# High-Starch and High-β-Glucan Barley Fractions Milled with Experimental Mills

Rolando A. Flores,[1,2] Kevin B. Hicks,[1] Dale W. Eustace,[3] and John Phillips[4]

## ABSTRACT

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This study focused on the performance of two hulless barley cultivars (Doyce and Merlin) and one commercial husked (hulled) sample using experimental milling. The purpose was to use experimental milling as a preliminary indicator of the milled streams with potential use for fuel ethanol production and fractions that could be used in food products. Experimental mills designed for flour production evaluation from wheat were Chopin CD1 Auto, Quadrumat Sr, Buhler, and an experimental Ross roller mill walking flow. Results indicate that the shorts had the highest levels of β-glucan from all the mills. However, the β-glucan content in the break flours was highest with the roller mill walking flow and the Chopin CD1 for the hulless cultivars. The lowest β-glucan content in the break flour was found with the Buhler for Doyce. Break flour and, to a slightly lesser extent, reduction flour from all cultivars tested on all mills contained the highest starch content (up to 83%) and are therefore most appropriate for use as feedstock for fuel ethanol production. Conversely, bran and shorts from all cultivars and mills were lowest in starch (as low as 25%), making them ideal as low-starch food ingredients.

Barley has traditionally been used in the brewing and feed industries. New cultivars are being developed that widen the applications of this crop to nontraditional food and nonfood value-added applications such as ethanol production. Hulless (hull-less) or naked cultivars present new alternatives and processing or evaluation challenges (Bhatty 1999b). Dry milling of barley for fractionation purposes is considered a viable process for isolating valuable components of hulless and hulled (husked) barley such as β-glucan-rich streams (Sundberg and Åman 1994; Andersson et al 2000; Warpala and Pandiella 2000; Wu and Doehlert 2002; Izydorczyk et al 2003).

Experimental milling is a well-established tool used by millers to evaluate commercial samples before processing a batch of wheat in the flour mill (Kim et al 1995). Most of the experimental milling techniques are based on the utilization of a milling unit designed for that purpose such as the Buhler, Chopin, or Quadrumat (Jr and Sr) experimental mills, among other types. Alternatively, mill walking flows are developed by millers to approximate the milling results and conditions in a given flour mill using different particle reduction equipment.

As new barley cultivars are developed and new processes designed to use some major components of that crop, experimental procedures for milling the samples need to be developed to determine the potential of a new cultivar based on the final application of the product. The fractionation of barley using dry milling is important not only for the production of bread and similar products and high β-glucan fractions, but also for alternative utilization in ethanol production. We are interested in dry fractionating barley into high and low starch fractions for fuel ethanol production and healthy food ingredients, respectively. Experimental milling results could assist in the determination of the potential of a barley cultivar in ethanol production with a dry fractionation process in front of the liquefaction and saccharification.

Therefore, the objective of this study was to evaluate two hulless barley cultivars (Doyce and Merlin) and one commercial hulled sample using three experimental milling procedures as preliminary indicators of the milled streams with potential use for fuel ethanol production and fractions that could be used in food products.

## MATERIALS AND METHODS

### Materials

Three barley samples, two hulless and one commercial hulled, were used in this study. The hulless samples were 1) Merlin, a two-row spring barley cultivar harvested in 2002 from Three Forks, MT; and 2) Doyce, a six-row winter barley, harvested in 2002 from Mt. Holly, VA. The commercial hulled sample was a mix of bin winter barley #3 cultivars harvested in 2003 and procured in Ridgely, MD. The moisture content of the samples was 10.9, 13.9 and 11.9% (wb) for Merlin, Doyce and commercial hulled barleys, respectively.

### Methods

The clean samples were processed in experimental mills according to the manufacturers unaltered flows: CD1 Auto (Chopin, 92396 Villeneuve-la-Garenne, France), Quadrumat Sr (Brabender, Duisburg, Germany), and MLU-202 (Buhler, Uzwil, Switzerland) with a 0.076-mm roll gap for the second break. Also, the samples were processed in a simplified short-walking Ross rolls flow shown in Fig. 1.

Two types of runs were conducted in the Chopin CD1 Auto with Merlin and commercial hulled barley. One run was identified as Chopin 1-pass and the other run as Chopin 2-pass. In the Chopin 1-pass, the barley was run through the mill and results were recorded. In the Chopin 2-pass, the barley was run once through the mill, then the reduction bran stream was run once more to optimize the flour extraction of the mill. The streams produced from the Chopin CD1 were break bran, redo break bran, break flour, redo break flour, reduction flour, redo reduction flour, and redo reduction bran. These seven streams were combined into break flour, shorts and fine bran (now on shorts), bran (includes the hulls when hulled barley was milled), and reduction flour. Tempering of barley samples before milling has shown an increase in the millfeeds material and reduced the flour products (Izydorczyk et al 2003). However, to achieve better results, samples processed in the Chopin CD1 were conditioned to 16% moisture (wb) for 20 hr before milling. An additional run was processed in the Quadrumat Sr with Merlin barley tempered at 15.5% for 18 hr. No tempering was done with the other experimental milling processes. The average milling rate was 69.5 g/min in the Chopin

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[1] Crop Conversion Science and Engineering Research Unit, Eastern Regional Research Center, Agricultural Research Service, U.S. Department of Agriculture, 600 East Mermaid Lane, Wyndmoor, PA 19038.

