Comparison of Chemical Composition and Properties between Hard Red Spring and Durum Wheat Endosperm Pentosans[1]

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ABSTRACT

Pentosans (arabinoxylans) from the water-soluble and water-insoluble (sludge) portions of endosperm of durum and hard red spring wheat (HRS) were compared under two different isolation procedures. The first method used acetylation and fractional precipitation of a low-protein pentosan preparation. In the second method, pentosan preparations treated with alpha-amylase were fractionated by DEAE-cellulose column chromatography. The relative proportion of the component sugars in each fraction was determined after acid hydrolysis by gas chromatography. In all cases, durum pentosans contained a higher proportion of arabinose, indicating a more highly branched structure. Pentosans from the water-soluble portion of the endosperm were generally similar in degree of branching to the corresponding "sludge" pentosans. Durum pentosans isolated by the acetylation procedure were somewhat higher in molecular weight than the corresponding HRS pentosans. With the DEAE-cellulose isolation procedure, the HRS pentosans appeared to be of higher molecular weight than the durum pentosans.

Wheat flour pentosans can be generally classified into two types: the water-soluble pentosans and the pentosans associated with the water-insoluble or "sludge" portion of wheat flour. Both the water-soluble pentosans (1) and those associated with the sludge fraction (2) appear to be mixtures of free arabinoxylans and polysaccharide-protein complexes.

The general structural features of wheat pentosans from both the soluble and insoluble portions of the endosperm have been established (3–6) for bread wheats *(Triticum aestivum)*. Perlin (3,4) concluded that the water-soluble pentosans from durum wheat (*T. durum*) were generally similar in structure to those from bread wheat. In general, wheat endosperm pentosans are composed of a main chain of D-xylopyranosyl units, linked beta-1,4. Single-unit L-arabinofuranosyl branches occur to a varying degree, attached to the 2- and 3-positions of the xylose units. Some xylose units contain branches at both the 2- and 3-positions.

Wheat flour pentosans appear to play a role in the quality characteristics of both common bread wheat (7–10) and durum wheat (11). It has been suggested (12) that the endosperm pentosans may be important in accounting for the differences in endosperm properties between durum and hard red spring (HRS) wheats. However, very few experimental data are available on durum pentosans, and little work has been reported on a direct comparison between the pentosans of the two wheat types.

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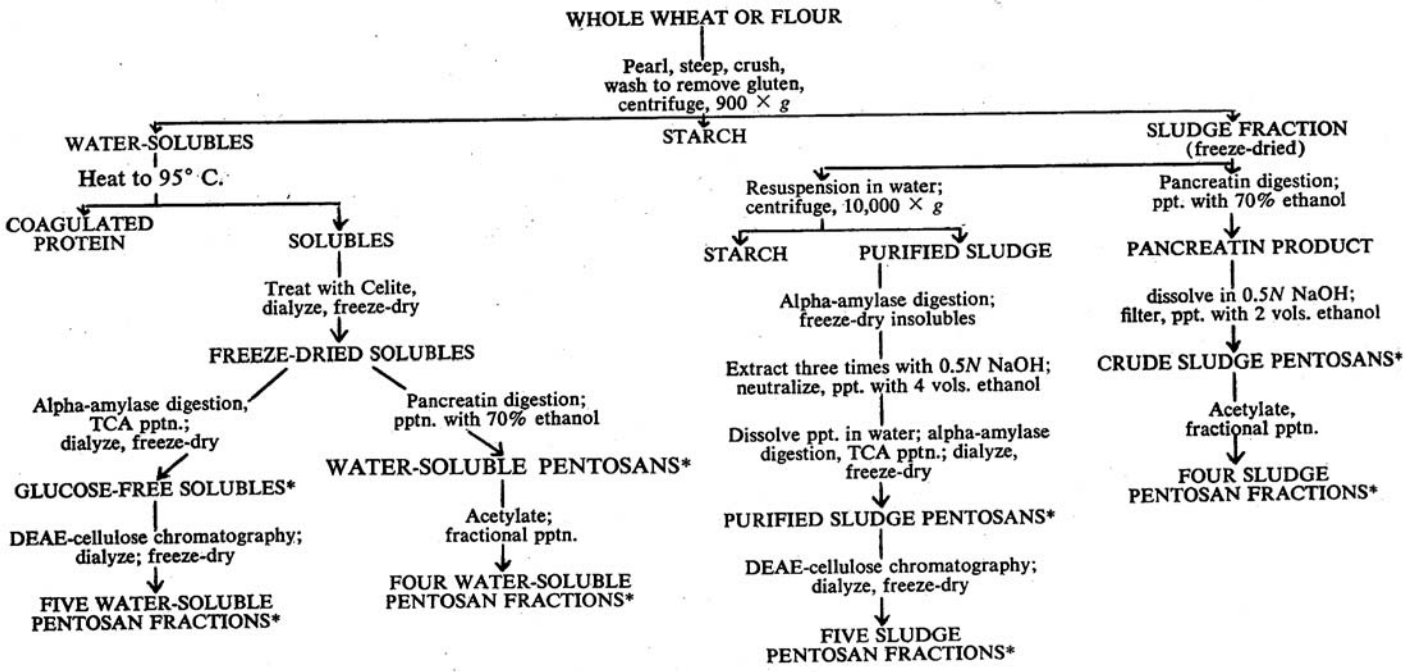

