Starch Purification After Pin Milling and Air Classification of Waxy, Normal, and High Amylose Barleys

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ABSTRACT

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Three barley cultivars with low (SB89528), normal (Condor), and high (Glacier) amylose starch contents were pin-milled and air-classified into coarse and fine fractions by two passes at a vane setting (particle-size cut) of 15 μ m and a third pass at 30 μ m. The coarse fractions from the final pass (C3) had 15-36% starch, 12-15% protein, and 13-24% β -glucan, whereas the fine fractions (F3) had 77-78% starch, 8-9% protein, and 6-8% β -glucan. The shifts in starch, protein, and β -glucan varied among the cultivars. Scanning electron microscopy of the pin-milled barleys and their air-classified fractions showed that the first two air-

classifications separated small granule starch into fine 1 (F1) and fine 2 (F2) fractions; the large granule starch was separated into coarse 1 (C1) and coarse 2 (C2) fractions. The third pass yielded a fine fraction (F3) rich in large granule starch. Further purification of F3 fractions by a wet process yielded almost pure large granule starch with an extraction efficiency $\sim 30\%$ higher than that obtained by conventional laboratory starch extraction from barley. Furthermore, the process yielded protein and β -glucan as by-products at high levels of purity.

On average, barley contains 64% starch, 11% protein, and 5% β -glucan; the remaining 20% includes moisture, fiber, and other minor components (MacGregor and Fincher 1993). Numerous studies have suggested that barley β -glucan has potential applications in the pharmaceutical and health food industries due to its ability to reduce serum cholesterol in hypercholesterolemic subjects (Fadel et al 1987, Klopfenstein and Hoseney 1987, Newman et al 1989, Ranhotra et al 1991). Barley cultivars containing <2% (waxy), ~25%, or ~40% amylose starches are available (MacGregor and Fincher 1993). Furthermore, barley starch exists in two clearly defined populations of large and small granules, and these granules differ in their physicochemical properties (Bathgate and Palmer 1972). Morrison and Scott (1986) reported that barley with high amylose starch contains more small starch granules (29.2-46.3 wt%) than barleys with waxy (3.5-13.6%) or normal (4.6-15.1%) starch.

Air classification of a flour, obtained through pin milling or fine grinding of seeds, separates and concentrates seed constituents into flour fractions differing in particle size and composition (Youngs 1975). Pin milling disintegrates seeds into fine particles, and air classification separates them on the basis of differences in density, mass, and projected area in the direction of flow. Starch and protein concentrates have thus been separated from cereal and legumes (Tyler et al 1981, Sosulski 1989, Sosulski and Nowakowski 1989). Pin milling and air classification, as a dry process, is relatively energy efficient and requires lower capital costs than does the conventional wet milling and separation process. However, the advantages of pin milling and air classification are partially offset by relative impurities of the starch and protein fractions, which may present difficulties in utilization.

While several studies on air classification of cereals have been conducted on wheat (Gracza 1959; Bean et al 1969a,b; Dick et al 1979; Nowakowski et al 1986; Sosulski et al 1988), only a few studies have been conducted on pin milling and air classification of barley. Pomeranz et al (1971) air-classified roller-milled barley into five fractions. The protein-rich fine fraction represented over 5% of the meal and, on average, contained about 15% more protein than that of the original meal. The low protein fractions were obtained in ~55% yield and contained ~4% less protein than that of the original meal. Vose and Youngs (1978) produced coarse and fine fractions by air classification of pinmilled waxy (CI4382) and normal (Betzes) barley. The yield of

the coarse fraction obtained from normal barley was ~65%, compared to 14% for the fine fraction. Recently, Wu et al (1994) pin-milled three cultivars of barleys containing different protein and β -glucan levels, and air-classified them into fractions with different particle-size ranges. In the three cultivars, the protein was concentrated in fractions with particle sizes <15 μ m; starch was concentrated in fractions with particle sizes <18 μ m, 15-30 μ m, and >30 μ m. In one barley cultivar, β -glucan was concentrated in fractions with particle sizes >18 μ m; and in two other cultivars, β -glucan was concentrated in fractions with particle sizes >30 μ m. Studies were also conducted by Pomeranz et al (1971) and Vose and Youngs (1978) on air classification of malted barley to produce special flours with various compositions and amylolytic activities.

