THE CARBOHYDRATES OF VARIOUS PIN MILLED AND AIR-CLASSIFIED FLOUR STREAMS. II. STARCH AND PENTOSANS[1]

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ABSTRACT

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Selected flour streams from each of three cultivars of hard red spring wheat obtained from a pilot mill were pin milled and air-classified into high-, intermediate-, and low-protein-containing fractions. The starch isolated from the high-protein-containing flour fractions showed the highest pasting temperature, highest peak height, and lowest setback values. This starch also contained the highest amount of lipid material, had the highest water-binding capacity values, and the greatest percentage of small granules. Total water-solubles and water-soluble pentosans were isolated from the pin milled and air-classified flour fractions, with the high-protein flour fraction containing the highest amount. The protein content of the amylase-treated soluble pentosans was lowest and highest for those pentosans extracted from the high- and intermediate-protein-containing flours, respectively. The ratio of component sugars for the pure pentosan fraction obtained by DEAE-cellulose chromatography of the pin milled and air-classified fractions from a particular cultivar revealed no differences. However, differences were noted between cultivars. Intrinsic viscosity values for the DEAE-cellulose essentially pure pentosan fraction obtained from the pin milled and air-classified flour fractions were similar.

The effect of overgrinding flour on its properties and in particular on the polysaccharides has been studied by various research groups. Alsberg and Griffing (1) reported that overgrinding flour injures the starch granules so that a part of the starch swells and disperses when the flour is doughed. Jones (2) found that, in general, the harder the wheat the higher the starch damage and maltose values. In more recent work, Sulivan *et al.* (3) studied the effect of fine grinding and air classification on maltose value and gassing power, and the dependence of these two measurements on specific surface and starch damage. These workers showed that the shearing action and pressure of roller grinding, such as occur in conventional milling, produce more starch damage in achieving the same granulation as pin, stud, or fluid energy mills. Farrand (4) air-classified a hard and a soft wheat flour into a fine and coarse fraction. His results showed that, for both flours and both fractions, the level of starch damage was increased in the fine fraction.

Kulp (5) determined that the swelling of wheat starches from soft wheats was higher than that of starches from hard wheats. He also found that solubility of hard wheat starches was increased by milling, whereas changes in solubility of soft wheat starches due to milling were nominal.

Garcia *et al.* (6) found air classification to have only a minor effect on modifying pentosan content of corn and wheat-germ flours. However, they reported that the pentosan content was higher in the fine air-classified fractions, which also contained the higher protein and ash contents.

In a study by Holas and Hampl (7), five cultivars of rye were milled on an

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automatic industrial mill. It was found that protein content of the flour streams ranged from 5.55 to 17.71% dry weight and increased continuously with rising ash content. Likewise, as ash content increased, total pentosan content increased and starch content decreased.

In a recent study by MacArthur and D'Appolonia (8), certain differences in reducing and nonreducing sugars, total sugars, and 5 individual free sugars were shown to exist among different pin milled and air-classified flour fractions obtained from selected flour streams from different hard red spring wheat cultivars.

In a continuation of that study (8), additional research was conducted to determine if there was a difference in the properties of starch and pentosans in flour fractions resulting from pin milling and air classification. The effects of flour stream and wheat cultivar on these components also were examined.

MATERIALS AND METHODS

Flour Samples

A description of the flour samples and milling methods used in this study has previously been reported (8). Flour streams from three cultivars of hard red spring wheat, Waldron, Red River 68, and Pitic 62 were selected for investigation. Each stream was pin milled and air-classified into three fractions designated as: F-1 (first cut fine fraction), a high-protein fraction; F-2 (second cut fine fraction), a low-protein fraction; and C-2 (second cut coarse fraction), the intermediate-protein fraction (9).

Starch Isolation

Starch was isolated from the unfractionated flour and from the three pin milled and air-classified flour fractions obtained from selected flour mill streams of Waldron, Red River 68, and Pitic 62 according to the dough kneading procedure described by Walden and McConnell (10).

Starch Pasting Properties

Starch gelatinization curves were obtained by means of a Brabender Visco-Amylograph®, using the procedure described by Medcalf and Gilles (11).

Water-Binding Capacity

The procedure for water-binding capacities initially described by Yamazaki (12) and later modified by Medcalf and Gilles (13) was used.

Starch Damage Determination

Starch damage was determined colorimetrically using the method proposed by Williams and Fegol (14). In this study, starch damage of the unfractionated flour, of the isolated starch, and of the pin milled and air-classified flour fractions was determined. The only modification was the amount of sample used for the analysis. Due to the higher level of starch damage in the pin milled and air-classified flour fractions, the sample size was reduced. Starch damage was expressed in Farrand Equivalent Units.

