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## Dietary fiber profile of barley flour as affected by extrusion cooking

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#### Abstract

Barley grains, Phoenix and CDC-Candle, were extruded in a twin-screw extruder at 90-140 °C and 20-50% moisture level. Effects of extrusion conditions on total (TDF), soluble (SDF), and insoluble dietary fiber (IDF) were determined. The content of SDF and TDF increased upon extrusion cooking of both types of barley flours. The changes in IDF content were found to be variety-dependent. Only a minor decrease in IDF content of CDC-Candle barley was found, but an increase in IDF content of Phoenix was observed at all extrusion temperatures. The increase in SDF, in both barleys, could be due to the transformation of some IDF into SDF during extrusion and the formation of additional SDF by transglycosidation. The increase in IDF in Phoenix flour could be due to the formation of retrograded amylose [resistant starch (RS3)] during extrusion cooking and subsequent cooling. © 2002 Elsevier Science Ltd. All rights reserved.

Keywords: Barley; Extrusion; Dietary fiber

#### 1. Introduction

Traditionally, the non-digestible (resistant to digestion/hydrolysis by the alimentary enzymes of humans) constituents of plant cell walls, which consist of polysaccharides (cellulose, hemi-cellulose, mucilage, oligosaccharides, pectins), lignin and associated substances, such as waxes, cutin and suberin, have been described as dietary fiber (Devries, Prosky, Li, & Cho, 1999). According to the solubility in water, total dietary fiber (TDF) can be categorized into two groups, namely soluble (SDF) and insoluble (IDF) dietary fiber. SDF and IDF have been known to play different physiological roles in human health. The enzymatic-gravimetric methods have been commonly employed in the determination of TDF, SDF and IDF in foods (Caldwell & Nelson, 1999). The present study uses the procedure defined by Megazyme International Ireland Ltd., Wicklow, Ireland to determine the content of various dietary fiber fractions in extrusion-cooked barley flour. This procedure, essentially, uses the protocols explained in AACC method 32-07 (1995) and AOAC method 991.43 (1995).

The cell wall structural polysaccharides of barley endosperm consist primarily of  $\beta$ -glucan, arabinoxylan and cellulose. The cell walls of barley endosperm contain about 75% β-glucan and 20% arabinoxylan, whereas the aleurone cell walls contain 26% β-glucan and 71% arabinoxylan (Jadhav, Lutz, Ghorpade, & Salunkhe, 1998). The minor components are cellulose, glucomannan, and  $(1 \rightarrow 3)$ - $\beta$ -glucan (Jadhav et al., 1998). During the past two decades, barley  $\beta$ -glucan has received considerable research attention due to its health benefits. Several studies have shown that barley β-glucan has significant blood cholesterol-lowering effects (Martinez, Newman, & Newman, 1992; McIntosh, Whyte, McArthur, & Nestel, 1991). β-Glucan in barley has also been known to increase the viscosity of intestinal fluid and thereby reduce the rate of sugar/ starch absorption (Anderson, Deakins, Floore, Smith, & Whites, 1990), which is beneficial in the management of diabetes (Gosain, 1996; Klopfenstein, 1988; Pick, 1994).

Extrusion cooking is a popular food-processing technique, especially for the production of fiber-rich products, such as breakfast cereals, flat breads, dextrinized or cooked flour. Due to its high content of TDF and a high proportion of SDF, an investigation into the use of barley flour in a variety of extruded products is of importance from a nutritional point of view. Extrusion

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conditions, such as temperature, moisture and pressure/ shear, may change the content as well as the physicochemical and nutritional/physiological properties of barley flour components, including dietary fiber. Little research has been done towards understanding the impact of extrusion cooking on the dietary fiber of barley flour. Østergård, Björck, and Vainionpää (1989) reported that dietary fiber content of barley increased upon extrusion cooking, accompanied by a decrease in total starch content. This is probably due to the formation of indigestible starch fragments. However, the increase in SDF did not account for the total decrease in starch. This is probably due to the newly-formed indigestible starch fragments, which are too small to precipitate with 80% ethanol employed in the dietary fiber assay (Østergard et al., 1989).

