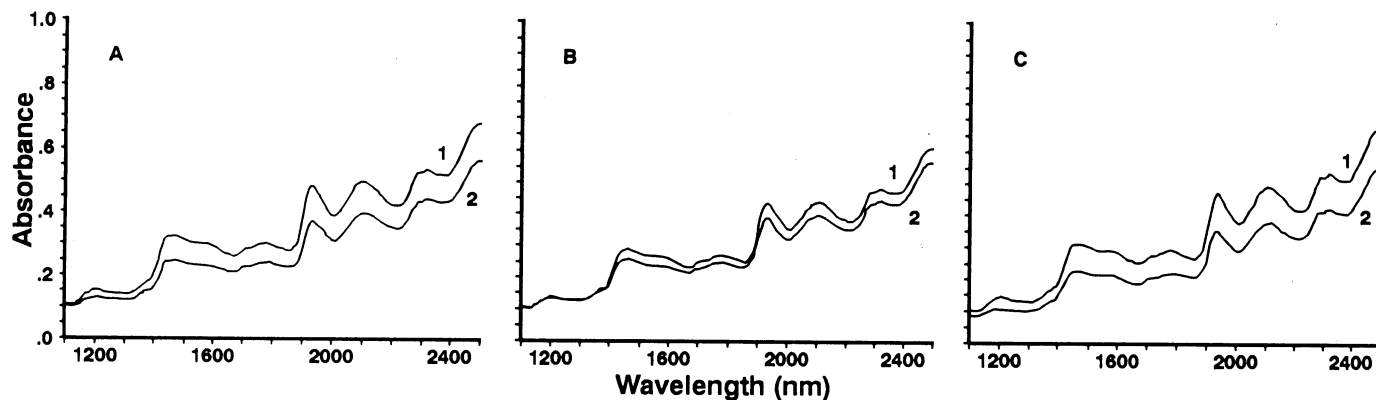


Fig. 4. Near-infrared reflectance spectra, $\log(1/R)$, of large (1) and small (2) starch granules from regular (A), high-amylose (B), and waxy (C) covered barleys.

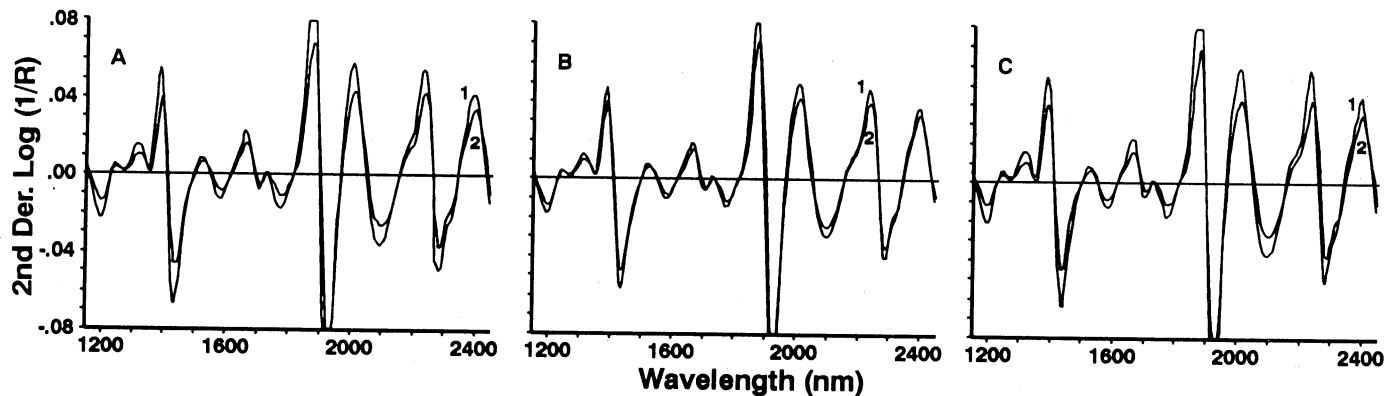


Fig. 5. Second derivatives of near-infrared reflectance spectra, $\log(1/R)$, of large (1) and small (2) starch granules from regular (A), high-amylose (B), and waxy (C) covered barleys.