[2] Corresponding author. Phone: 215-233-6489. Fax: 215-233-6406. E-mail: rflores@errc.ars.usda.gov

[3] Department of Grain Science and Industry, Kansas State University, Manhattan, KS..

[4] North Atlantic Area, Agricultural Research Service, U.S. Department of Agriculture, 600 East Mermaid Lane, Wyndmoor, PA 19038.

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CD1 for all samples, and 130 g/min for the hulless barley and 70 g/min for the hulled barley in the Quadrumat Sr and Buhler experimental mills.

The moisture measurement of the grain samples was conducted according to Method S352.2 (ASAE 2003b). The physical and chemical analyses of the milling streams were conducted using the Approved Methods 55-10, 44-19, 08-01, 46-30, 32-32, and 76-13 for test weight, moisture, ash, protein, β-glucan, and starch, respectively (AACC International 2000). Thousand kernel weight was calculated counting the kernels in a 40-g sample of clean and whole barley kernels using a Count-A-Pak seed totalizer (model 77, Seedburo, Chicago, IL). Lipid content of the grains was determined using the accelerated solvent extraction method (Moreau et al 2003). The color measurement of the flour streams was conducted using a spectrophotometer (ColorQuest XE, Hunter Associates Laboratory, Reston, VA) with the CIE Lab color scale ($L^*$, $a^*$, and $b^*$). Particle size analyses of the milled streams were conducted by determining geometric mean diameter and standard deviation of the streams using Method S319.3 (ASAE 2003a).

### Statistical Analyses

All tests were conducted in triplicate. The data were analyzed using analysis of variance to determine the effects and interactions of the experimental factors such as milling method, cultivar, and milling stream on the various measured responses. Mean separations were performed using the Bonferroni LSD technique. The level of significance referred to in this study is $\alpha = 0.05$.

## RESULTS AND DISCUSSION

The researchers were interested in fractionating barley into high and low starch fractions for fuel ethanol production and healthy food ingredients, respectively. Therefore, experimental milling is expected to be of assistance in identifying potential cultivars and in evaluating dry milling streams.

The commercial and chemical characteristics of barley samples studied are shown in Table I. The commercial hulled barley sample (hulled) had a significantly lower test weight than Merlin and Doyce samples due to the lack of hull in the Merlin and Doyce samples. The highest thousand kernel weight (TKW) corresponds to hulless barleys; Merlin was the highest of all (39.9 g), followed by Doyce (29.9 g), and the lowest was hulled barley (28.0 g). Also, the hulled barley had the highest ash content (2.4% db); however, it was not significantly different from that of Merlin (2.3% db). The Doyce sample had the highest starch content (66.5% db) and lowest amount of lipids (1.9% db), which were both significantly different from those of Merlin and hulled barleys. The β-glucan (6.2% db) and protein (15.2% db) contents of Merlin were significantly higher; the levels of Merlin for β-glucan and protein were 1.4 and 1.8 times higher, respectively, than the levels of the other two samples, which were not significantly different in β-glucan and protein contents among themselves.

The characteristics under study were milling yields, moisture, ash, β-glucan, starch, protein, color, and particle size. To evaluate the streams from each milling procedure, the streams produced by each experimental mill were arranged into four major streams: break flour, reduction flour, shorts, and bran.

### Flour Yield

Table II shows the flour yields obtained from the experimental milling procedures. The highest amount of break flour was obtained from Merlin under the walking Ross rolls flow (33%). The break flour yield produced from Merlin was 11% more than that produced from Doyce when the same walking Ross rolls flow was used for both barleys. Merlin was a plumper kernel than Doyce with very similar test weights (≈58 lb/bu) but a 33% higher TKW. The hulled barley showed the lowest break flour yield with the Buhler. A comparison of the milling methods and the cultivars showed that the Buhler produced the largest amount of reduction flour with Doyce (45%). For shorts production, the walking Ross rolls flow produced the highest yield with Doyce (51%) and the second highest with hulled barley (37%). The Buhler experimental mill produced the largest yield of shorts from Merlin (39%), closely followed by the hulled barley (38%). The Chopin 2-pass of Merlin produced a more even distribution of streams than the Chopin 1-pass because the 2-pass redistributed the amount of bran mostly to shorts (+12%) and flours (+2%).

From a statistical point of view, the interactions of the milling method, cultivar, and milling stream on milling yield were not significantly different.