The objectives of the present study are to investigate the effect of extrusion cooking of barley flour (waxy and regular starch types), under different temperature/ moisture combinations, on the TDF, IDF, SDF,  $\beta$ -glucan and resistant starch contents of barley flour. Furthermore, an attempt will be made to rationalize the changes in various dietary fiber fractions during extrusion cooking.

#### 2. Materials and methods

### 2.1. Materials

Waxy barley grains (CDC-Candle) were obtained from Agricore, Calgary, AB. Regular barley grains (Phoenix) were obtained from Nakonechney Family Farms, Millet, AB. The analytical kits for dietary fiber and  $\beta$ -glucan were purchased from Megazyme International Ireland Ltd., Wicklow, Ireland.

# 2.2. Pearling and milling of barley grains and extrusion processing of barley flour

Pearling and milling of barley grains were carried out at the POS pilot plant, Saskatoon, SK. Grains were pearled to 30–32% in a "Satake" mill (Model-TM05, Satake, Toyko, Japan). The pearled grains were pinmilled (Alpine Contraplex wide chamber mill Type A 250, Hosokawa Micron Systems, Summit, NJ) into flour at 6000 rpm and a feed rate of 150 kg/h. The flour was extruded in a twin-screw extruder (Werner and Pfleiderer ZSK 57W 50P, Stuttgart, Germany). The barrel temperatures used in this study were 90, 100, 120 and 140 °C, while moisture contents of 20, 35 and 50% (w/ w, dry basis) were employed. The feed rate, screw speed and L/D ratio of die nozzle were set at 50 lb/h, 50 rpm and 20:1, respectively. Under each set of extruder processing conditions, the extruder was allowed to reach a steady state prior to sample collection (three times at regular intervals). Extruded samples were dried for a week in a draft oven at 75 °C, ground and screened (60 mesh). The extruder screw arrangement was set according to an orthogonal design.

#### 2.3. Chemical analysis

The contents of TDF, SDF and IDF in the native and extruded barley flour were measured by using the Megazyme total dietary fiber analysis kit (Megazyme International Ireland Ltd, Wicklow, Ireland). The contents of mixed linkage  $\beta$ -glucan, total starch and resistant starch in native and extruded barley flours were determined by using the Megazyme analysis kits. The total lipid content was measured using the procedure of Bligh and Dyer (1959). The determination of resistant starch involved a treatment step with dimethyl sulfoxide (DMSO). Standard AOAC (1990) procedures were employed to determine the total nitrogen (method 979.09) and ash (method 900.02) contents of the SDF, IDF and TDF.

The solubility of  $\beta$ -glucan in the native (at 100 and 25 °C) and extruded flours (at 25 °C) was determined as follows. Flour (100 mg) was mixed with 10 ml of distilled water and subjected to continuous shaking for 2 h in a water bath maintained at 100 or 25 °C, followed by the centrifugation at 10,000 rpm for 5 min.  $\beta$ -Glucan content in a 0.1 ml aliquot of the supernatant was determined. The solubility was calculated as % of the total  $\beta$ -glucan content in the flour.

Table 1						
<b>Composition</b> <sup>a</sup>	(%,	dry	basis)	of	barley	flour

Flour <sup>b</sup>	Starch	Protein	β-glucan	Lipid	Ash	Dietary fiber	
						SDF	IDF
CDC-Candle	$73.6 \pm 0.9$	$10.2 \pm 0.6$	$6.5 \pm 0.2$	$1.1 \pm 0.1$	$1.2 \pm 0.0$	$5.6 \pm 0.1$	$1.9 \pm 0.1$
Phoenix	$76.5 \pm 0.4$	$10.4 \pm 0.8$	$3.9 \pm 0.0$	$1.1\!\pm\!0.0$	$1.0\pm0.0$	$2.4 \pm 0.0$	$2.4 \pm 0.6$

<sup>a</sup> Values are means of three determinations±standard deviations.

<sup>b</sup> Barley flour produced by pin-milling of pearled (30-32%) grains.