* Asterisk indicates samples which were characterized further.

Fig. 1. Schematic diagram for isolation and fractionation of wheat endosperm pentosans.

This paper reports the results from a study on the chemical composition and properties of the pentosans from both the water-soluble and water-insoluble portions of durum and HRS wheat endosperm. A subsequent paper will report the detailed structural characterization of a pentosan fraction from the insoluble portion of durum wheat endosperm.

MATERIALS AND METHODS

Paper Chromatography

Paper chromatography was performed on Whatman No. 1 paper with ethyl acetate-pyridine-water (10:4:3 v./v.) as solvent. Sugars were visualized with silver nitrate spray reagent (13).

Wheat Samples

Pure-variety samples of HRS wheat (Justin, Selkirk, Thatcher) and durum wheat (Wells, Lakota, Leeds) were used. In addition, a sample of soft wheat (Nugaines) and one of rye were included.

Isolation of Water-Soluble Pentosans

The general procedure for isolation of the various pentosan fractions is outlined in Fig. 1. Pentosan fractions from both the water-soluble and sludge fractions were isolated, under two different techniques: one involved removal of protein and starch with pancreatin, followed by acetylation and fractional precipitation; in the second, starch was removed with alpha-amylase and the remaining protein-carbohydrate mixture was fractionated on DEAE-cellulose.

Acetylation Procedure. Water-solubles and sludge were isolated from pearled Wells (durum) and Selkirk (HRS) wheat. Wheat samples were pearled in a barley pearler and from 500 g. of grain, approximately 400 g. of pearled product was obtained. The pearled grain was steeped in distilled water at 4°C. for 36 hr. The softened grain was crushed in a mortar and the sludge, starch, and water-solubles were washed from the gluten by hand with distilled water. After centrifugation at 900 $\times$ g for 30 min., the water-soluble fraction was decanted from the sedimented starch and sludge. The mucilaginous sludge fraction was scraped from the lower starch layer with a spatula and freeze-dried.

The water-soluble fraction was treated further essentially as described by Simpson (14). The solubles were heated to 95°C. for 5 min., cooled, centrifuged to remove coagulated protein, and stirred with Celite (50 g./liter) for 30 min. to remove additional protein. The water-soluble fraction then was dialyzed for 5 days against distilled water and the nondialyzable material was freeze-dried. Yield of freeze-dried water-solubles was approximately 5 g./800 g. pearled wheat.