The variability in amylose content and granule size in barley starch offers potential industrial and food uses. Therefore, separation and concentration of barley grains into its major components warrants investigation. The present study was conducted to determine the mass balance and shifts of protein, starch, and β -glucan in pin-milled and air-classified waxy, regular, and high amylose barleys. The objective was twofold: 1) to obtain a fraction rich in large granule starch; and 2) to compare the efficiencies of starch extractions from the large granule starch-rich fraction with a conventional starch extraction procedure.

MATERIALS AND METHODS

Materials

Barleys containing normal (Condor), waxy (SB89528), and high amylose (Glacier) starch were used in the study. Condor was a two-rowed, hull-less, registered Canadian cultivar obtained from J. Helm, Alberta Agriculture (Lacomb). SB89528 was two-rowed, hull-less genotype obtained from B. G. Rossnagel, Crop Development Centre, University of Saskatchewan (Saskatoon). Glacier was a six-rowed, hulled genotype obtained from R. K. Newman, Montana State University, (Bozeman). Glacier barley was dehulled in a stone mill at 2,500 rpm for 5 min. The separated hulls were removed with a mechanical air blower attached to the mill. The hull yield was ~22% higher than the 10-12% present in normal, hulled barleys. Subsamples of the barleys were ground in a Udy cyclone mill to pass a $0.5-\mu m$ screen and used for chemical analyses and starch extraction.

Grain Hardness

Barley grain hardness was determined with a Brabender microhardness tester (C. W. Brabender, Inc., South Hackensack, NJ) that automatically records the time required to mill 4.5 g of grain.

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Pin Milling and Air Classification

Pin milling and air classification of barleys were performed according to the protocol given in Figure 1 at the POS Pilot Plant Corporation, Saskatoon. The grain (~20 kg) was pin-milled at a feed rate of 150 kg/hr in a Contraplex wide-chamber A250 mill (Alpine Corp., Augsburg, Germany) with counter-rotating disks operating at 5,600 rpm (door side) and 11,200 rpm (house side). A single pass was sufficient to mill Condor barley; two passes were required to mill SB89528 and Glacier barleys.

The pin-milled barley was fractionated into coarse and fine fractions (C1 and F1) using a 132 Mikroplex spiral air classifier (Alpine) at a feed rate of 20 kg/hr, a rotor speed of 11,000 rpm, and a vane setting (cut-off size) of 15 μ m. The C1 fraction was again pin-milled and air-classified into C2 and F2 fractions as described above. The C2 fraction was again air-classified into C3 and F3 fractions at a vane setting of 30 μ m, while other air-classifier operating parameters were kept as before.

Yields and compositions of fractions were reported on a dryweight basis. The protein shift was calculated according to Gracza (1959) as:

Protein shift =
$$[\Sigma(Y_{PEF} \times P_{PEF}) - (\Sigma Y_{PEF} \times P_{B})] + [(\Sigma Y_{PDF} \times P_{B})] + [(\Sigma Y_{PDF} \times P_{DEF})] / P_{B}$$

where $Y_{\rm PEF}$ and $Y_{\rm PDF}$ are yields (%) of protein-enriched and protein-depleted air-classified fractions, respectively; $P_{\rm B}$, protein content (%) of pin-milled barley; $P_{\rm PEF}$ and $P_{\rm PDF}$ are protein contents (%) of protein-enriched and protein-depleted air-classified fractions, respectively. Similar calculations were made to obtain starch and β -glucan shifts.

The F3 fraction from pin milling and air classification contained starch, protein, and β -glucan. The starch in this fraction was further purified by a wet-extraction procedure (Fig. 2). The final products were large granule starch, protein, and β -glucan.