#### 2.4. Statistical analysis

All experiments were carried out in triplicates. Analysis of variance of the results was performed using the General Linear Model procedure of SAS Statistical Software, Version 6 (SAS Institute, 1989). Multiple comparison of the means was performed by least significant difference (LSD) test at  $\alpha = 0.05$  level.

#### 3. Results and discussion

The proximate composition of native barley flours is shown in Table 1. There were minor differences in protein, lipid and ash contents of CDC-Candle and Phoenix flours, whereas their starch, IDF, SDF and  $\beta$ -glucan contents were considerably different. Compared to CDC-Candle, Phoenix had higher starch and IDF but

Table 2 Solubility<sup>a</sup> of  $\beta$ -glucan in the native and extruded barley flours

lower  $\beta$ -glucan and SDF contents. Furthermore, the  $\beta$ glucan contents of CDC-Candle and Phoenix (6.5 and 3.9%, respectively) were higher than SDF contents (5.6 and 2.4%, respectively). This suggests that barley  $\beta$ glucan contained water-soluble as well as insoluble fractions. The water-solubility of  $\beta$ -glucan in the native and extruded flours is given in Table 2. The solubility (determined at 100 °C) of native  $\beta$ -glucan was 79% for CDC-Candle and 57% for Phoenix, which suggests that the SDF of native flour might contain a large amount of  $\beta$ -glucan (91% in CDC-Candle and 93% in Phoenix). The  $\beta$ -glucan in extruded flours had higher solubility (determined at 25 °C) than its native counterparts (Table 2), and, at each extrusion temperature, the solubility increased with an increase in the extrusion-moisture level. The solubility differences between native CDC-Candle and Phoenix  $\beta$ -glucans may be attributed to the variety-dependent molecular variations, while the

Table 3				
The IDF, SDF and	d TDF contents <sup>a</sup>	of native and	extruded	flours