Pancreatin (Nutritional Biochemical Corp., Cleveland, Ohio) digestion was performed at pH 7.4 and pH 8.0 according to the procedure of Simpson (14). Pentosans were precipitated from the digestion solution by addition of ethanol to a concentration of 70%. The precipitate was centrifuged, washed with ethanol and ethyl ether, and dried under vacuum at 40°C. for 12 hr. This product (water-soluble pentosans) was obtained in approximately 30% yield from the freeze-dried water-solubles. The protein content

was 2% in both the durum and HRS wheat samples. After acid hydrolysis, paper chromatography indicated that D-xylose and L-arabinose were the major components. Traces of D-glucose and D-galactose were visible on heavily spotted papers.

Water-soluble pentosan fractions (2 g.) were acetylated according to the procedure described by Perlin (3). The acetylated products were dissolved in acetone (1.0 g./100 ml.) and fractionally precipitated with petroleum ether (b.p. 30°–60°C.) (3). Four pentosan fractions were obtained from each sample in this way. Recoveries ranged from 72 to 79%.

Acetylated pentosan fractions were deacetylated with sodium hydroxide solution as described by Montgomery and Smith (5). The pentosan fractions were isolated as white to light tan products.

DEAE-Cellulose Procedure. Water-solubles were isolated from HRS, durum, and soft wheat flours and rye flour by extraction with water (1 part water to 2 parts sample). Water and sample were mixed in a Waring Blendor for 4 min. After centrifugation for 20 min. at 10,000 × *g*, the supernatant solution was heated, treated with Celite, dialyzed, and freeze-dried as described above. Yields of freeze-dried water-solubles were approximately 1%.

Soluble starch was removed from the water-solubles with crystalline alpha-amylase (Nutritional Biochemical Corp., Cleveland, Ohio) as described by Kündig *et al.* (1). Glucose-free solubles were obtained in 50-60% yield from the water-solubles. Protein contents were 13, 20, 23, and 14% for the glucose-free solubles from the durum, HRS, and soft wheats and rye samples, respectively. In all cases, paper chromatography after acid hydrolysis indicated that D-xylose, L-arabinose, and D-galactose were the only sugars present.

Glucose-free solubles were fractionated into five fractions by stepwise elution from a 2 × 50-cm. column of DEAE-cellulose (borate form) as described by Kündig *et al.* (1). The DEAE-cellulose (exchange capacity 1.0 meq./g.) column was prepared in the borate form according to Neukom and Kündig (15).

The sample (300 mg.) was dissolved in a small amount of distilled water and applied to the top of the column. After the sample had been allowed to penetrate into the DEAE-cellulose, elution was accomplished stepwise with the following eluants: 1) distilled water; 2) 0.0025*M* $Na_2B_4O_7$; 3) 0.025*M* $Na_2B_4O_7$; 4) 0.125*M* $Na_2B_4O_7$; 5) 0.5*N* NaOH. The column effluent was collected in 15-ml. fractions with the use of an automatic fraction collector. Protein elution was followed by means of a recording UV monitor. Carbohydrate content in each tube was estimated by the orcinol procedure (16). Tubes corresponding to each carbohydrate peak were combined, dialyzed to remove inorganic ions, and freeze-dried. The nitrogen content of each fraction was estimated by the method of Folin-Ciocalteu as modified by Lowry *et al.* (17). Recoveries of material from the column were 70–80%. In all cases, the component sugar ratio was essentially identical in the combined recovered fractions and the unfractionated material.

Isolation of Sludge Pentosans

Acetylation Procedure. The isolation of the sludge fraction from Wells

(durum) and Selkirk (HRS) wheats was described above. Yields of sludge were approximately 100 g./800 g. pearled wheat. Sludge was treated with pancreatin for 4–6 days as described by Simpson (14). The pancreatin product was precipitated from the digestion mixture by addition of an equal volume of 95% ethanol. The precipitate was isolated, washed with ethanol and ethyl ether, and dried under vacuum at 40°C. for 12 hr.