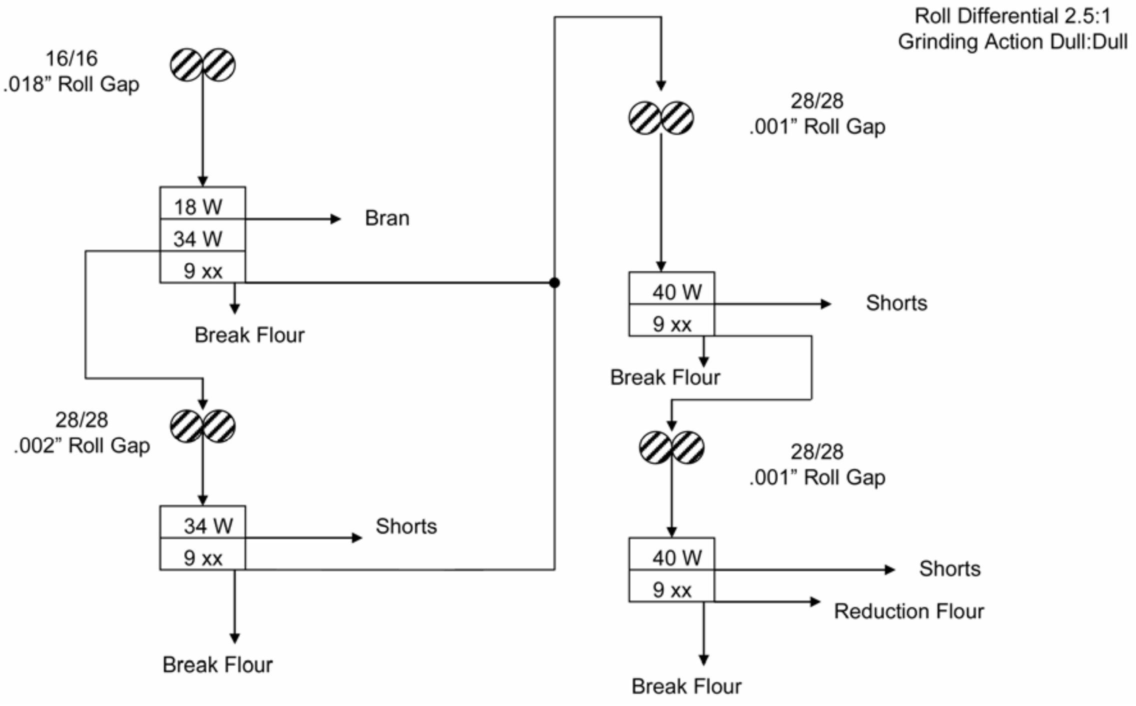

**Fig. 1.** Simplified short-walking Ross rolls flow.

For evaluation purposes, an additional run was processed in the Quadrumat Sr mill with Merlin barley tempered at 15.5% for 18 hr. Comparing the untempered versus the tempered results for the Merlin samples, the break and reduction flour yields as well as the shorts yields decreased from 19 to 17%, from 21 to 12%, and from 25 to 14%, respectively. In turn, the bran yield increased from 35 to 63%. Thus, the tempering increased the millfeeds material and reduced the flour products. This finding is consistent with similar findings regarding other hulless cultivars previously milled (Bhatty 1997; Kiryluk et al 2000; Izydorczyk et al 2003). Tempering is used in wheat milling to assist in the separation of the bran; however in Merlin the results were not as definitive. The increase in the bran yield could be the result of a different aleurone layer structure of Merlin barley when compared with wheat. Because the tempered run was used for evaluation purposes, other characteristics of Merlin tempered streams were not evaluated; therefore, the tempering conditions need to be studied further.

Bhatty (1999a) milled Merlin barley in a Buhler mill under conditions similar to the ones use in this study. Bhatty indicated that the flour extraction was inversely proportional to the β-glucan content. Bhatty reported combined flour yield for the Merlin was 45.1%, while in this study we obtained 24% for β-glucan barley of 6.2% db. Bhatty reported a β-glucan content of 6.4% without indicating dry or wet basis; nonetheless, differences in β-glucan

**TABLE I**
**Characteristics of Unprocessed Barley Samples[a]**

| Barley | Test Wt (lb/bu) | 1000 Kernel Wt (g) | Ash (% db) | β-Glucan (% db) | Starch (% db) | Lipids (% db) | Protein (% db) |
|---|---|---|---|---|---|---|---|
| Commercial (hulled) | 47.7 ± 0.3b | 28.0 ± 0.1c | 2.4 ± 0.1a | 4.3 ± 0.1b | 55.3 ± 1.3b | 2.07 ± 0.03b | 8.3 ± 0.1b |
| Merlin (hulless) | 57.8 ± 0.3a | 39.9 ± 0.2a | 2.3 ± 0.0a | 6.2 ± 0.2a | 57.0 ± 1.1b | 2.52 ± 0.09a | 15.2 ± 0.4a |
| Doyce (hulless) | 58.4 ± 0.2a | 29.9 ± 0.1b | 1.9 ± 0.0b | 4.0 ± 0.1b | 66.5 ± 1.2a | 1.87 ± 0.01c | 8.7 ± 0.1b |

[a] Mean values ± one standard deviation. Values followed by the same letter are not significantly different ($P < 0.05$).