Total Lipid Content

Total lipid in selected flour and starch samples was determined by AACC methods (15); however, for starch, certain modifications were incorporated. The initial acid concentration was changed to a 1:1 ratio and the hydrolysis time was increased to 2 hr according to Rogols (16). During hydrolysis, the beakers were covered to prevent evaporation. After extraction of the fat, and drying, the residue was extracted with carbon tetrachloride as described by Schoch (17). The extract was dried at 100°C for 2 hr and allowed to cool 0.5 hr at room temperature before it was weighed.

Microscopy of Starch Granules

A Nikon microscope Model L-Ke with a built-in Koehler illuminator was used in this study. The microscope was fitted with a Nikon automatic exposure setting Microflex Model AFM photomicrographic unit. Starch isolated from the unfractionated and pin milled and air-classified flour fractions of selected streams was examined; a staining solution containing 0.1% potassium iodate and 0.4% iodine was used.

Photomicrographs of the different starches were obtained using a magnification of 400× under normal and polarized light.

Isolation of Crude Water-Soluble Pentosans and Total Water-Solubles

Crude water-soluble pentosans were isolated from the unfractionated flour and pin milled and air-classified flour fractions according to the procedure of D'Appolonia (18). The flour sample was mixed in a Waring Blendor at a ratio of 1 part flour to 2.5 parts water for 2 min at low speed, then centrifuged for 20 min at 10,000 × *g*. The supernatant was filtered quantitatively into a graduated cylinder, stirred thoroughly, and divided into two equal portions. One portion was shell frozen and freeze-dried, and represented the total water-soluble extractable material. The remaining portion of the supernatant was used for crude pentosan isolation (18).

Amylase Treatment of Crude Pentosans

The crude pentosans were treated with hog pancreas α-amylase 2× crystallized (Nutritional Biochemical Corp., Cleveland, Ohio) to remove soluble starch according to the procedure of Kündig *et al.* (19).

DEAE-Cellulose Chromatography of Pentosans

The α-amylase-treated water-soluble pentosans were fractionated into five fractions by stepwise elution from a 2.4 × 30-cm column of DEAE-cellulose (borate form). The DEAE-cellulose (Whatman DE 23) had an exchange capacity of 1.0 meq/g.

The sample (250 mg) was dissolved in a small amount of distilled water and applied to the top of the column. The procedure used to fractionate the pentosans has been described previously (20).

Ratio of Component Sugars in DEAE-Cellulose Pentosan Fractions

The ratio of component sugars in the various pentosan fractions was determined by gas chromatography according to the procedure of Medcalf *et al.* (20).

Protein Content

Protein content (N × 5.7) of the crude and α-amylase-treated pentosans was determined by micro-Kjeldahl according to AACC methods (15). The protein content of the DEAE-cellulose fractions was estimated by the Folin-Ciocalteu method as modified by Lowry *et al.* (21).

Intrinsic Viscosity

Intrinsic viscosity of the DEAE-cellulose pentosan fraction (FI) was measured by dissolving a portion in $0.5N$ NaOH solution at 25° C. An Ubbelohde viscometer was used for the viscosity measurement.

Total Pentosan Content

Total pentosan content in the various flours was determined according to the method of Dische and Borenfreund (22) as modified by Cracknell and Moye (23). An exact weight of flour sample (2–5 mg) was placed into test tubes and washed down with 10 ml of a freshly prepared reagent mixture consisting of 110 ml cholesterol grade glacial acetic acid, 2 ml concentrated hydrochloric acid, 4.5 ml of a freshly prepared solution of 20% phloroglucinol in ethanol, and 1 ml of 1.75% aqueous glucose solution. The tubes were then placed in a vigorously boiling water bath for 16 min, quickly cooled to room temperature, and immediately read at 552 and 510 nm. Pentosan content was calculated by