Starch Extraction

Starch was extracted from barleys using a conventional laboratory procedure. The ground barley (50 g) was homogenized with distilled water (500 ml) in a blender for 5 min. The slurry was passed through a cheesecloth (additional water was used to wash the residue) and then through a 53- μ m polypropylene screen. The filtrate was adjusted to 0.05N NaOH, stirred for 30 min, and centrifuged at $5,500 \times g$ for 15 min. The starch residue was

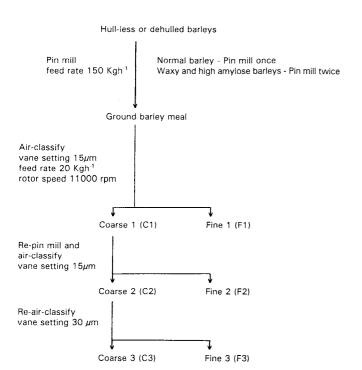


Fig. 1. Procedure used for pin milling and air classification of barleys.

washed twice with 0.05N NaOH, neutralized, and recovered by filtration. The filter cake was washed with distilled water, then washed with 80% ethanol and air-dried.

Analyses

Standard methods (AACC 1983) were used to determine moisture, total nitrogen, and starch damage. Starch was determined using the method of Holm et al (1986) after boiling with 80% ethanol for 5 min. β -Glucan was determined by the method of McCleary and Glennie-Holmes (1985), using an assay kit from MegaZyme (North Rocks, Sydney, Australia). True amylose content of the starches was determined by the method of Chrastil (1987), after refluxing with a mixture of n-propanol and water (3:1, v/v) for 7 hr.

Scanning Electron Microscopy

The pin-milled barleys and their air-classified fractions were individually sprinkled on double-sided adhesive tapes mounted on circular aluminum stubs, coated with gold, and examined in a scanning electron microscope (Phillips SEM 505) at an accelerating potential of 20 kV.

Starch Granule Size Analyses

Mounts for light microscopy of the starch samples were prepared from a dilute slurry of a starch and ethanol mixture (95%, w/v). Images of the starch granules from the light microscope (Leitz Laborlux K and D, Wetzlar, Germany) at 63× magnification were analyzed for particle-size distribution on an image analyzer (BioQuant System IV, Image Technology, New York) equipped with an image acquisition and processing station. In each measurement, an image of an optical micrometer was introduced for size calibration.

Granules (2,000) from each starch sample were measured for diameter (the diameter of a circle of equal area to the particle). The percentage of granules occurring within predetermined diameter ranges were obtained.

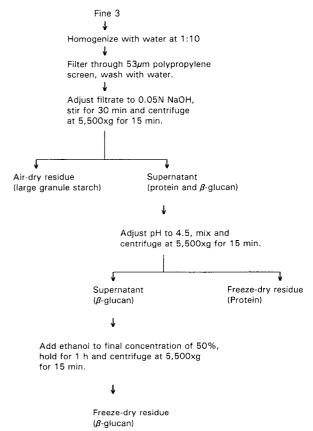


Fig. 2. Wet-extraction procedure for further purification of fine 3 fractions obtained from pin milling and air classification.

RESULTS AND DISCUSSION

Pin Milling

Barleys were pin-milled into meals using identical equipment before air classification (Fig. 1). After the first pin milling in Condor barley, 78% of the meal passed through a $53-\mu m$ screen (270, Tyler). In SB89528 and Glacier barleys, the corresponding values were 71 and 70%, respectively; therefore, they were somewhat coarser than the Condor meal. Particle size affects the separation of meal constituents in an air classifier. Vose and Young (1978) suggested that barley should be finely pin-milled (\sim 80% of the meal passing through a $53-\mu m$ screen) to obtain a successful air classification. Thus, SB89528 and Glacier meals needed additional pin milling, after which, 77% of the particles in SB89528 and 79% in Glacier meals passed through a $53-\mu m$ screen.