The pancreatin product was suspended in oxygen-free 0.5*N* sodium hydroxide solution (1 g./200 ml.) and stirred under nitrogen for 3 hr. The mixture was filtered through cloth and neutralized with 50% acetic acid, and the pentosans were precipitated with 2 vols. of 95% ethanol. The precipitate was collected, washed with ethanol and ethyl ether, and dried under vacuum at 40°C. for 12 hr. Yields of product (crude sludge pentosans) were approximately 3.5 g./5 g. pancreatin product. Protein contents were 1.5–2%. After acid hydrolysis, paper chromatography indicated that D-xylose and L-arabinose were the major sugar components, with significant amounts of D-glucose also present. Traces of D-galactose were visible on heavily spotted papers.

Crude sludge pentosans (2 g.) were acetylated as described by Montgomery and Smith (6). The acetylated product was dissolved in acetone. Complete dissolution was not obtained, and the acetone-insoluble fraction was isolated. The acetone solution was fractionally precipitated with petroleum ether (b.p. 30°–60°C.) as described by Perlin (3). Four pentosan fractions were obtained from each sample in this way. Recoveries ranged from 75 to 82%.

Acetylated sludge pentosan fractions were deacetylated as described by Montgomery and Smith (5). Sludge pentosans were similar in appearance to the water-soluble pentosans.

DEAE-Cellulose Procedure. Crude sludge was obtained from HRS (Selkirk) flour and durum (Leeds) semolina by the procedure of Gilles, Kaelble, and Youngs (18). This material was further purified by resuspension in water in a Waring Blendor followed by centrifugation at 10,000 $\times$ *g* (19). The upper layer of mucilaginous, purified sludge was removed from the starch layer with a spatula and freeze-dried.

Further purification was done as described by Cole (2). Sludge was treated with crystalline alpha-amylase, followed by sodium hydroxide extraction and precipitation with ethanol. The purified sludge pentosans were isolated by freeze-drying after a final alpha-amylase treatment. Yields were 0.5–0.8% from purified sludge. Paper chromatography after acid hydrolysis indicated that D-xylose, L-arabinose, and D-glucose were the major sugars, with traces of D-galactose also present. Protein content was 7% for the durum sample and 18% for the HRS sample.

The purified sludge pentosans were fractionated into five fractions on DEAE-cellulose by the procedure described above for the water-soluble pentosans.

Ratio of Component Sugars

Ratio of component sugars in the various pentosan fractions was de-

termined by gas chromatography. The procedure used was essentially that of Sawardeker, Sloneker, and Jeanes (20).

The pentosan sample (10 mg.) was hydrolyzed with 1*N* sulfuric acid for 4 hr. at 100°C. and neutralized with barium carbonate. After centrifugation, the solution was evaporated to dryness under reduced pressure and reduced with sodium borohydride, and the alditols were concentrated to a syrup under reduced pressure. The alditol mixture was acetylated with acetic anhydride and pyridine (20). The acetylation mixture was concentrated under reduced pressure and taken up in chloroform for injection into the gas chromatograph.

Gas chromatography was performed with a Beckman GC-2A chromatograph equipped with a flame ionization detector. Separations were made on a ⅛-in. Cu column, 10 ft. long, packed with 3% ECNSS-M (Applied Science Laboratories, Inc.) on 100- to 120-mesh Gas Chrom Q (20). Column temperature was 190°C. with a helium carrier gas flow rate of 30 cc./min. Mannitol hexaacetate was used as an internal standard. The acetates of galactitol and glucitol were not separated under these conditions. When both alditols were present, they were calculated as the major fraction (determined by paper chromatography). Relative proportions of the various sugars present were calculated from peak areas determined by triangulation (20).

Optical Rotation and Intrinsic Viscosity

Specific optical rotations of various pentosan fractions were determined in 0.5*N* sodium hydroxide solution.