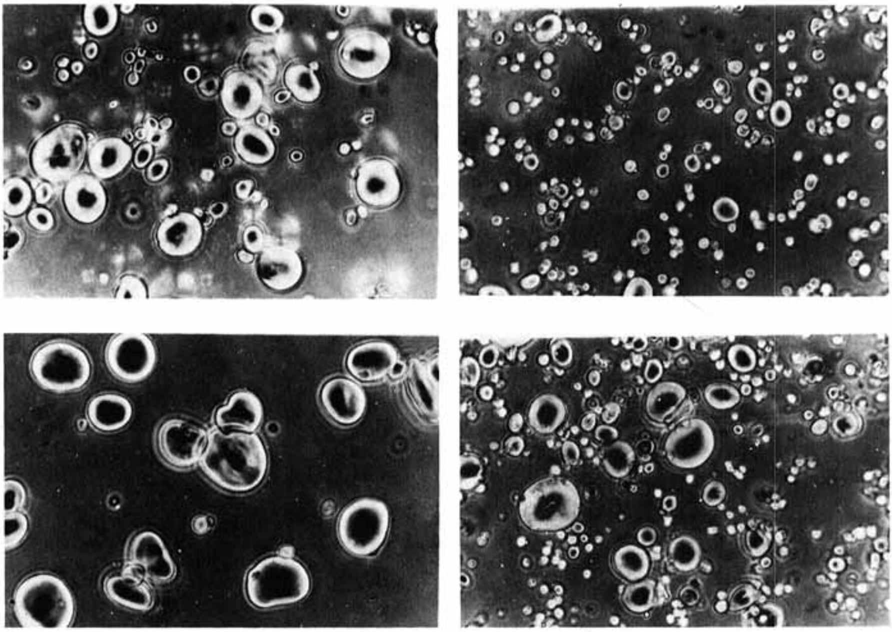

Fig. 1. Photomicrographs of different starch granules. Magnification (400×). Upper left: unfractionated flour; upper right: high-protein fraction F-1; lower left: low-protein fraction F-2; lower right: intermediate-protein fraction C-2.

subtracting the 510-nm reading from the 552-nm reading [the difference being directly proportional to the pentose content of the hydrolysates (23)] and referring to a standard curve based on pure xylose.

RESULTS AND DISCUSSION

Starch Isolation

Photomicrographs of the starch granules isolated from the unfractionated, high-protein fraction F-1, low-protein fraction F-2, and intermediate-protein fraction C-2 of flour stream 2M, cultivar Waldron, are shown in Fig. 1. The starch from the unfractionated flour shows a distribution of large, intermediate, and small granules, whereas the F-1 fraction consists mainly of small starch granules. Starch isolated from the F-2 fraction consists mainly of large granules, while the C-2 fraction starch has a distribution similar to that isolated from the unfractionated flour.

Table I shows the amounts of prime starch and sludge components obtained from Waldron, Red River 68, and Pitic 62 for the unfractionated flour and pin

TABLE I
Recovery of Starch and Sludge from Unfractionated Flour and Pin Milled and Air-Classified Flour Fractions of Different Wheat Cultivars[a]

Flour Stream	Flour Fraction	Prime Starch			Sludge			Prime Starch and Sludge		
		A[b] %	B[b] %	C[b] %	A[b] %	B[b] %	C[b] %	A[b] %	B[b] %	C[b] %
2M	Unfractionated	56	52	53	19	25	27	75	77	80
	F-1	44	44	48	20	19	14	64	63	62
	C-2	60	64	54	13	9	21	73	73	75
	F-2	64	67	72	16	12	11	80	79	83
4M	Unfractionated	51	61	46	25	17	30	76	78	76
	F-1	43	44	59	20	20	6	63	64	65
	C-2	58	65	39	15	10	31	73	75	70
	F-2	49	69	61	29	12	16	78	81	76
2B	Unfractionated	58	58	65	14	18	18	72	76	83
	F-1	42	45	46	20	18	18	62	63	58
	C-2	46	59	76	25	13	13	71	72	89
	F-2	49	67	65	20	12	15	69	79	80
5B	Unfractionated	48	43	49	15	17	25	63	60	74
	F-1	31	50	40	28	30	15	59	80	55
	C-2	34	46	44	23	12	22	57	58	66
	F-2	41	55	50	30	17	17	71	72	67
T	Unfractionated	56	57	21	24	16	60	80	73	81
	F-1	40	37	12	19	20	40	59	57	52
	C-2	51	62	28	24	16	31	75	78	59
	F-2	50	59	47	29	14	29	79	73	76

[a] Results expressed on a dry basis.

[b] Data obtained for the cultivars Waldron, Red River 68, and Pitic 62, respectively.

milled and air-classified flour fractions for each of the selected streams. In general, only small differences in the yield of prime starch plus sludge were noted among the different streams for the same flour fractions. For all streams except one, F-1 contained the least amount of starch plus sludge as would be expected due to the higher protein contents of this fraction (8).

The nitrogen content of the isolated starches (data not shown) was similar in all cases except for the slightly higher content of that isolated from the high protein-containing fraction of Pitic 62. The protein content of the isolated sludges showed a wide range of values among the three cultivars. However, it was not possible to establish any definite trends. In general, Waldron showed the lowest values for sludge protein, while the tailings flour of Pitic 62 showed the highest.