Degree of particle-size reduction in wheat flours caused by roller or pin milling gives an indication of the friability of wheat endosperm; softer endosperm is more difficult for milling (Donelson and Yamazaki 1972, Pratt 1978). Such information is equally applicable to barley milling. Table I shows that SB89528 and Glacier barleys had longer grind time (50 and 36 sec, respectively), and thus had softer endosperm than that of Condor barley (grind time, 27 sec). This is probably the reason that a second pin milling was needed to reduce the particle size to $<53~\mu m$ in these two barleys.

Milling of grains causes starch damage and thereby alters the physicochemical properties of starch (Tipples and Kilborn 1968, Nowakowski et al 1986). The increased susceptibility of damaged starch granules to α -amylolysis is a measure of starch damage. The percentage of damaged starch in F1 (high in small granule starch) and F3 (high in large granule starch) fractions is given in Table I. The starch damage in both the fractions ranged between 3.5 and 6.0%. Among the three barleys, SB89528 showed the highest starch damage, followed by Glacier and Condor. However, marginal differences in starch damage were observed between F1 and F3 fractions from the same barley cultivars. The damaged starch contents of barley fractions (Table I) are considerably lower than the >20% reported in pin-milled wheat (Nowakowski et al 1986), but comparable to that of pin-milled legumes (0.5-2%) (Tyler 1982).

Air Classification

The starch, protein, and β -glucan contents of the barleys are given in Table II, in addition to data on the yields and compositions of the air-classified fractions. The three barleys contained

12.3-13.6% protein, 55.3-62.0% starch, and 5.9-7.8% β -glucan. Condor had the highest starch concentration (62%), but its β -glucan content, as expected, was lower (5.9%) when compared to that of the other two barleys (7.2-7.8%). Low and high amylose barleys contain more β -glucan than normal amylose barley (R. S. Bhatty, *unpublished data*).

On the first pass, the yields of C1 and F1 fractions were: 85 and 9% (SB89528); 87 and 7% (Condor); and 75 and 23% (Glacier); respectively. Thus, there was a lower separation of the C1 and F1 fractions in Glacier barley, despite its somewhat softer endosperm as compared to Condor (Table I). Glacier's particle size ($<53 \mu m$) was generally similar to that of SB89528 and Condor. Starch granule size may have influenced the separation of the two fractions. Morrison and Scott (1986) reported that high amylose barley contains more small starch granules (29.2-46.3%) than do waxy (3.5-13.6%) or normal amylose (4.6-15.1%) barleys. Scanning electron micrographs of the coarse and fine barley fractions obtained in the present study (Fig. 3) showed that air classification concentrated most of the small starch granules into F1 and F2 fractions. Thus, the higher yield of Glacier F1 (23.8%) may be due to a higher content of small starch granules as compared to the F1 fractions of SB89528 and Condor.

In all the barleys, compositions of C1 and F1 fractions were different from those of the ground barley meals; in each case, C1 had lower protein but higher starch and β -glucan contents, and F1 had higher protein but lower starch and β -glucan contents. The starch contents of C1 and F1 were: 59.1 and 27.4% (SB89528); 70.5 and 27.2% (Condor); and 61.8 and 35.5% (Glacier); respectively. The starch enrichment (increase in concentration) in C1 compared to that of ground barley was: Condor (13.7%) > Glacier (11.8%) > SB89528 (10.3%). The starch content of Glacier F1 may be highest because air classification concentrated small starch granules into the F1 fraction. The protein contents of C1 and F1 were: 11.6 and 27.7% (SB89528); 11.5 and 29.5% (Condor); and 9.8 and 17.5% (Glacier), respectively. The protein enrichment in F1 fraction, compared to that of ground barley, was: Condor (120%) > SB89528 (104%) > Glacier (42%). The lower protein enrichment in Glacier F1 is again due to its higher starch content. The β -glucan contents of C1 and F1 were: 8.0 and 1.9% (SB89528); 6.1 and 1.7% (Condor); and 9.3 and 1.8% (Glacier); respectively. β -Glucan is largely a component of endosperm cell walls. This study suggested that most of the β -glucan in the ground barleys appeared with the coarse fractions. The first pass through the air classifier caused only a marginal enrichment of β -glucan in the C1 fraction, which ranged from 3% in