(% of total B-glucan)	The IDF, SDF a	The IDF, SDF and TDF contents <sup>a</sup> of native and extruded flours			
		IDF	SDF	TDF	
DC-Candle		(%, dry basis)	(%, dry basis)	(%, dry bas	
$41.5 \pm 0.17 (79.4 \pm 0.35)^{\circ}$	and a "				
Extruded	CDC-Candle	1 00 1 0 07	5 (2 ) 0 11	<b>5 5 3 4 6 6 6</b>	
$0/20^{d}$ $61.9 \pm 1.20$	Native	$1.89 \pm 0.07$	$5.63 \pm 0.11$	$7.52 \pm 0.09$	
$0/35$ $62.9 \pm 0.92$	Extruded				
0/50 76.4±1.41	90/20 <sup>c</sup>	$1.82 \pm 0.23$	$5.69 \pm 0.29$	$7.51 \pm 0.26$	
00/20 86.7±1.65	90/35	$1.39 \pm 0.10$	$6.63 \pm 0.24$	$9.02 \pm 0.17$	
00/35 91.6±1.32	90/50	$1.30 \pm 0.28$	$6.82 \pm 0.29$	$8.28 \pm 0.29$	
$00/50$ $94.1 \pm 0.81$	100/20	$1.53 \pm 0.43$	$6.51 \pm 0.51$	$8.04 \pm 0.47$	
20/20 89.5±1.36	100/35	$1.29 \pm 0.07$	$6.62 \pm 0.13$	$7.91 \pm 0.10$	
20/35 93.6±0.94	100/50	$1.10 \pm 0.11$	$6.59 \pm 0.02$	$7.59 \pm 0.07$	
20/50 95.3±1.26	120/20	$1.53 \pm 0.31$	$7.16 \pm 0.30$	$8.69 \pm 0.31$	
$40/20$ $74.9 \pm 1.15$	120/35	$1.44 \pm 0.07$	$6.63 \pm 0.23$	$7.79 \pm 0.15$	
40/35 84.2±1.41	120/50	$1.17 \pm 0.14$	$6.52 \pm 0.10$	$7.96 \pm 0.12$	
40/50 89.6±1.62	140/20	$1.71 \pm 0.14$	$7.16 \pm 0.34$	$8.85 \pm 0.24$	
SD (P < 0.05) 1.52	140/35	$1.68 \pm 0.15$	$6.57 \pm 0.20$	$8.36 \pm 0.18$	
Phoenix	140/50	$1.40 \pm 0.13$	$7.24 \pm 0.27$	$9.14 \pm 0.20$	
Variative $26.8 \pm 0.46 (56.7 \pm 1.29)^{\circ}$	LSD $(P < 0.05)$	0.15	0.20	0.18	
Extruded	Phoenix				
0/20 27.0±1.01	Native	$2.43 \pm 0.57$	$2.35 \pm 0.03$	$4.78 \pm 0.30$	
0/35 28.1±1.25	Extruded				
0/50 39.3±0.16	90/20	$5.56 \pm 0.24$	$3.63 \pm 0.19$	$9.19 \pm 0.22$	
$\frac{1}{20}$ 29.5±0.30	90/35	$6.42 \pm 0.23$	$3.59 \pm 0.31$	$10.01 \pm 0.27$	
00/35 32.6+1.27	90/50	$6.43 \pm 0.22$	$3.80 \pm 0.10$	$10.23 \pm 0.16$	
$00/50$ $40.0 \pm 1.58$	100/20	$5.51 \pm 0.24$	$3.46 \pm 0.24$	$8.96 \pm 0.24$	
$20/20$ $31.6\pm1.51$	100/35	$4.74 \pm 0.21$	$3.06 \pm 0.06$	$7.79 \pm 0.14$	
$\frac{20}{35}$ $\frac{338+115}{338+115}$	100/50	$5.90 \pm 0.06$	$3.45 \pm 0.24$	$9.36 \pm 0.15$	
$20/50$ $41.0 \pm 1.07$	120/20	$5.08 \pm 0.09$	$322 \pm 021$	$8.30 \pm 0.15$	
$\frac{40}{20}$ $\frac{360 \pm 0.98}{360 \pm 0.98}$	120/35	$557 \pm 042$	$341 \pm 0.15$	$883 \pm 029$	
$40/35$ $38.9\pm0.30$	120/50	$559 \pm 0.43$	$3.61 \pm 0.25$	$9.19 \pm 0.29$	
$40/50$ $41.1 \pm 1.37$	120/00	1 10 1 0 20	2.01 + 0.45	$7.20 \pm 0.31$	
71.1 ± 1.57	140/20	$419\pm0.32$	$521\pm0.45$	/ 19 + 11 19	
SD(P < 0.05) 1.20	140/20 140/35	$4.19 \pm 0.32$ $4.77 \pm 0.49$	$3.21 \pm 0.45$ $3.03 \pm 0.12$	$7.39 \pm 0.39$ $7.75 \pm 0.31$	

<sup>a</sup> Solubility determined at 25 °C. Values are means of three determinations  $\pm$  S.D.

<sup>b</sup> Barley flour produced by pin-milling of pearled (30–32%) grains. <sup>c</sup> Solubility determined at 100 °C.

<sup>d</sup> The temperature (°C)/moisture (%) used to produce extruded barley flour.

<sup>a</sup> Values are means of three determinations  $\pm$  S.D.

0.31

LSD (P < 0.05)

<sup>b</sup> Barley flour produced by pin-milling of pearled (30-32%) grains. <sup>c</sup> The temperature (°C)/moisture (%) used to produce extruded barley flour.

0.26

dry basis)

0.29

enhanced solubility of extruded  $\beta$ -glucans may be attributed to the extrusion-induced molecular alterations (Jiang and Vasanthan, 2000).

The dietary fiber (IDF, SDF and TDF) contents of the native and extruded barley flours are shown in Table 3. In general, the contents of SDF and TDF increased upon extrusion cooking of both types of barley flours. The changes in the IDF content were found to be variety-dependent. A minor decrease (compared to native flour) in IDF content of CDC-Candle barley was evident at all extrusion temperatures (Table 3). This suggests that the increase in TDF of CDC-Candle flour, upon extrusion cooking, was primarily due to the increase in SDF. This increase in SDF could be partially due to the transformation of some IDF into SDF during extrusion. In a study involving white and wholemeal wheat flour, Björck, Nyman, and Asp (1984) have reported a slight increase in TDF with a substantial shift from IDF to SDF in the extruded white wheat flour. In the present study, the % increase in SDF in extrusion-cooked CDC-Candle flour was considerably higher than the decrease observed in IDF (Table 3). suggesting the formation of additional SDF from nondietary fiber components (i.e. starch) of native flour. Theander and Westerlund (1987) reported that highly reactive anhydro-compounds (i.e. 1,6-anhydrosaccharides) were generated during extrusion cooking and these compounds would react with starch or fragmented