Starch Pasting Properties

The starch pasting properties of the isolated starches from Waldron are shown in Table II. In all streams the starch pasting temperature was highest for the F-1 fraction. Such a result may be due to the greater number of small starch granules

TABLE II
Pasting Properties of Starches Isolated from Waldron Unfractionated Flour and Pin Milled and Air-Classified Flour Fractions

Flour Stream	Flour Fraction	Pasting Temperature °C	Peak Height BU	Peak Temperature[a] °C	15-min Height BU	Setback BU
2M	Unfractionated	83.5	380	95 (3.00 min)	380	920
	F-1	87.0	480	95 (2.00 min)	455	790
	C-2	81.0	365	95 (1.50 min)	355	890
	F-2	80.0	400	95 (1.00 min)	390	920
4M	Unfractionated	81.5	455	95 (4.00 min)	460	<1000
	F-1	85.0	520	95 (4.00 min)	440	890
	C-2	78.5	355	95 (1.00 min)	340	930
	F-2	81.0	480	95 (6.00 min)	480	950
2B	Unfractionated	84.5	360	95 (2.50 min)	360	860
	F-1	87.0	480	95 (1.25 min)	330	770
	C-2	81.0	380	95 (5.00 min)	380	910
	F-2	80.5	460	95 (3.00 min)	450	970
5B	Unfractionated	83.5	375	95 (1.50 min)	340	840
	F-1[b]	...	...	...	...	...
	C-2	81.5	380	95 (1.00 min)	360	970
	F-2[b]	...	...	...	...	...
T	Unfractionated	84.0	400	95 (1.00 min)	395	940
	F-1	85.0	530	95 (1.00 min)	310	635
	C-2	84.0	390	95 (1.50 min)	380	910
	F-2	81.5	460	95 (2.50 min)	440	920

[a] Time in parentheses is the number of min required to reach the peak height after the temperature first reached 95° C.

[b] Insufficient amount of sample.

present in this fraction. It has been reported (24) that small starch granules gelatinize consistently within higher ranges than large granules. This was attributed to the higher degree of crystalline order in the small granules, contributing to the different physical structure and chemical composition.

Although it was not possible to detect any definite trend in peak height among the three cultivars analyzed, it was generally noted that the peak height was highest for the high-protein-containing fraction (Fig. 2). The starches derived from the F-1 fractions showed a larger drop in consistency at 95° C, as indicated by the difference between peak height and 15-min height. This drop can be attributed to lower stability of starch derived from F-1. Setback values were lower for F-1 starches within a particular stream, which would indicate a lower tendency to retrograde.

Water-Binding Capacity

Water-binding capacity values of isolated starch from selected flour streams are shown in Table III. Waldron and Red River 68 showed higher water-binding capacity values than Pitic 62, and in particular for the F-1 fractions. This was expected because of the greater starch damage associated with hard-type wheat cultivars during milling. The higher water-binding capacity values obtained for F-1 may be attributed not only to starch damage content of this particular fraction but also to granule size.

Starch Damage

Starch damage data on the flour and corresponding starches for five selected streams are shown in Table IV for Waldron, Red River 68, and Pitic 62,

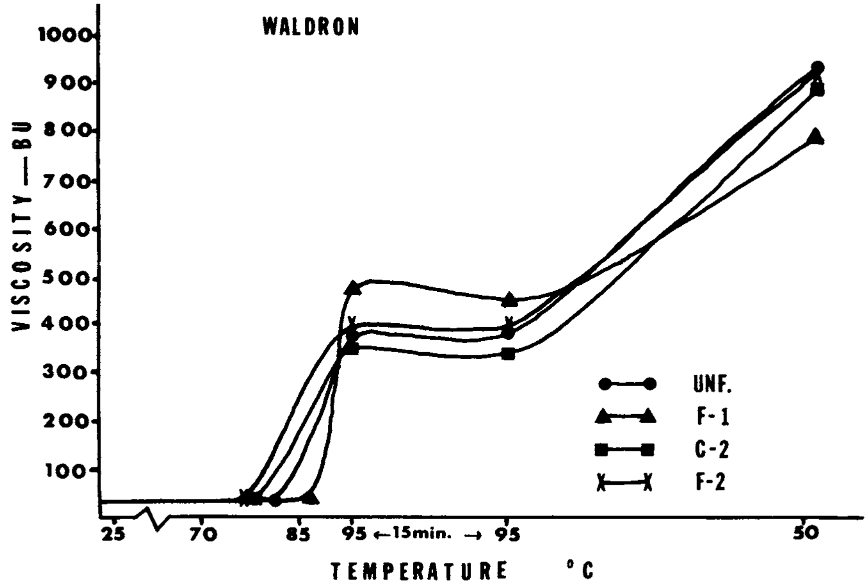

Fig. 2. Starch pasting properties of starches isolated from unfractionated and pin milled and air-classified flour.

respectively.