Before comparing the above samples with samples of β -D-glucans, it might be useful to summarize the consistent results for NIR spectra. 1) No differences were recorded for the three genotypes of covered and hull-less barleys. 2) Among white starches (large granules) from the three barley genotypes, only the high-amylose starch differed slightly from the regular and waxy starches (Fig. 1C). 3) The brown starch layer (small granules) differed slightly from the white starch layer ($2\text{--}5\ \mu\text{m}$ and above $10\ \mu\text{m}$ in starch granule diameter, respectively) (Fig. 4). 4) Among the three genotypes there were no differences in NIR spectra of the brown starch layers. 5) Within each genotype, the whole meal and the isolated starch yielded spectra that differed widely (Fig. 3).

Figure 6 illustrates the scans and second derivatives of $\log(1/R)$ of a pure barley β -glucan and a pure oat β -glucan. Although particle size is affected on the $\log(1/R)$ curves (Fig. 6A), the second derivative curves are very similar (Fig. 6B). The NIR spectrum of pure barley β -D-glucan is compared with spectra of whole meals of regular, high-amylose, and waxy barleys in Figure 7A and with spectra of large granule starches from the three barley genotypes in Figure 7C. The second derivatives of the corresponding spectra are given in Figure 7B and D, respectively. Major differences between β -D-glucan, on one hand, and whole meals and starches, on the other hand, appeared in the range $1,600\text{--}2,400\ \text{nm}$. This particular region was therefore subjected to a more detailed search. Near-infrared spectra of β -D-glucan, whole meals, and starches were recorded from $1,600$ to $2,400\ \text{nm}$ at 2-nm intervals to collect 400 data points per sample. The second derivatives of regular whole meal barley and starch and barley β -D-glucan are compared in Figure 8. Second derivative patterns similar to those of regular barley samples were obtained for the waxy and high-amylose genotypes (not shown here). Figure 8A compares the second derivatives of whole meal and starch (both regular barley). Figure 8B compares the second derivative of a whole-meal regular barley with barley β -D-glucan. A comparison between second derivatives of purified barley β -D-glucan and large starch granules of regular barley is depicted in Figure 8C and among the three samples (whole meal, β -D-glucan, and starch) in Figure 8D.

A close look indicated three areas of major differences, identified by different shading patterns in Figure 8. In the first area, differences were noticed in the $1,700\text{--}1,780\text{-nm}$ range. The second derivative of β -D-glucans is represented by an almost straight line. The whole meal and starch showed peaks that were mutually shifted in position and magnitude. This is indicated by an area located between the derivative curves. The largest difference can be observed between pure starch and β -D-glucan and the smallest one between the whole meal and β -D-glucan. The distinction between β -D-glucan and wheat starch in this range was also reported by Henry (1985b). The region contains the wavelengths $1,702, 1,707, 1,772,$ and $1,773\ \text{nm}$, which are considered principal bands of starch, cellulose, and hemicellulose in food products (for a review of the complex nature of bands, see Osborne and Fearn 1986). Note that the C-H first overtones appear in the above region.

A second area of differences among the three sets of samples in Figure 8 is located between $2,050$ and $2,180\ \text{nm}$. The differences

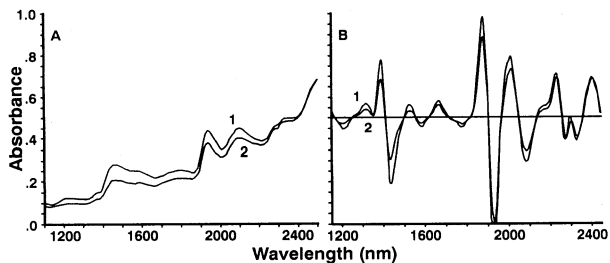


Fig. 6. Near-infrared reflectance spectra, $\log(1/R)$, (A), and second derivatives of spectra (B) of purified β -glucans from barley (1) and oats (2).

concern the wavelengths commonly associated with the interactions of starch and protein. Three wavelengths in this region, $2,060, 2,096,$ and $2,132\ \text{nm}$, were reported as the main absorbance bands for starch in a study of feed grain (Kim and Williams 1990). In our study, large differences were found between starch and barley whole meal and even larger differences between β -D-glucan and barley whole meal. Differences between starch and β -D-glucan probably were due to differences in the structures of these two barley polymers.

Finally, the third region of differences is between $2,260$ and $2,380\ \text{nm}$. Differences in the second derivatives in this region are clearly the most pronounced. The differences are most obvious between β -D-glucan and starch. The assignment of the absorbance wavelengths typical for the region is as follows: $2,268\ \text{nm}$, cellulose; $2,282\ \text{nm}$, starch; and $2,335\ \text{nm}$, cellulose. The wavelength at $2,342\ \text{nm}$ has been reported as a primary point in the NIR calibration for dietary fiber in wheat bran (Horvath et al 1984).