Table 4								
Resistant	starch	(RS3)	content <sup>a</sup>	of nat	ive and	selected	extruded	flours

Flour sample		RS3 (%, w/w)
CDC-Candle		
Native <sup>b</sup>		0
Extruded	$90/20^{c}$	0
	100/20	0
	120/20	0
	140/20	0
	90/50	0
	100/50	0
	120/50	0
	140/50	$0.58 \pm 0.20$
LSD (P < 0.05)	,	-
Phoenix		
Native		$0.83 \pm 0.15$
Extruded	90/20	$1.02 \pm 0.20$
	100/20	$1.08 \pm 0.15$
	120/20	$1.43 \pm 0.10$
	140/20	$1.10 \pm 0.21$
	90/50	$1.62 \pm 0.38$
	100/50	$1.75 \pm 0.25$
	120/50	$2.87 \pm 0.41$
	140/50	$1.94 \pm 0.33$
LSD (P<0.05)	,	0.28

<sup>a</sup> Values are means of three determinations  $\pm$  S.D.

<sup>b</sup> Barley flour produced by pin-milling of pearled (30-32%) grains.

 $^{\rm c}$  The temperature (°C)/moisture (%) used to produce extruded barley flour.

starch through transglycosidation reactions to form new branched glucans, which were resistant to amylase hydrolysis. In the present study, the formation of "indigestible glucans" might have contributed to the increase in SDF content of CDC-Candle flour.

Conversely, both IDF and SDF contributed to the increase in TDF content in extrusion-cooked Phoenix flour, with the contribution by IDF being prominent (Table 3). The substantial increase in IDF of the extruded Phoenix flour was unexpected. A number of mechanisms, have been suggested to explain the changes in dietary fiber profile (IDF, SDF and TDF) of processed grain flours. Fornalm, Soral-Smietana, Smietana, and Szpenlowski (1987) reported a decrease in the contents of cellulose and lignin in extruded flours from barley and buckwheat. The occurrence of resistant starch (RS3, retrograded amylose) in thermally-processed grains was reported by Englyst, Anderson, and Cummings (1983), Englyst and Cummings (1984), Ranhotra, Gelroth, and Leinen (1999), and Englyst, Kingman, and Cummings (1992). Englyst et al. (1983) and Englyst and Cummings (1984) reported that the resistant starch was insoluble in water and had properties similar to those of insoluble dietary fiber, but could be solubilized in 2 M KOH or dimethyl sulphoxide (DMSO). Megazyme has developed a method to quantify RS3 in food samples and this methodology uses the difference in starch content before and after DMSO treatment to account for RS3. This protocol was used in this study to quantify RS3. Although there are contradictory reports on the presence of RS3 in TDF of extruded wheat flour (Björck, Nyman, Pirkhed, & Siljeström, 1986; Siljeström, Westerlund, Björck, Holm, Asp, & Theander, 1986), the data given in Table 4 suggest that the formation of RS3 might have been responsible for the increased content of IDF and TDF in extruded Phoenix flour. Regardless of the extrusionmoisture levels (i.e. 20 and 50%), the RS3 content of extruded Phoenix flour increased up to an extrusion temperature of 120 °C and then showed a decrease at 140 °C. For all temperatures employed, the RS3 formation was observed to be higher at 50% than at 20% moisture level. The RS3 contents of both native and extruded CDC-Candle flour were low.