Intrinsic viscosities were determined in 0.5*N* sodium hydroxide solution at 25°C. with a Ubbelohde viscometer.

Molecular Weight

Number average molecular weights were determined (21) with a high-speed recording membrane osmometer (Model CSM-2, Melabs, Inc., Palo Alto, Calif.). Measurements were made in 0.1*M* sodium chloride solutions at 25°C.

Hydration Capacity

Portions of various pentosan fractions isolated by the acetylation procedure were weighed into small aluminum weighing dishes. These were placed in a desiccator containing a water reservoir. The samples were weighed every 2 days until constant weight was reached. The gain in weight divided by the original sample weight was considered the percent hydration of that sample.

RESULTS AND DISCUSSION

Pentosans were isolated in this work by two separate methods. The acetylation procedure has been used generally (3–6) to isolate a pentosan fraction for structural characterization. Protein is removed as completely as possible in this case. Recently, the DEAE-cellulose procedure (1,2) has been used to fractionate pentosan preparations which contain significant amounts of protein. The essentially pure arabinoxylan fractions (I and

II) obtained with this method (eluted with water and 0.0025*M* $Na_2B_4O_7$) appear to be generally similar to the major fractions obtained by the acetylation procedure (1). The other fractions are believed to be protein-carbohydrate complexes (1,22).

In this work, both procedures were used to compare the endosperm pentosans from durum and HRS wheat. While all fractions from the DEAE-cellulose column were collected and analyzed, major emphasis was placed on the pure arabinoxylan fractions I and II.

Table I summarizes the data for the water-soluble pentosan fractions obtained by the acetylation procedure. The degree of branching (arabinose: xylose) is about the same for all fractions from a given sample. Fractionation apparently was based primarily on molecular-weight differences.

Table II shows typical data for the water-soluble pentosans fractionated by DEAE-cellulose column chromatography. The data for the major water-

TABLE I

WATER-SOLUBLE PENTOSAN FRACTIONS (ACETYLATION PROCEDURE)

FRACTION	YIELD	RATIO, ARABINOSE: XYLOSE	$[\alpha]_D^{25}$	$[\eta]$	MOLECULAR WEIGHT
	%				
Durum (Wells)					
Unfractionated	....	1:1.2	....	....	....
I	16.5	1:1.2	−145°	4.9	119,000
II	28.8	1:1.3	−152°	3.8	95,000
III	25.2	1:1.2	−122°	2.2	55,000
IV	7.7[a]	....	....	....	....
HRS (Selkirk)					
Unfractionated	....	1:1.5	....	....	....
I	16.1	1:1.7	−137°	2.5	64,000
II	23.1	1:1.5	−136°	3.1	90,000
III	24.1	1:1.4	−113°	1.6	50,000
IV	8.9[a]	....	....	....	....

[a] Material insufficient for further testing.

TABLE II

DEAE-CELLULOSE COLUMN CHROMATOGRAPHY OF WATER-SOLUBLE PENTOSANS

FRACTION	YIELD[a]	PROTEIN	RATIO ARABINOSE: XYLOSE: GLUCOSE	FRACTION	YIELD[a]	PROTEIN	RATIO ARABINOSE: XYLOSE: GALACTOSE
	%	%			%	%	
Durum (Leeds)				HRS (Thatcher)			
Unfractionated	....	12.0	1:0.8:0.2	Unfractionated	....	17.0	1:0.9:0.3
I	16.6	1.6	1:1.5:	I	20.4	1.8	1:1.8: ....
II	35.9	1.2	1:1.0:	II	18.0	1.4	1:1.2: ....
III	7.4	11.9	1:0.7:0.3	III	5.4	2.9	1:0.9:0.1
IV	27.2	17.7	1:0.2:0.7	IV	25.3	12.5	1:0.2:0.8
V	12.9	27.0	1:0.9:0.4	V	30.9	29.3	1:1.1:0.3