**TABLE II**
**Milling Yields, Moisture, and Ash of Milled Streams**

| Barley Mill Stream | Buhler | Ross Rolls | Quadrumat Sr | Chopin (1-pass) | Chopin (2-pass) |
|---|---|---|---|---|---|
| Milling yields (%) | | | | | |
| Doyce | | | | | |
| Bran | 12 | 7 | 16 | | |
| Shorts | 18 | 51 | 28 | | |
| Reduction flour | 45 | 20 | 31 | | |
| Break flour | 25 | 22 | 25 | | |
| Merlin | | | | | |
| Bran | 37 | 17 | 35 | 37 | 20 |
| Shorts | 39 | 7 | 25 | 18 | 30 |
| Reduction flour | 15 | 43 | 21 | 20 | 22 |
| Break flour | 9 | 33 | 19 | 26 | 28 |
| Commercial hulled | | | | | |
| Bran | 26 | 29 | 23 | 36 | 26 |
| Shorts | 38 | 37 | 29 | 19 | 25 |
| Reduction flour | 26 | 14 | 26 | 22 | 22 |
| Break flour | 10 | 20 | 22 | 23 | 27 |
| Moisture (%, wb) | | | | | |
| Doyce | | | | | |
| Bran | 8.15 | 7.82 | 8.60 | | |
| Shorts | 7.77 | 8.14 | 9.02 | | |
| Reduction flour | 8.16 | 9.05 | 9.89 | | |
| Break flour | 8.59 | 9.90 | 10.8 | | |
| Merlin | | | | | |
| Bran | 7.89 | 8.34 | 8.13 | 10.7 | 10.1 |
| Shorts | 7.49 | 7.69 | 8.24 | 8.92 | 9.28 |
| Reduction flour | 7.80 | 8.37 | 9.27 | 11.7 | 12.2 |
| Break flour | 7.88 | 9.05 | 10.1 | 13.7 | 14.6 |
| Commercial hulled | | | | | |
| Bran | 5.94 | 6.52 | 6.54 | 9.58 | 8.40 |
| Shorts | 6.95 | 6.30 | 8.68 | 9.38 | 9.06 |
| Reduction flour | 7.85 | 8.31 | 9.82 | 12.8 | 13.0 |
| Break flour | 7.67 | 9.35 | 10.6 | 16.3 | 16.7 |
| Ash (%, db) | | | | | |
| Doyce | | | | | |
| Bran | 2.84 | 2.8 | 2.9 | | |
| Shorts | 2.62 | 3.48 | 3.05 | | |
| Reduction flour | 1.14 | 2.61 | 1.72 | | |
| Break flour | 1.04 | 1.02 | 0.86 | | |
| Merlin | | | | | |
| Bran | 2.22 | 3.12 | 2.57 | 3.03 | 3.27 |
| Shorts | 2.71 | 2.89 | 2.76 | 3.38 | 3.21 |
| Reduction flour | 1.46 | 2.59 | 2.2 | 1.50 | 1.46 |
| Break flour | 1.35 | 1.29 | 0.97 | 1.09 | 1.07 |
| Commercial hulled | | | | | |
| Bran | 2.82 | 2.54 | 3.23 | 4.03 | 4.45 |
| Shorts | 2.45 | 3.42 | 2.99 | 3.23 | 3.36 |
| Reduction flour | 1.39 | 2.75 | 1.53 | 1.26 | 1.20 |
| Break flour | 1.49 | 1.37 | 1.29 | 1.12 | 1.07 |

were relatively minor and the difference in the flour yield must also be attributed to some other factor such as the much larger TKW of Merlin barley milled by Bhatty (50.5 g) compared with the one used in this study (39.9 g). The assertion that the β-glucan content negatively affects the flour yield was found herein for the Buhler and Quadrumat milling methods. In our study, the flour yield for Merlin was 46 and 16% lower than Doyce in the Buhler and Quadrumat, respectively. Nonetheless, in the walking Ross rolls flow, Merlin had a 34% higher flour yield than Doyce as a result of using a more flexible and adjustable milling flow when compared with the less flexible methods in the Buhler, Quadrumat, and Chopin. However, the Buhler and Quadrumat are less susceptible to operator effect and thus more suitable for uniform comparison of barleys. To produce a wider classification of the flours, additional sieving could have been conducted on the flour streams produced by the different milling methods but it was outside the scope of this study. Sundberg and Åman (1994) used additional sieving on flour streams produced with a similar Buhler system on hulless barleys to identify other components such as xylan, cellulose, and Klason lignin in addition to β-glucan. Also, Izydorczyk et al (2003) developed and optimized a long milling flow that allowed them to obtain fiber-rich fractions.

## Moisture

Moisture distribution in the milled streams obtained from the different milling methods is shown in Table II. Table II shows results for the tempered barleys processed in the Chopin 1-pass and 2-pass methods. Thus, the streams with the highest moisture content were the ones resulting from the Chopin CD1; the moisture content of these streams were significantly different from the moisture content of all the other streams as a consequence of the tempering. The moisture range for all the other untempered streams was 6.3–10.76% (wb) for the hulled shorts and the Doyce break flour. There was evidence that all two-factor interactions were statistically significant. Excluding the stream moisture results of the Chopin, the bran and shorts streams were not significantly different when the milling methods are compared. However, the break and reduction flours are not significantly different between the Quadrumat and Ross rolls and between the Ross rolls and Buhler methods; but significantly different between the Buhler and Quadrumat.