TABLE I

Barley Grain Hardness and Starch Damage of Fine 1 and Fine 2 Fractions^a

				Starch Damage, %c		
Cultivar	Starch Type	True Amylose, %b	Grain Hardness, sec	Fine 1	Fine 3	
SB89528	Waxy	2.1 ± 0.9	49.6 ± 2.1 (softest)	5.7 ± 0.0	5.8 ± 0.0	
Condor	Normal	32.6 ± 0.8	27.0 ± 2.8 (hardest)	4.3 ± 0.1	3.8 ± 0.0	
Glacier	High amylose	45.2 ± 1.5	36.0 ± 1.4	5.5 ± 0.0	4.9 ± 0.1	

^a Means of three determinations ± standard deviation.

TABLE II
Yields and Compositions (%) of Pin-Milled and Air-Classified Barleys^a

	Waxy Starch (SB89528)			Normal Starch (Condor)			High Amylose Starch (Glacier)					
	Yield	Starch	Protein	β -Glucan	Yield	Starch	Protein	β -Glucan	Yield	Starch	Protein	β -Glucan
Ground barley	100.0	53.6 ± 0.7	13.6 ± 0.3	7.2 ± 0.2	100.0	62.0 ± 0.9	13.4 ± 0.6	5.9 ± 0.3	100.0	55.3 ± 2.7	12.3 ± 0.5	7.8 ± 0.5
Fine 1 (F1)	9.1	27.4 ± 0.9	27.7 ± 0.1	1.9 ± 0.2	6.8	27.2 ± 1.0	29.5 ± 0.4	1.7 ± 0.1	22.8	35.5 ± 1.3	17.5 ± 0.9	1.8 ± 0.5
Coarse 1 (C1)	85.2	59.1 ± 0.8	11.6 ± 0.4	8.0 ± 0.0	87.0	70.5 ± 1.4	11.5 ± 0.0	6.1 ± 0.2	75.1	61.8 ± 1.7	9.8 ± 0.8	9.3 ± 0.1
Fine 2 (F2)	29.4	35.7 ± 1.7	14.9 ± 0.5	3.5 ± 0.1	7.9	29.2 ± 0.6	26.7 ± 0.1	4.3 ± 0.1	6.1	24.2 ± 1.6	18.6 ± 1.1	2.2 ± 0.1
Coarse 2 (C2)	54.2	71.7 ± 1.4	10.5 ± 0.1	11.0 ± 0.7	75.0	74.9 ± 0.8	8.9 ± 0.1	7.0 ± 0.3	64.6	69.6 ± 1.9	8.9 ± 0.8	10.5 ± 0.1
Fine 3 (F3)	46.1	77.1 ± 0.5	9.2 ± 0.2	8.1 ± 0.4	63.4	78.0 ± 1.1	8.3 ± 0.1	6.5 ± 0.6	43.7	78.1 ± 1.6	8.0 ± 0.6	6.7 ± 0.8
Coarse 3 (C3)	7.6	15.6 ± 0.3	13.7 ± 0.2	23.8 ± 0.1	10.4	32.7 ± 1.7	15.1 ± 0.0	13.1 ± 0.3	20.9	36.4 ± 2.8	12.2 ± 0.0	21.8 ± 1.9

^a Means of three determinations ± standard deviation (except yield), given on dry weight basis.

^bDetermined after defatting starches with propanol-water (3:1, v/v) mixture.

^c Fine 1 and fine 3 fractions contain small and large granule starch, respectively.

SB89528 to 19% in Glacier barley.