[a] Yields based on material recovered.

soluble arabinoxylan fractions (I and II) obtained by this method from all the samples studied are shown in Table III. Fractions III, IV, and V contained galactose and higher amounts of protein. Fraction IV was primarily an arabinogalactan. Fractions I and II showed no UV absorption on the column monitor, indicating very low protein content. Some protein was detected by the Folin-Ciocalteu method. However, values were generally below 2%. Fraction I was less highly branched than fraction II in all cases. Some degree of fractionation apparently was a result of degree of branching. Molecular weight also appeared to affect fractionation, since in the samples studied, fraction I was of lower molecular weight than the corresponding fraction II.

TABLE III
WATER-SOLUBLE PENTOSAN FRACTIONS (DEAE-CELLULOSE PROCEDURE)

FRACTION	YIELD	PROTEIN	RATIO, ARABINOSE: XYLOSE	RATIO, ARABINOSE: XYLOSE FRACTION I + FRACTION II	MOLECULAR WEIGHT
	%	%			
Durum (Lakota)					
I	13.2	2.0	1:1.4	1:1.1	....
II	25.8	4.4	1:1.0		51,000
Durum (Leeds)					
I	16.6	1.6	1:1.5	1:1.1	22,000
II	35.9	1.2	1:1.0		47,000
HRS (Justin)					
I	17.2	0.7	1:1.8	1:1.5	56,000
II	23.0	6.6	1:1.3		92,000
HRS (Thatcher)					
I	20.4	1.8	1:1.8	1:1.5	96,000
II	18.0	1.4	1:1.2		109,000
Soft wheat (Nugaines)					
I	17.1	1.8	1:2.0	1:1.9	76,000
II	23.8	1.5	1:1.8		....
Rye					
I	22.0	1.7	1:1.7	1:1.5	120,000
II	19.6	2.0	1:1.2		133,000

The pentosans from the water-soluble portion of durum wheat endosperm were more highly branched than those from HRS wheat. This was true for pentosans isolated by both the acetylation and DEAE-cellulose procedures. There were fewer xylose units per arabinose unit in the durum fractions compared to corresponding HRS fractions. This was particularly evident in the DEAE-cellulose procedure for the ratio of the combined fractions I and II. The fact that both procedures led to the same conclusion is strong evidence that the observation is a real one and not an artifact of the isolation procedure. The soft wheat sample had a degree of branching lower than that of either durum or HRS wheat. Rye water-soluble pentosans gave essentially the same degree of branching as the HRS wheat sample.

An examination of Perlin's data (3) showed that the durum sample

studied in his work may have been more highly branched than the corresponding bread wheat sample.

Specific optical rotation, intrinsic viscosity, and molecular weight were used to characterize the water-soluble pentosan fractions isolated by the acetylation procedure (Table I). All had large negative optical rotations, which indicated a basic structure of beta-1,4-linked D-xylose units. As would be expected for polysaccharides of similar structure, intrinsic viscosity was directly related to molecular weight. It appeared that, for the fractions isolated by this procedure, the durum pentosans had somewhat higher molecular weights. The opposite result was noted for the DEAE-cellulose fractions. In this case, the water-soluble pentosans of HRS wheat appeared to be of higher molecular weight.

Table IV summarizes the data for the sludge pentosan fractions isolated by the acetylation procedure. Fractions I and II were essentially pure

TABLE IV
SLUDGE PENTOSAN FRACTIONS (ACETYLATION PROCEDURE)

FRACTION	YIELD	RATIO, ARABINOSE: XYLOSE: GLUCOSE	$[\alpha]_D^{25}$	$[\eta]$	MOLECULAR WEIGHT
	%				
Durum (Wells)					
Unfractionated	....	1:1.2:0.6	....	....	....
I	35	1:1.3:0.2	−126°	7.0	148,000
II	27	1:1.1:0.2	−104°	4.9	132,000
III	7	1:1.0:4.3	....[a]	....	....
Acetone-insoluble	13	1:1.2:0.5	....	....	....
HRS (Selkirk)					
Unfractionated	....	1:1.4:1.3	....	....	....
I	36	1:1.5:0.2	−100°	5.9	....
II	11	1:1.3:0.2	....	4.3	....
III	14	1:1.1:2.8	....	....	....
Acetone-insoluble	14	1:1.4:0.4	....	....	....