**TABLE III**
**Biochemical Composition of Milled Streams**

| Barley Mill Stream | Buhler | Ross Rolls | Quadrumat Sr | Chopin (1-pass) | Chopin (2-pass) |
|---|---|---|---|---|---|
| β-Glucan (%, db) | | | | | |
| Doyce | | | | | |
| Bran | 4.45 | 3.88 | 4.23 | | |
| Shorts | 5.30 | 4.26 | 5.56 | | |
| Reduction flour | 1.46 | 5.55 | 3.46 | | |
| Break flour | 1.17 | 1.59 | 1.31 | | |
| Merlin | | | | | |
| Bran | 6.90 | 7.02 | 7.09 | 7.28 | 7.79 |
| Shorts | 7.56 | 8.16 | 8.74 | 9.86 | 3.33 |
| Reduction flour | 1.91 | 7.22 | 3.15 | 4.50 | 4.58 |
| Break flour | 1.50 | 1.64 | 1.24 | 2.85 | 2.99 |
| Commercial hulled | | | | | |
| Bran | 1.96 | 2.87 | 2.12 | 3.24 | 2.98 |
| Shorts | 5.59 | 2.91 | 5.32 | 7.11 | 6.62 |
| Reduction flour | 2.21 | 2.90 | 3.77 | 4.11 | 4.82 |
| Break flour | 1.78 | 2.55 | 1.76 | 3.32 | 3.32 |
| Starch (%, db) | | | | | |
| Doyce | | | | | |
| Bran | 56.5 | 57.5 | 55.3 | | |
| Shorts | 57.2 | 50.3 | 52.0 | | |
| Reduction flour | 77.7 | 58.0 | 67.9 | | |
| Break flour | 82.6 | 81.2 | 83.5 | | |
| Merlin | | | | | |
| Bran | 53.9 | 45.5 | 52.2 | 51.2 | 42.6 |
| Shorts | 49.9 | 52.5 | 46.1 | 40.6 | 42.8 |
| Reduction flour | 65.6 | 52.3 | 55.9 | 67.4 | 67.9 |
| Break flour | 70.7 | 72.0 | 71.9 | 76.6 | 75.9 |
| Commercial hulled | | | | | |
| Bran | 54.3 | 40.0 | 23.6 | 32.2 | 25.4 |
| Shorts | 54.3 | 31.2 | 49.9 | 44.6 | 25.4 |
| Reduction flour | 54.8 | 58.4 | 69.8 | 75.2 | 77.1 |
| Break flour | 74.8 | 79.6 | 82.5 | 80.5 | 83.4 |
| Protein (%, db) | | | | | |
| Doyce | | | | | |
| Bran | 11.0 | 11.1 | 10.9 | | |
| Shorts | 11.3 | 11.7 | 11.2 | | |
| Reduction flour | 10.1 | 11.8 | 11.3 | | |
| Break flour | 8.1 | 7.6 | 6.5 | | |
| Merlin | | | | | |
| Bran | 16.3 | 19.3 | 17.2 | 17.0 | 17.6 |
| Shorts | 17.0 | 16.4 | 16.6 | 16.5 | 17.1 |
| Reduction flour | 15.2 | 15.7 | 18.9 | 13.2 | 12.7 |
| Break flour | 13.3 | 13.8 | 13.8 | 10.7 | 10.5 |
| Commercial hulled | | | | | |
| Bran | 7.8 | 8.3 | 8.9 | 8.8 | 8.9 |
| Shorts | 13.3 | 11.7 | 13.3 | 10.6 | 11.1 |
| Reduction flour | 9.8 | 12.5 | 12.1 | 7.8 | 7.4 |
| Break flour | 9.3 | 9.0 | 8.4 | 6.0 | 5.7 |

## Ash

Ash content in a flour stream is an indicator of the presence of pericarp material for hulless barley and also hulls for hulled barley. The largest amount of ash was obtained from the bran streams (4.45% db) of the hulled barley with the Chopin 2-pass, as a result of having the hulls in these streams (Table II). Because hulled barley has the hull attached to the kernel, the ash content is slightly larger in hulled than in hulless kernels (2.36% db). All experimental mill processes allocated the streams with the largest amount of ash to the shorts and bran for the hulless barleys; and to the shorts, bran and reduction flour for the hulled barleys. This could indicate that some hull was ground into the reduction flour for the hulled barley. All systems produced less ash in break flour.

The statistical evaluation of the interaction of the three factors studied indicated that there was no evidence of a significant cultivar effect while there was a significant milling method and milling stream interaction effect. The bran and shorts streams had significantly larger amounts of ash when compared with the flour streams, regardless of the type of barley milled, for all milling methods. However, on the average, the Buhler mill produced bran and shorts with lower ash contents than the other mills. The ash content of the break flour was significantly lower only for the Ross rolls walking flow. For the other milling methods (Buhler, Chopin 1-pass and 2- pass, and Quadrumat), the ash contents of the break and reduction flours were not significantly different.