The C1 fractions were pin-milled again before the second pass through the air classifier. On the second pass, the yields (as percent of original meal) of C2 and F2 were: 54 and 29% (SB89528); 75 and 8% (Condor); 65 and 6% (Glacier); respectively (Table II). The F2 yield in SB89528 (29%) was considerably higher because its separation was lower than that of Condor and Glacier barleys where the yields were only 6-8%. The lower C2 yield obtained in SB89528 is difficult to explain because all three barleys were air-classified under similar conditions.

In all the barleys, the composition of C2 and F2 were different from that of their original C1 fractions. The starch contents of C2 and F2 were: 71.7 and 35.7% (SB89528); 74.9 and 29.2% (Condor); and 69.6 and 24.2% (Glacier), respectively. The starch content of C2 was higher than that of C1 in each case, indicating that the second pass further enriched starch in the C2 fraction. Starch enrichment during second pass was considerably higher in SB89528 (21%) and lower in Condor (6%), but almost similar in Glacier (13%) when compared to the first pass. The protein contents of C2 and F2 were: 10.5 and 14.9% (SB89528); 8.9 and 26.7% (Condor); and 8.9 and 18.6% (Glacier); respectively. F2 had higher protein content than C2. The β -glucan contents of C2 and F2 were: 11.0 and 3.5% (SB89528); 7.0 and 4.3% (Condor); and 10.5 and 2.2% (Glacier); respectively. As in the first pass, β -glucan concentrated into coarse (C2) fractions. The β -glucan enrichment in C2 (from the C1 fraction) ranged from a low of 13% in Glacier to a high of 25% in SB89528.

The C2 fractions from all three barleys were air-classified again (third pass) into C3 and F3 fractions. The yields (as percent of original meal) of C3 and F3 were: 8 and 46% (SB89528); 10 and 63% (Condor); and 21 and 44% (Glacier); respectively. The

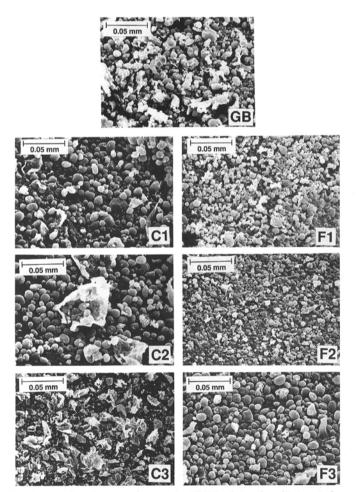


Fig. 3. Scanning electron micrographs (600 \times) of the pin-milled and airclassified Condor barley fractions. GB = ground barley; C1-C3 = coarse fractions; F1-F3 = fine fractions.

composition of C3 and F3 fractions were different from that of the C2 fractions. The starch contents of C3 and F3 were: 15.6 and 77.1% (SB89528); 32.7 and 78.0% (Condor); and 36.4 and 78.1% (Glacier); respectively. Because a higher air-classifier vane setting (30 µm) was used in this case, the third pass enriched starch to a larger extent into fine (F3) fractions, unlike the previous two cases where the starch was largely present in the coarse fractions. The starch enrichment was considerably lower in SB89528 (7.5%) and Condor (4.1%) barleys, but generally similar in Glacier barley (12.2%), when compared to that of the second pass. The protein contents of C3 and F3 were: 13.7 and 9.2% (SB89528); 15.1 and 8.3% (Condor); and 12.2 and 8.0% (Glacier); respectively. C3 had higher protein content than the F3 fractions. The β -glucan contents of C3 and F3 were: 23.8 and 8.1% (SB89528); 13.1 and 6.5% (Condor); and 21.8 and 6.7% (Glacier); respectively. Unlike the first two passes, the third pass at a vane setting of 30 μ m considerably enriched β -glucan in C3 fractions that ranged from 87.1% in Condor to 116.4% in SB89528. In all three barleys, the third pass separated β -glucan and starch into C3 and F3 fractions, respectively. Separation of considerable amounts of β glucan from starch was highly desirable because it helps to reduce slurry thickening during wet-extraction of starch from the F3 fraction.