Differences in starch damage among the flour streams appear to be directly related to the order of mill streams, type of rolls, and roll pressure. The middling flour streams received the greatest amount of roll pressure because of roll type (fine corrugated or smooth rolls), and the break streams received the least pressure during the milling process because of coarse corrugated rolls, thus, the higher starch damage values for the middling flour streams. The F-1 and F-2 fractions for Waldron and Red River 68 showed the highest amount of starch damage. The much higher values obtained for these two fractions, compared to the corresponding unfractionated flour, is due undoubtedly to the effects of pin milling. Starch damage values for the unfractionated flour and flour fractions obtained from Pitic 62 were lower than those for Waldron and Red River 68. Due to its softer nature, it is less susceptible to damage, as previously indicated.

The isolated starches showed somewhat different results. The F-2 fraction of Red River 68 had the highest starch damage values, followed by the C-2 fraction for all five flour streams. The C-2 fractions containing the highest starch damage were derived from the middling flour streams and the tailings (T) stream. The F-1

TABLE III
Water-Binding Capacity of Isolated Starches from the Unfractionated and Pin Milled and Air-Classified Flour Fractions of Three HRS Wheat Cultivars[a]

Flour Stream	Flour Fraction	Water-Binding Capacity		
		Waldron %	Red River 68 %	Pitic 62 %
2M	Unfractionated	89	86	79
	F-1	103	96	84
	C-2	85	84	86
	F-2	93	94	88
4M	Unfractionated	89	84	82
	F-1	106	96	85
	C-2	88	92	87
	F-2	90	97	85
2B	Unfractionated	82	82	83
	F-1	94	96	86
	C-2	82	84	83
	F-2	87	99	74
5B	Unfractionated	82	85	86
	F-1	103	99	92
	C-2	90	92	88
	F-2	84	100	85
T	Unfractionated	83	91	80
	F-1	104	100	97
	C-2	93	86	89
	F-2	83	96	93

[a] Values reported are an average of two determinations expressed on a dry basis.

TABLE IV
Starch Damage on Unfractionated Flour and Pin Milled and Air-Classified Flour Fractions and Corresponding Isolated Starches[a]

Flour Stream	Flour Fraction	Starch Damage (F.E.U.)					
		Flour			Starch		
		A[b]	B[b]	C[b]	A[b]	B[b]	C[b]
2M	Unfractionated	39.2	31.5	14.4	9.2	7.8	3.4
	F-1	114.2	94.5	18.0	15.6	7.6	7.3
	C-2	31.0	33.9	17.4	10.6	15.3	3.7
	F-2	85.8	97.1	19.3	15.1	23.6	2.7
4M	Unfractionated	44.1	40.9	15.8	10.5	10.4	2.5
	F-1	106.8	88.9	16.7	16.9	10.3	11.0
	C-2	38.8	38.9	20.9	12.2	21.8	3.2
	F-2	89.7	103.7	28.8	8.3	25.1	3.0
2B	Unfractionated	18.6	18.8	8.2	5.4	8.0	2.7
	F-1	62.2	54.1	11.6	8.2	8.7	10.1
	C-2	24.8	24.4	9.7	5.3	9.3	0.9
	F-2	56.2	54.5	15.5	10.5	17.6	3.3
5B	Unfractionated	12.3	13.5	13.6	4.9	4.2	3.7
	F-1	46.6	31.3	17.1	10.4	5.9	7.7
	C-2	19.4	22.5	15.3	6.1	8.0	3.4
	F-2	39.4	42.4	18.4	6.5	11.6	6.7
T	Unfractionated	38.2	37.2	17.4	10.3	8.9	...
	F-1	71.2	61.4	16.7	8.5	7.8	9.0
	C-2	44.0	45.0	20.9	18.4	11.1	1.6
	F-2	88.8	92.9	23.5	9.0	22.6	4.0

[a] Values reported are an average of two determinations with results expressed as Farrand Equivalent Units (F.E.U.) on a dry basis.

[b] Data obtained for the cultivars Waldron, Red River 68, and Pitic 62, respectively.