[a] Because of insufficient material available, not all tests were performed on all samples.

arabinoxylans. Fraction III contained the major portion of D-glucose in each case. Some degree of fractionation due to branching was indicated; however, as was the case for the water-soluble pentosans, molecular size appeared to be the major variable that controlled fractionation.

Table V shows the data for the sludge fractions isolated by the DEAE-cellulose procedure. The unfractionated material contained only traces of D-galactose, but did contain appreciable D-glucose, in spite of two alpha-amylase treatments. Fractions I, II, and III were arabinoxylans. Glucose was contained in fractions IV and V. Some protein was detected in all fractions. However, fraction V contained the major portion of the protein. As was indicated for the water-soluble pentosan fractions, fraction I was less highly branched than fraction II. Fraction III was the most highly branched of the sludge arabinoxylan fractions. The ratio of arabinose:xylose for the combined fractions I and II was 1:1.3 for the durum sample and 1:1.5 for the HRS sample.

TABLE V

DEAE-CELLULOSE COLUMN CHROMATOGRAPHY OF SLUDGE PENTOSANS

FRACTION	YIELD[a]	PROTEIN	RATIO, ARABINOSE: XYLOSE: GLUCOSE
	%	%	
Durum (Leeds)			
Unfractionated	....	6.6	1:1.1:0.3
I	12.0	2.1	1:1.7: ....
II	12.0	2.7	1:1.1: ....
III	16.1	2.0	1:1.0: ....
IV	21.0	3.2	1:0.8:0.1
V	38.9	6.5	1:1.2:0.5
HRS (Selkirk)			
Unfractionated	....	18.0	1:1.2:0.1
I	6.9	2.0	1:1.8: ....
II	9.4	3.0	1:1.2: ....
III	9.9	6.3	1:1.0: ....
IV	28.3	4.0	1:0.8:trace
V	45.5	17.0	1:1.5:0.3

[a] Yields based on material recovered.

As was the case for the water-soluble pentosans, the sludge pentosan fractions from durum wheat were more highly branched than the corresponding HRS wheat fractions. This was true for both isolation procedures used in this work. It is apparent from these data that durum endosperm arabinoxylans, in general, have a slightly higher degree of branching than the corresponding endosperm arabinoxylans from HRS wheat.

Viscosity data for the major sludge fractions isolated by the acetylation procedure (Table IV) indicated that the durum pentosans were of slightly higher molecular weight than the corresponding HRS wheat sludge pentosans.

The differences observed between HRS and durum wheat endosperm pentosans are significant but relatively small. However, a small difference in degree of branching might markedly alter the degree and type of the interaction of polysaccharide with protein. Differences in molecular weight also could alter the interaction reaction and affect such things as water-absorption. These differences may account, in part, for the observed differences in endosperm properties between durum and HRS wheats.

The amount of water taken up from a saturated atmosphere by the various fractions was determined. All were highly hygroscopic. Hydration ranged from 80 to 300%. In these experiments, no definite trend of differences between the durum and HRS wheat fractions was observed.

Montgomery and Smith (23) have suggested that the pentosans from the water-soluble portion of wheat endosperm may be less branched than the pentosans from the sludge portion of the endosperm. The data obtained in this work do not support this theory. In general, degree of branching was similar for both water-soluble and sludge pentosans within each wheat type. The major difference observed between the water-soluble and sludge pentosans isolated by the acetylation procedure was the higher molecular weight of the sludge fractions.

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