Gracza (1959) described protein shift during air-classification of a soft wheat flour as a measure of the degree to which protein is concentrated or depleted, relative to the original meal. In the present study, similar calculations were made, and the protein, starch, and β -glucan shifts (for the overall process) are given in Table III. The shifts for each component varied considerably among the cultivars. Protein shift was comparatively higher in Condor (41.3%) than in SB89528 (27.2%) or Glacier (27.8%) barleys. However, starch and β -glucan shifts were comparatively higher in SB89528 (46.1 and 75.3%, respectively) and Glacier (36.4 and 53.3%, respectively) than in Condor (26.9 and 26.3%, respectively).

Scanning Electron Microscopy

Scanning electron micrographs of pin-milled barleys and their air-classified fractions were taken. Due to their general similarities, only the results for Condor barley fractions are given (Fig. 3). Separation of small and large starch granules from the proteinaceous matrix was observed in all three barleys. The bi-modal size and oval shape, typical of regular barley starch, is visible in pin-milled barley. The air-classification of ground barleys (first pass) concentrated most of the small starch granules into F1 fractions and most of the large starch granules into C1 fractions. The second pass further concentrated the small starch granules of C1 fractions into F2 fractions and large starch granules into C2 fractions. The third pass (vane setting, 30 μ m) generated a starch-rich F3 fraction that contained mainly large starch granules. Examinations of the small and large starch granules under high magnification (2,000×) (results not shown) revealed that pinmilling did not cause extensive starch damage; this is in agreement with the data in Table I.

Small and large starch granules from barley differ in their physicochemical properties (Bathgate and Palmer 1972). Youngs (1993) described wet milling of barley by Alko Ltd., Finland.

TABLE III Starch, Protein, and β -Glucan Shifts During Air Classification of Pin-Milled Barleys

		Shifts (%) ^a				
Cultivar	Starch Type	Starch (F3) ^b	Protein (F1 + F2 + C3) ^b	β-Glucan (F3 + C3) ^b		
SB89528	Waxy	46.1	27.2	75.3		
Condor	Normal	26.9	41.3	26.3		
Glacier	High amylose	36.4	27.8	53.3		

^aCalculated according to Gracza (1959).

^bFractions considered as rich in starch, protein, or β -glucan for the shift calculation. F1-F3 = fine fractions; C3 = coarse fraction.

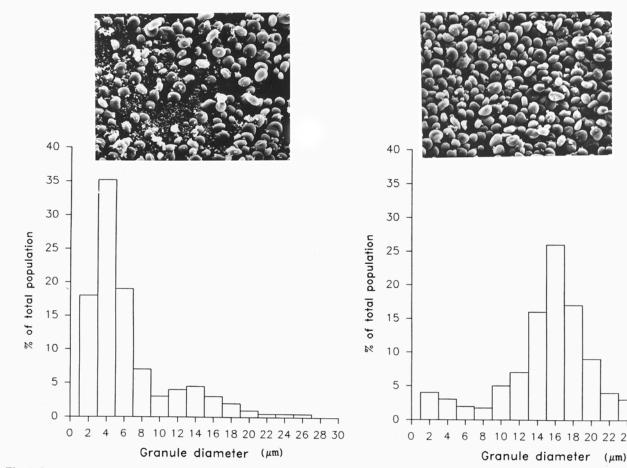


Fig. 4. Granule size distributions of Condor starches purified from ground barley and the fine 3 fraction.

TABLE IV Recoveries (%) and Efficiencies (%) of Starch Extractions^a

Cultivar	Extraction Procedure	Starch Content ^b	Starch Recovery ^c	Starch Extraction Efficiency ^d
SB89528	Conventional	53.6 ± 0.7	35.3 ± 2.5	65.8
	Short wet processing	77.1 ± 0.5	66.6 ± 3.4	86.4
Condor	Conventional	62.0 ± 0.9	39.7 ± 1.9	64.0
	Short wet processing	78.0 ± 1.1	65.6 ± 3.1	84.4
Glacier	Conventional	55.3 ± 2.7	37.5 ± 2.6	67.8
	Short wet processing	78.1 ± 1.6	68.4 ± 3.1	87.6

^aValues are means of three determinations.