Forrest and Wainwright (1977) were able to extract a substance, which had a covalent association between a nitrogenous material and  $\beta$ -glucan from heated (65 °C) barley endosperm cell walls. The crude nitrogen contents of the IDF and SDF fractions (from the dietary fiber determination procedure) of native and extruded flour are shown in Table 5. The nitrogen contents of both IDF and SDF fractions from extruded flours were generally higher than those of native flours. This suggests that the extrusion cooking has induced interactions between the nitrogenous compounds (i.e. protein) in barley flour and fiber. Table 5 further suggests that

 Table 5

 Nitrogen content<sup>a</sup> of the insoluble and soluble dietary fiber

Flour sample	Insoluble dietary fiber	Soluble dietary fiber
CDC-Candle		
Native <sup>b</sup>	$1.21 \pm 0.19$	$0.62 \pm 0.16$
Extruded		
90/20 <sup>c</sup>	$1.05 \pm 0.18$	$1.27 \pm 0.11$
90/35	$1.27 \pm 0.19$	$1.00 \pm 0.27$
90/50	$1.21 \pm 0.26$	$0.89 \pm 0.23$
100/20	$1.75 \pm 0.28$	$0.87 \pm 0.06$
100/35	$1.88 \pm 0.19$	$1.03 \pm 0.08$
100/50	$2.37 \pm 0.36$	$0.85 \pm 0.05$
120/20	$1.40 \pm 0.16$	$0.60 \pm 0.19$
120/35	$1.59 \pm 0.19$	$1.13 \pm 0.11$
120/50	$2.01 \pm 0.33$	$0.86 \pm 0.12$
140/20	$0.89 \pm 0.03$	$0.61 \pm 0.08$
140/35	$1.09 \pm 0.14$	$1.14 \pm 0.18$
140/50	$1.33 \pm 0.07$	$0.70 \pm 0.05$
LSD (P < 0.05)	0.21	0.12
Phoenix		
Native <sup>a</sup>	$1.29 \pm 0.31$	$1.09 \pm 0.10$
Extruded		
90/20 <sup>b</sup>	$1.61 \pm 0.09$	$1.30 \pm 0.18$
90/35	$1.91 \pm 0.12$	$1.40 \pm 0.01$
90/50	$1.99 \pm 0.03$	$1.41 \pm 0.04$
100/20	$2.38 \pm 0.05$	$1.10 \pm 0.12$
100/35	$2.45 \pm 0.04$	$1.05 \pm 0.10$
100/50	$2.26 \pm 0.07$	$1.04 \pm 0.16$
120/20	$1.92 \pm 0.03$	$1.03 \pm 0.17$
120/35	$1.64 \pm 0.14$	$1.09 \pm 0.12$
120/50	$1.65 \pm 0.15$	$1.02 \pm 0.19$
140/20	$1.64 \pm 0.19$	$1.02 \pm 0.19$
140/35	$1.63 \pm 0.08$	$0.82 \pm 0.16$
140/50	$1.44 \pm 0.36$	$0.84 \pm 0.14$
LSD ( $P < 0.05$ )	0.14	0.15

<sup>a</sup> Values are means of three determinations $\pm$ S.D.

<sup>b</sup> Barley flour produced by pin-milling of pearled (30–32%) grains.

 $^{\rm c}$  The temperature (°C)/moisture (%) used to produce extruded barley flour.

IDF participated more in these interactions, especially at 100 °C. However, no definite correlation was established between the nitrogen content of the fiber and extrusion conditions. It should be noted that such protein-fiber interactions would not have contributed to increased dietary fiber content of the flour (Table 3) because appropriate deductions were made for protein content (calculated from the crude nitrogen content during the quantification of IDF and SDF). However, the fiber–protein complexes present in extruded barley may resist digestion by enzymes in the human intestine and pass on to the colon, along with dietary fiber. The fate of this protein–fiber complex in the colon or its benefit to human health is yet to be determined.

#### 4. Conclusions

Extrusion cooking increased the TDF of barley flours. The TDF increase in waxy-CDC-Candle barley was mainly due to an increase in SDF. For Phoenix, the increase in both IDF and SDF contributed to the increased TDF content. The change in dietary fiber profile during extrusion of barley flour may be attributed, primarily, to a shift from IDF to SDF, as well as the formation of RS3 and "enzyme resistant indigestible glucans" formed by transglycosidation.

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