## β-Glucan

For the Doyce barley, the highest β-glucan yielding streams were found in the shorts of the Buhler and the Quadrumat Sr mills, and in the reduction flour of the walking Ross rolls flow, all between 5.3 to 5.6% db (Table III). Merlin shorts had the highest β-glucan content for the Buhler, walking Ross rolls flow, and Quadrumat mills. However, the highest β-glucan Merlin stream was found in the shorts produced with the Chopin 1-pass (9.9%). The Chopin 2-pass redistributed Merlin β-glucan material more into the bran stream, from 9.9 to 3.3% db. The milling yields of Merlin shorts and bran streams went from 18 and 37% for the Chopin 1-pass, to 30 and 20% for the Chopin 2-pass (Table II), respectively. In the hulled barley, the streams with the highest content of β-glucan corresponded to the shorts for the Chopin 1-pass (7.1% db) and Chopin 2-pass (6.6% db), respectively.

There was no evidence of an overall significant effect of the milling method, while there was a significant cultivar and milling stream interaction effect on the β-glucan yields. Regardless of the milling method, the β-glucan content was highest in the shorts and lowest in the break flour for the interaction of all three barleys. A similar finding was reported by Kiryluk et al (2000) using a Quadrumat Jr. Additional grinding and air classification has been used to increase the β-glucan content of selected milling of streams barley and oats (Andersson et al 2000; Wu and Doehlert 2002).

## Starch

As expected, the highest concentration of starch was found in the break and reduction flours for all barleys under all processes (Table III). For Doyce, the highest concentration of starch was obtained in the break flour produced with the Quadrumat Sr (83.5% db) and Buhler (82.6% db) experimental mills, followed closely by the walking Ross rolls flow (81.1% db). The Merlin reduction flour had a starch range for all milling processes of 52.3–67.9% (db). The bran and shorts streams produced by the Buhler had similar starch level ranges; the difference went from 0.7 to 3.9%, a low difference for the magnitude of the starch recovered.

When the mean values of the experimental factors were statistically analyzed, it was found that there was no evidence of interaction among the milling method, cultivar, and milling stream; however, there were significant cultivar and milling stream interaction effects. Doyce was significantly different from hulled and Merlin barleys; this difference was a result of the original starch content of the unprocessed seed (Table I). The starch contents of the break and reduction flour streams were significantly different among them and were also significantly different from the bran and shorts streams; however, the bran and shorts streams were not significantly different among themselves. A coefficient of determination of 0.845 was found between the ash and starch contents relationship for all the milling streams.

## Protein

The protein streams were similar among all milling methods for the shorts and bran from Doyce (Table III). For Doyce flour protein content, the numbers for the reduction flour were higher for the walking Ross rolls flow and lowest for the break flour; alternatively, the protein for Doyce flour was higher in the reduction than in the break flour with the Quadrumat Sr. With the exception of the break flour stream, the Merlin streams with the Buhler process were evenly distributed at 15.1–17.0% (db). With the walking Ross rolls flow, the highest protein for Merlin was found in the bran stream (19.3% db). Merlin shorts and bran showed the highest concentration of protein with the Chopin 1-pass or 2-pass (16.5–17.6% db). The hulled barley had the lowest protein levels in the break flour streams of the samples processed in the Chopin CD1 (6.0% db for the 1-pass and 5.7% db for 2-pass, respectively).

There was no evidence of the interaction of the three main effects, while there was a significant milling method and milling stream interaction effect and a significant barley and milling stream interaction effect. Therefore, the protein content for all the barleys under all milling methods was significantly lowest in the break flour. However, for the hulled barley, the protein content difference between the bran and break flour streams was not significantly different. For Doyce, the bran, reduction flour, and shorts were not significantly different.

**TABLE IV**
**Color Parameters for Average Interaction Between Barley and Milling Stream[a]**

| Barley Sample | Milling Stream | *L** | *a** | *b** |
|---|---|---|---|---|
| Commercial hulled | Bran | 76.5 ± 3.6c | 2.5 ± 0.4a | 12.1 ± 0.6a |
| | Shorts | 81.1 ± 3.8bc | 1.9 ± 0.5ab | 9.5 ± 1.2b |
| | Reduction flour | 87.3 ± 3.9a–c | 1.2 ± 0.6c–e | 7.4 ± 1.3bc |
| | Break flour | 88.6 ± 3.3ab | 1.0 ± 0.5d–f | 6.5 ± 1.3c |
| Merlin | Bran | 89.5 ± 1.5ab | 1.3 ± 0.2c–e | 9.3 ± 1.1b |
| | Shorts | 88.7 ± 1.0ab | 1.4 ± 0.2b–e | 9.4 ± 0.7b |
| | Reduction flour | 90.8 ± 2.0ab | 0.7 ± 0.4ef | 7.7 ± 0.8bc |
| | Break flour | 92.8 ± 1.7a | 0.5 ± 0.2f | 6.6 ± 0.3c |
| Doyce | Bran | 76.8 ± 15.5c | 1.6 ± 0.1b–d | 9.1 ± 1.4b |
| | Shorts | 85.0 ± 1.6a–c | 1.7 ± 0.1bc | 9.1 ± 1.4b |
| | Reduction flour | 90.2 ± 4.1ab | 0.8 ± 0.5ef | 7.3 ± 0.9bc |
| | Break flour | 92.6 ± 0.8a | 0.5 ± 0.1f | 6.7 ± 0.5c |

[a] Averaged value for all milling streams and milling methods for each barley cultivar. Mean value ± one standard deviation. Values followed by the same letter are not significantly different ($P < 0.05$).