In the study of Henry (1985b), a simplified enzymatic procedure of β -D-glucan determination was used as a reference method. Near-infrared spectra were determined on isolated monosaccharides, cellulose, wheat starch, barley β -D-glucan, and their

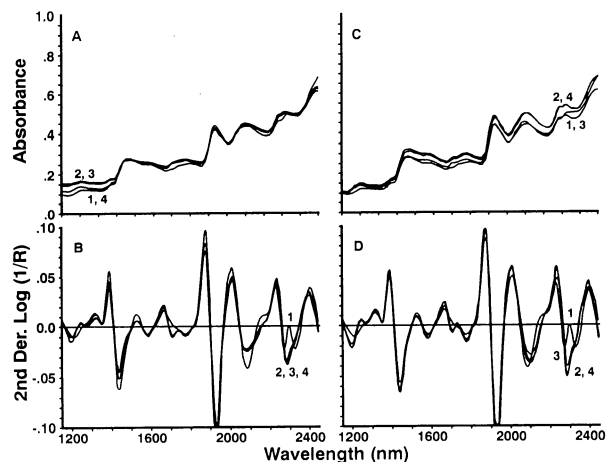


Fig. 7. A and B, Near-infrared reflectance spectra, $\log(1/R)$, and second derivatives of spectra, respectively, of barley β -glucan (1) and meals of regular (2), high-amylose (3), and waxy (4) covered barleys. C and D, Near-infrared reflectance spectra ($\log(1/R)$) and second derivatives of spectra, respectively, of barley β -glucan (1) and starches (large granules) from regular (2), high-amylose (3), and waxy (4) covered barleys.

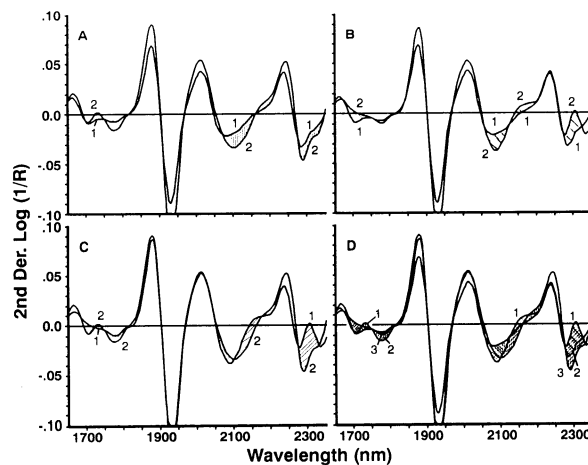


Fig. 8. Second derivatives of near-infrared reflectance spectra, $\log(1/R)$, of barley β -glucan, regular covered barley starch (large granules), and regular covered barley meal. A, Whole meal (1) and starch (2); B, whole meal (1) and β -glucan (2); C, β -glucan (1) and starch (2); and D, β -glucan (1), whole meal (2), and starch (3). Shaded areas show difference between spectra.

mixtures. The correlation coefficient between enzymatically assayed and NIRS-predicted β -D-glucan (based on three wavelengths) was 0.691. When a 12-wavelength equation was used, the correlation coefficient increased to 0.931, but even then the root-mean square error was 0.557%. The best combination of three wavelengths in the study of Henry (1985b) included 1,656, 1,676, and 1,752 nm and did not include the wavelengths in the 2,260- to 2,380-nm range found in this study to be of greatest promise in determination of β -D-glucans.

CONCLUSIONS

This article provides and compares previously unavailable detailed NIR spectra of starches and β -glucans in, and isolated from, regular, waxy, and high-amylose barleys. The differences in NIR spectra of whole meals, purified starches, and β -glucans were described and interpreted. This information should be useful to researchers in studies of cereal polysaccharides, in general, and of barley starches and β -glucans, in particular. Once the characterization by NIRS of the barleys, starches, and β -glucans was completed, the three best wavelengths for estimation of β -glucans were selected, and the results were applied to a series of highly diverse barleys in which β -glucan was determined by a specific but complex and time-consuming method (SzcZodrak et al 1992). The NIRS calibration results were then confirmed by prediction of β -glucans in an independent series of diverse barleys of known (by enzymatic assay) β -glucan content.

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