TABLE V
Total Lipid Extracted from the Unfractionated Flour and Pin Milled and Air-Classified Flour Fractions of Two HRS Wheat Cultivars[a]

Flour Stream	Flour Fraction	Flour		Starch	
		Waldron %	Red River 68 %	Waldron %	Red River 68 %
2M	Unfractionated	1.6	2.0	0.48	0.67
	F-1	2.3	3.5	0.78	0.91
	C-2	1.3	2.0	0.53	0.82
	F-2	1.7	1.8	0.54	0.66
2B	Unfractionated	1.3	2.2	0.73	0.81
	F-1	2.5	2.5	1.17	1.09
	C-2	1.7	1.3	0.68	0.88
	F-2	1.4	1.3	0.74	0.90

[a] Values reported are an average of two or more determinations expressed on a dry basis.

fraction of Pitic 62, the softer-type wheat variety, had the highest starch damage values when compared to the other fractions obtained from this variety.

Total Lipid Content

Lipid values obtained for the unfractionated and pin milled and air-classified flour fractions of Waldron and Red River 68 for a middling and a break flour stream and for the corresponding isolated starches are shown in Table V.

The high-protein, fine-fraction flour (F-1) showed the highest lipid values for both the middling and the break flour streams and corresponding isolated starches. In general, the variety Red River 68 showed higher values than Waldron for both the flour and starch.

The higher lipid content present in the starch isolated from the F-1 flour fractions would agree with results reported by Kulp (24), who found more than twice the amount of lipid present in small granules than in large ones. Although differences were noted in the pasting properties of the starches isolated from the different flour fractions (Table II), suggesting a possible role for the lipid, Kulp

TABLE VI
Yield and Protein Content of Water-Solubles Extracted from the Unfractionated Flour and Pin Milled and Air-Classified Flour Fractions of Three HRS Wheat Cultivars[a]

Flour Stream	Flour Fraction	Total Water-Solubles			Protein Content			Protein-Free Water-Solubles		
		A[b] %	B[b] %	C[b] %	A[b] %	B[b] %	C[b] %	A[b] %	B[b] %	C[b] %
2M	Unfractionated	3.5	4.0	3.1	32.3	34.2	30.7	2.5	2.6	2.1
	F-1	5.5	6.1	4.2	21.1	20.7	36.2	4.4	4.8	2.7
	C-2	4.2	4.1	2.9	43.9	39.6	28.2	2.3	2.5	2.1
	F-2	3.4	4.5	2.8	27.4	22.5	23.9	2.5	3.5	2.1
4M	Unfractionated	4.0	4.7	4.6	30.5	30.0	29.0	2.8	3.3	3.3
	F-1	5.2	6.3	4.1	19.8	20.7	31.9	4.2	5.0	2.8
	C-2	3.9	4.1	3.2	41.4	36.6	24.1	2.3	2.6	2.5
	F-2	3.9	4.7	3.3	24.3	22.2	22.4	2.9	3.6	2.6
2B	Unfractionated	3.2	3.8	2.9	33.6	30.6	27.8	2.2	2.6	2.1
	F-1	5.1	5.8	3.6	23.9	22.7	34.3	3.9	4.5	2.4
	C-2	3.6	3.6	1.8	38.0	32.4	19.4	2.2	2.4	1.5
	F-2	3.7	4.0	2.5	26.1	24.3	20.8	2.7	3.0	2.0
5B	Unfractionated	4.6	4.1	5.1	37.0	33.2	24.5	2.9	3.2	3.8
	F-1	7.6	5.7	5.0	27.3	26.7	28.1	5.5	4.2	3.6
	C-2	4.2	4.2	2.9	38.3	35.1	23.9	2.6	2.7	2.2
	F-2	4.8	4.9	3.6	30.9	27.7	21.5	3.4	3.5	2.8
T	Unfractionated	4.2	4.2	7.2	30.0	26.9	25.0	2.9	3.1	5.4
	F-1	4.7	6.2	7.7	22.3	23.0	27.6	3.7	4.8	5.6
	C-2	4.6	5.3	5.8	30.1	30.5	26.0	3.2	3.7	4.3
	F-2	4.7	5.4	6.0	21.9	23.0	25.1	3.7	4.1	4.5

[a] Results expressed on a dry basis.

[b] Data obtained for the cultivars Waldron, Red River 68, and Pitic 62, respectively.

(24) reported that the basic differences in pasting patterns between small and large granules were retained with removal of the lipids. Although the per cent lipid is higher in the small granule starch, it is possible that the amount of lipid per granule is constant.