## Color

The color data shown in Table IV are the resulting averages for the significant double interaction between the type of barley and the milling stream for the $L^*$, $a^*$, and $b^*$ tristimulus parameters. The measure of lightness ($L^*$) for Doyce and hulled bran were not significantly different among themselves but were significantly different from the other streams and barleys. The flour lightness was higher for the hulless than the hulled barley streams as a consequence of the hull and pericarp particles present in the flour, even though this difference was not statistically significant. The $L^*$ values found in this study are lower than the values reported by Izydorczyk et al (2003) using a long flow roller milling with hulless barley cultivars. The level of redness ($+a^*$) proved to be a significantly different parameter when the bran and shorts were compared with the flours. The level of yellowness ($+b^*$) was significantly different for the hulled bran stream because of the hull presence. For the interaction of the milling stream and milling method, the only significant color parameter was $a^*$. In the double interaction between the barley type and milling stream, the bran and shorts were significantly different from the two flour steams. The double interaction between the milling method and barley type was significant for the $a^*$ parameter. The hulled barley was significantly different for all the milling methods with the exception of the Chopin 1-pass.

The relationship between the chemical characteristics of each stream and the color parameters was investigated using all the streams as a data source. There were correlations between the ash and starch of each stream and the color tristimulus parameters. The coefficients of determination for ash were 0.47, 0.60, and 0.71 for $L^*$, $a^*$, and $b^*$, respectively; and for starch they were 0.49, 0.60, and 0.73 for $L^*$, $a^*$, and $b^*$, respectively.

## Particle Size

The particle size analysis was based on the determination of the geometric mean and standard deviation for each stream. As expected, the statistical analyses of the resulting data indicated that there was evidence of significant milling method by milling stream interaction and cultivar by milling stream interaction. Table V shows the geometric particle size mean diameter results (±1 SD). The geometric mean diameter was 214.5–381.1 μm for the shorts, 330.6–385.7 μm for bran, 69.1–224.0 μm for break flour, and 88.1–262.8 μm for reduction flour. Even though from a statistical point of view there are significant differences, the milled streams are within close ranges, with the exception of the commercial hulled barley break flours for the Buhler and Ross walking flows. The Quadrumat Sr produced the milled streams with lowest particle size range for all barleys milled within a given stream.

In the identification of potential barley cultivars for ethanol production, the goals of the researchers are the production of high starch fractions for fermentation and low starch fractions for healthy food ingredients. One example, focusing on starch levels in one of the best fractions, was the Doyce break flour from the Buhler mill. It has ≈83% starch, an increase of 18% over the starting grain. The yield is ≈25%. The reduction flour has ≈78% starch and a yield of ≈45%. Combining these two fractions gives ≈70% of the original mass that contains 85% of the starch. Use of these fractions together for fermentation could give higher fermentable solids and higher ethanol concentrations, making ethanol production more efficient. The remaining two fractions, containing high levels of pericarp and dietary fiber and lower starch concentrations (56.5%) could be useful for lower carbohydrate food ingredients. The hulled barley, which is a typical Mid Atlantic cultivar, has a very low starch level of 55%, which would preclude its use as an efficient feedstock for fuel ethanol. The Chopin 2-pass break flour from this hulled barley has a yield of ≈28% and has a remarkable starch content of 83%, an enrichment approaching 30%. The reduction flour from the same mill has 77% starch and a yield of ≈22%. Combining these two fractions would give a yield of >50% flour containing almost 81% starch. The shorts and bran fraction, containing very low levels of starch (25%) could be very useful as a low starch, high dietary fiber, β-glucan-enriched food ingredient. As mentioned earlier, these milling systems were developed primarily for wheat. Optimization and modifying them for hulled and hulless barley could yield even better enrichment of starch and nonstarch fractions. In this regard, the Ross rolls flow is a very flexible option for the dry fractionation before ethanol production.