The operation fractionates dehulled barley into fiber, solubles, protein, A-starch (large granule starch), and B-starch (small granule starch). A-starch is used for paper coating and B-starch is used for the production of potable alcohol. The other fractions are utilized by the feed industries. Zhao and Whistler (1994) reported that when small starch granules from amaranth and wheat are spray-dried with small amounts of bonding agents, they can be transformed into porous spheres to produce flavor carriers in foods. Separation of small and large starch granules by an aqueous sedimentation and decantation process (Bathgate and Palmer 1972) would not be economical because it would generate a voluminous effluent for high-cost disposal. The present study suggests that air classification can substantially separate large and small granule starches. However, the final starch-rich fraction (F3) contained protein and β -glucan. These were removed by a short wet-extraction procedure (Fig. 2) that produced almost pure large granule starch, protein, and β -glucan fractions.

Further Purification of F3 Fractions

Starch was purified from all three F3 fractions by a short wetextraction procedure (Fig. 2) for comparison with a conventional laboratory starch-extraction method for barley. The granule size distributions and starch extraction efficiencies obtained for the two procedures were compared. F3 fraction was slurried in distilled water and filtered through a 53- μ m polypropylene screen to remove cell-wall materials. The filtrate was adjusted to 0.05NNaOH to solubilize proteins adhering to starch granules, and pure starch was recovered by centrifugation (Fig. 2). In the conventional procedure, barleys (seeds) were ground into meals, slurried in distilled water, and further purified by screening, alkali-washing, and centrifugation to yield starch.

10 12 14 16 18 20 22 24 26 28 30

The granule-size distributions of starches purified from barley grains by the conventional procedure and by the short wetextraction of F3 fractions (Fig. 2) were investigated for all three barleys. Because of their general similarities, only the results for Condor barley are given (Fig. 4). Starch purified from barley by the conventional procedure contained ~80% small starch granules (number basis) with a diameter range of 2-8 μ m. The large granules made up $\sim 20\%$ and ranged from 10-26 μm in diameter. However, starch purified from the F3 fractions contained ~85% large granules and ~15% small granules, thus showing improved uniformity of granule size.

The starch-extraction efficiencies for the two procedures are given in Table IV. The starch-extraction efficiencies of the conventional procedure were: 65.8% (SB89528); 64.0% (Condor); 67.8% (Glacier); the mean was 66%. The mean value obtained for starch extracted from the F3 fractions using the short wet-extraction (Fig. 2) was 86% (30% higher). Furthermore, purification of starch from the F3 fractions had a number of advantages: 1) using a high-starch fraction (F3) as raw material reduces the water requirement, centrifuge load, effluent disposal cost, and drying cost; 2) during pin milling and air classification, considerable amounts

b% dry weight basis.

^cWeight (g) of starch recovered from 100 g of ground barley or fine 3 fraction.

^dEfficiency = (recovery/starch content) \times 100.

of β -glucan shifted into fractions other than F3; therefore, less β -glucan was taken through the wet process, which reduced slurry thickening; and 3) purification of F3 fraction yielded almost pure large granule starch.

CONCLUSIONS

The study showed that pin-mill grinding and air classification produced barley fractions rich in protein (17–29%), β -glucan (13–24%), and starch (77–78%). Protein-rich fractions (F1 and F2) had considerable amounts of starch (24–36%) due to concentration of small granule starch during air classification. The β -glucan concentrated with the C1, C2, and C3 fractions. However, the enrichment was highly pronounced during air classification at the 30- μ m vane setting. In all three barleys, air classification separated large starch granules from the small granules and concentrated them into F3 fractions. This enabled further purification of large granule barley starch by a short wet-extraction procedure. Purification of starch from F3 fractions gave ~30% higher extraction efficiencies than did the conventional method of starch extraction.

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