Total Water-Solubles

Yield of total water-solubles, protein content, and yield of protein-free water-soluble material extracted from the cultivars Waldron, Red River 68, and Pitic 62 for the unfractionated and pin milled and air-classified flour fractions of selected streams are shown in Table VI. For all three cultivars, the greatest amount of total and protein-free water-solubles was extracted from the F-1 fraction. The greater amount of protein-free water-solubles in F-1 would indicate the presence of more soluble carbohydrate material as a result of the pin milling and air-classification process. Such an increase in carbohydrate material could be due to: 1) more soluble starch resulting from more starch damage in this fraction, 2) more water-soluble pentosan material, and 3) more sugars present. Pitic 62, when compared to Waldron and Red River 68, showed the least amount of protein-free water-soluble material in almost all streams and fractions, except for the tailings stream which was higher than the other two cultivars. Low and high protein levels were present in the water-solubles extracted from the F-1 and

TABLE VII
Yield of Crude and Amylase-Treated Pentosans and Protein Content Obtained from Selected Streams of Unfractionated Flour and Pin Milled and Air-Classified Flour Fractions

Variety	Flour Stream	Flour Fraction	Crude Pentosans[a] %	Amylase-Treated Pentosans[a] %	Protein Content[b] %	Amylase-Treated Pentosans[c] %
Waldron	1M	Unfractionated	1.05	0.63	26.2	0.47
		F-1	1.75	1.08	19.0	0.87
		C-2	1.50	0.67	38.6	0.41
		F-2	1.32	0.50	25.2	0.37
Waldron	T	Unfractionated	0.88	0.56	11.5	0.49
		F-1	1.32	0.79	8.2	0.73
		C-2	1.34	0.70	27.0	0.51
		F-2	1.35	0.75	18.7	0.61
Red River 68	1M	Unfractionated	0.75	0.44	28.8	0.32
		F-1	1.50	0.88	12.1	0.77
		C-2	1.12	0.63	32.1	0.43
		F-2	0.89	0.41	19.6	0.33
Red River 68	T	Unfractionated	0.99	0.66	14.2	0.56
		F-1	0.90	0.61	6.0	0.57
		C-2	1.09	0.60	19.1	0.49
		F-2	0.82	0.44	11.3	0.39

[a] Yield from flour.
[b] Results expressed on a dry basis.
[c] Yield (protein free) from flour.

C-2 fractions, respectively, for Waldron and Red River 68. However, the flour protein content of F-1 and C-2 was highest and intermediate, respectively, of the three-pin milled and air-classified fractions. These data would indicate that the higher protein content in the F-1 flour fractions is due to larger amounts of the gluten proteins, while the intermediate protein-containing flours have greater proportions of the water-soluble proteins. With Pitic 62, the water-soluble material extracted from F-1 flour fraction contained the highest protein content of the three fractions. Such results are attributed to the extremely weak gluten properties of Pitic 62.

Crude and Amylase-Treated Water-Soluble Pentosans

Yields of crude and amylase-treated water-soluble pentosans extracted from the cultivars Waldron and Red River 68 for two streams of pin milled and air-classified flour fractions are presented in Table VII. Highest yield of amylase-treated pentosans (protein-free) from the flour was obtained in the high-protein fraction (F-1). The amount obtained with both cultivars from the middling and break flour streams was approximately twice that recovered from the corresponding unfractionated flours and from C-2 and F-2. The higher amount

TABLE VIII
Total Pentosan in the Unfractionated and Fractionated Flours for Selected Streams from Three HRS Wheat Cultivars[a]

Flour Stream	Flour Fraction	Pentosan Content Waldron %	Red River 68 %	Pitic 62 %
1M	Unfractionated	1.5	1.6	1.6
	F-1	3.2	2.8	1.2
	C-2	1.6	1.4	1.4
	F-2	1.2	1.2	1.2
4M	Unfractionated	1.8	1.5	2.6
	F-1	3.7	3.0	1.8
	C-2	1.5	1.4	3.0
	F-2	1.3	1.2	2.5
2B	Unfractionated	1.6	1.5	1.5
	F-1	3.6	2.9	1.7
	C-2	1.3	1.4	1.6
	F-2	1.3	1.2	1.2
5B	Unfractionated	2.4	2.0	2.5
	F-1	3.9	3.9	2.6
	C-2	1.5	1.5	2.0
	F-2	1.7	1.8	2.1
T	Unfractionated	2.0	1.8	3.9
	F-1	3.9	4.0	2.8
	C-2	1.7	1.5	4.1
	F-2	1.7	1.7	2.5

[a] Values reported are an average of two determinations expressed on a dry basis.

of pentosan material found in F-1 would explain, in part, the higher yield of total water-solubles in this fraction (Table VI). The yield of amylase-treated protein-free pentosans extracted from F-1 of Pitic 62 (not shown in this Table) was not as high as with the other two cultivars, which agreed with the results obtained for total water-solubles (Table VI). A comparison of the yield of crude pentosans obtained from the three cultivars did not reveal the differences noted with the amylase-treated protein-free pentosans. This result was due primarily to the differences in protein content of the pentosan preparations. The average protein content of the crude pentosans extracted from F-1 of Waldron, Red River 68, and Pitic 62 for five streams was 17.4, 14.9, and 31.7%, respectively, whereas the corresponding amylase-treated pentosans gave protein contents of 12.6, 10.3, and 23.9%, respectively.