## CONCLUSIONS

The experimental milling techniques evaluated in this study used equipment and flows that were designed for wheat flour production. Nonetheless, the techniques studied on the three barley cultivars provide guidance for their application depending on the final utilization of the barley. Ultimately, the final use of a given barley (ethanol production in our case) is what is going to determine which procedure is appropriate for the barley. Even though the tested processes were not optimized for each cultivar,

**TABLE V**
**Geometric Mean Diameter of Milled Streams (μm)[a]**

| Milling Method and Barley Sample | Bran | Shorts | Reduction Flour | Break Flour |
|---|---|---|---|---|
| Buhler | | | | |
| Doyce | 347 ± 2G | 224 ± 2Q | 160 ± 1U | 144 ± 1X |
| Merlin | 361 ± 1F | 249 ± 2O | 171 ± 2T | 155 ± 1V |
| Commercial hulled | 361 ± 1F | 215 ± 2R | 150 ± 1W | 69 ± 2g |
| Ross Rolls | | | | |
| Doyce | 378 ± 1CD | 381 ± 1BC | 230 ± 2P | 107 ± 2d |
| Merlin | 379 ± 1BC | 375 ± 1D | 263 ± 2N | 176 ± 2S |
| Commercial hulled | 386 ± 1A | 381 ± 1B | 224 ± 2Q | 91 ± 2e |
| Quadrumat Sr | | | | |
| Doyce | 360 ± 1F | 318 ± 1J | 137 ± 2h | 118 ± 2bc |
| Merlin | 331 ± 2H | 285 ± 2L | 146 ± 1X | 138 ± 1Y |
| Commercial hulled | 382 ± 1B | 281 ± 1M | 128 ± 2Z | 116 ± 2c |
| Chopin CD1 (1-pass) | | | | |
| Merlin | 386 ± 1A | 323 ± 1K | 125 ± 1Za | 119 ± 1b |
| Commercial Hulled | 386 ± 1A | 305 ± 1K | 88 ± 2e | 84 ± 2f |
| Chopin CD1 (2-pass) | | | | |
| Merlin | 380 ± 1BC | 381 ± 1B | 161 ± 1U | 224 ± 2Q |
| Commercial hulled | 386 ± 1A | 371 ± 1E | 124 ± 2a | 107 ± 2d |

[a] Mean value ± one standard deviation. Values followed by the same letter are not significantly different; capital and lowercase letters indicate significant differences ($P < 0.05$).

the results of this study identified that shorts had some of the highest levels of β-glucan with all milling methods. The β-glucan content in the break flours was lowest with the walking flow for the hulless cultivars versus the hulled barley. The lowest β-glucan in the break flour was found with the Buhler for the Doyce sample and with the Quadrumat Sr for the Merlin sample. In terms of starch concentration, the break flour streams produced the highest starch concentration with the Chopin CD1 for Merlin and commercial hulled barley and with the Quadrumat Sr for Doyce hulless barley.

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## LITERATURE CITED

AACC International. 2000. Approved Methods of the American Association of Cereal Chemists, 10th Ed. Methods 08-01, 32-32, 44-19, 46-30, 55-10, and 76-13. The Association: St. Paul, MN.

Andersson, A. A. M., Andersson, R., and Åman, P. 2000. Air classification of barley flours. Cereal Chem. 77:463-467.

ASAE. 2003a. Method for determining and expressing fineness of feed materials by sieving. ANSI/ASAE S319.3. Pages 589-592. The Society: St. Joseph, MI.

ASAE. 2003b. Moisture Measurement—Unground grain and seed. ASAE S352.2. Page 593. The Society: St. Joseph, MI.

Bhatty, R. S. 1987. Milling yield and flour quality of hulless barley. Cereal Foods World 32:268-272.

Bhatty, R. S. 1997. Milling of regular and waxy starch hull-less barleys for the production of bran and flour. Cereal Chem. 74:693-699.

Bhatty, R. S. 1999a. β-Glucan and flour yield of hull-less barley. Cereal Chem. 76:314-315.

Bhatty, R. S. 1999b. Review: The potential of hull-less barley. Cereal Chem. 76:589-599.

Izydorczyk, M. S., Dexter, R. G., Desjardins, R. G., Rossnagel, B. G., Lagasse, S. L., and Hatcher, D. W. 2003. Roller milling of Canadian hull-less barley: Optimization of roller milling conditions and composition of mill streams. Cereal Chem. 80:637-644.

Kim, Y. S., Flores, R. A., Deyoe, C. W., and Chung. O. K. 1995. Relation of physical characteristics of wheat blends and experimental milling to some commercial milling performance parameters. Appl. Eng. Agric. 11:699-706.

Kiryluk, J., Kawka, A., Gasiorowski, H., Chalcarz, A., and Aniola, J. 2000. Milling of barley to obtain β-glucan enriched products. Nahrung 44:238-241.

Moreau, R. A., Powell, M. J., and Singh, V. 2003. Pressurized liquid extraction of polar and nonpolar lipids in corn and oats with hexane, methylene chloride, isopropanol, and ethanol. J. Am. Oil Chem. Soc. 80:1063-1067.

Sundberg, B., and Åman, P. 1994. Fractionation of different types of barley by roller milling and sieving. J. Cereal Sci. 19:179-184.

Warpala, I. W. S., and Pandiella, S. S. 2000. Grist fractionation and starch modification during the milling of malt. Trans IChemE, Part C, Food Bioproducts Process. 78:85-89.

Wu, Y. V., and Doehlert, D. C. 2002. Enrichment of β-glucan in oat bran by fine grinding and air classification. Lebens. Wiss. Technol. 35:30-33.

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