Total Pentosans

Total pentosans content for the unfractionated and fractionated flours for selected streams from three cultivars is shown in Table VIII. Values reported using the colorimetric procedure (22,23) have been shown to be consistently lower than those obtained using the AACC method (15) which is based on digestion of the sample with 12% HCl followed by distillation and measurement of furfural (15). However, relative differences between the various flours were demonstrated. Duplicate values obtained using the colorimetric procedure on a sample agreed within 0.1 to 0.2%. As expected from the recoveries obtained for

TABLE IX
Ratio of Component Sugars, Intrinsic Viscosity Values, and Protein Content of DEAE-Cellulose Fraction F_1 of α-Amylase-Treated Water-Soluble Pentosans

Variety	Flour Stream	Flour Fraction	Ratio of Component Sugars ARAB:XYL	Intrinsic Viscosity n	Protein Content[a] %
Waldron	1M	Unfractionated	1:1.67	2.8	1.9
		F-1	1:1.63	2.5	0.9
		C-2	1:1.69	2.5	...
		F-2	1:1.64	2.4	1.4
Waldron	T	Unfractionated	1:1.66	1.6	0.4
		F-1	1:1.70	2.5	0.8
		C-2	1:1.65	3.0	1.4
		F-2	1:1.73	2.5	0.6
Red River 68	1M	Unfractionated	...	...	...
		F-1	1:1.38	3.1	1.3
		C-2	1:1.33	2.7	5.3
		F-2	...	...	...
Red River 68	T	Unfractionated	...	...	...
		F-1	1:1.53	2.8	3.4
		C-2	1:1.52	3.4	4.4
		F-2	...	...	...

[a] Results expressed on a dry basis.

the amylase-treated pentosans (Table VII), F-1 contained the highest amount of total pentosans. Waldron and Red River 68 contained higher amounts for this fraction than Pitic 62. In general, F-2 contained the lowest pentosan content for all three cultivars, while C-2 showed values similar to the unfractionated flour except for flour stream 5B.

DEAE-Cellulose Fractionation

The ratio of component sugars, intrinsic viscosity values, and protein content for DEAE-cellulose fraction 1 of the α-amylase-treated pentosans isolated from the unfractionated flour and pin milled and air-classified flour fractions from two streams of Waldron and Red River 68 are presented in Table IX.

Fraction 1 is generally considered a pure pentosan fraction, containing small amounts of protein and only arabinose and xylose as component sugars after acid hydrolysis (20). In general, the ratio of component sugars in fraction 1 was similar for the unfractionated and the various pin milled and air-classified flour fractions within each cultivar. This was true for both flour streams investigated. However, the ratio of arabinose:xylose for pentosans isolated from Red River 68 was lower than the ratio for the sugar components in the pentosans isolated from Waldron. A lower ratio of arabinose:xylose would indicate a greater degree of branching.

Intrinsic viscosity values (Table IX) revealed no definite trends; however, the values for the F-1 high-protein fraction and C-2 intermediate-protein fraction were slightly higher for both streams examined for cultivar Red River 68 as compared to the corresponding flour fractions of Waldron.

The protein content of the DEAE-cellulose fraction 1 (Table IX) showed a pattern similar to that observed with the α-amylase-treated water-soluble pentosans (Table VII). Such results are most probably due to the different type of protein present in the different pin milled and air-classified flour fractions. The amount of water-soluble protein associated with the high-protein flour fractions is less than that present in the other flour fractions.

This study has shown that certain differences exist in the starch and pentosans among pin milled and air-classified flour streams from different cultivars of hard red spring wheat. Specifically, the differences were in the high-protein-containing flour fractions, regardless of cultivar or flour stream. Starch isolated from this fraction had the highest pasting temperature, highest peak height, and lowest setback values when compared to starch isolated from the other flour fractions. This starch also contained the highest amount of lipid material, had the highest water-binding capacity values, and the greatest percentage of small granules. The high-protein-containing flour fraction also contained the highest amount of pentosan material, although the ratio of arabinose:xylose in an essentially pure pentosan isolated from the different flour fractions was similar, as was the intrinsic viscosity. Such data could provide useful information in preparing custom flours for specific end products.

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