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Effect of extrusion conditions on resistant starch formation from pastry wheat flour

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Abstract

Pastry wheat flour was extruded under various conditions of feed moisture (20%, 40%, and 60%) and screw speed (150, 200, and 250 rpm), at constant barrel temperature profile (40, 60, 80, 100, and 120 °C, feed port to exit die). The extruded samples were stored at 4 °C for 0, 7, or 14 days, at which times resistant starch (RS) formation was analyzed. Thermal and pasting properties of extruded samples stored for 14 days were analyzed using a differential scanning calorimeter and rapid visco analyzer (RVA), respectively. The RS content increased after extrusion compared to non-extruded pastry wheat flour. High significant positive correlations of feed moisture ($P < 0.01$) and storage period ($P < 0.05$) with RS formation were observed. The RS derived from extrusion and storage showed higher thermal stability with decreasing feed moisture and screw speed. Statistically significant differences in pasting properties were observed with feed moisture or screw speed. In particular, the setback value from RVA of the sample was significantly increased with increasing feed moisture. These results indicate that feed moisture and storage time were both important factors for the formation of RS from pastry wheat flour during extrusion.

Keywords: Resistant starch; Extrusion; Pastry wheat flour; Thermal property; Pasting property

1. Introduction

Resistant starch (RS) is starch that escapes digestion in the small intestine and may be digested in the large intestine where it is fermented by colonic microflora (Englyst, Kingman, & Cummings, 1992; Niba, 2002). The fermentation of RS in the colon by colonic bacteria, to produce short-chain fatty acids, such as butyrate, has been associated with various health benefits, including lowering colorectal cancer risk (Escarpa, Gonzalez, Manas, Garcia-Diz, & Saura-Calixto, 1996; Langkilde, Ekwall, Bjork, Asp, & Andersson, 1998; Niba, 2002; Topping & Clifton, 2001). The produced butyrate inhibits division of cancer cells and proliferation of colonic mucosal cells (Barnard & Warwick, 1993; Niba, 2002; Taylor, Steer, & Gibson, 1999; Van Munster, Tangerman, & Negengast, 1994), and inhibits potential mutagens, such as nitrosamide and hydrogen peroxide in human colon cells (Niba, 2002). RS occurs in four forms, RS_1: physically entrapped, inaccessible starch in a non-digestible matrix such as whole or partially milled seeds; RS_2: native granular starch, consisting of ungelatinized granules; RS_3: retrograded starch; RS_4: chemically modified starch (Englyst et al., 1992; Haralampu, 2000; Niba, 2002). Therefore, products containing high levels of RS might well qualify as functional foods and could be manufactured in great variety and with high palatability (Johnson & Gee, 1996).

Extrusion is a thermal processing that involves the application of high heat, high pressure, and shear forces to an uncooked mass, such as cereal foods (Riha, Hwang, Karwe, Hartman, & Ho, 1996). Extrusion cooking is an important and popular food processing technique and

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cereal grains are common ingredients in extruded products (Faraj, Vasanthan, & Hoover, 2004). Extrusion of cereal-based products has advantages over other usual processing methods because of low cost, short time, high productivity, versatility, unique product shapes, and energy savings (Faraj et al., 2004; Farouk, Pudil, Janda, & Pokoený, 2000). The extrusion process results in a number of chemical changes that occur, including gelatinization of starch molecules, cross-linking of proteins, and the generation of flavors (Riha et al., 1996). Retrogradation of gelatinized starch induces the formation of RS_3 (Faraj et al., 2004; Sievert & Pomeranz, 1989). There are a number of studies about effects of extrusion on RS formation using various starches, including wheat starch, high-amylose corn starch, corn starch, potato starch, and other cereal grains (Adamu, 2001; Faraj et al., 2004; Kim & Lee, 1998; Parchure & Kulkarni, 1997; Rabe & Sievert, 1992; Shin, Mun, & Bae, 2002; Unlu & Faller, 1998). Most of the studies on RS formation have been done on pure starch systems, while just a few researchers have studied the formation of RS during extrusion of wheat and barley flours (Faraj et al., 2004; Huth, Dongowski, Gebhardt, & Flamme, 2000; Siljestöm et al., 1986). Considering the impact it could have on commercial RS production and utilization, information on RS_3 formation from cereal flours may be important.

The objectives of the present study were to investigate the effects of extrusion processing conditions (feed moisture and screw speed) and storage time of extrudates on the production of RS_3 from pastry wheat flour, and to determine the thermal and pasting properties of the stored extrudates.

2. Materials and methods

2.1. Sample preparation

Extruded samples were prepared using pastry wheat flour (The Mennel Milling Company, Fostoria, OH, USA) and a co-rotating twin-screw extruder (Model MP19T2-25, APV Baker, Grand Rapids, MI, USA) with a 19 mm barrel diameter, a L/D ratio of 25:1, and a 3 mm diameter exit die. Screw configuration is shown in Table 1 and barrel temperature profile was maintained at 40, 60, 80, 100, and 120 °C for zones 1, 2, 3, 4, and 5 (feed port to exit die), respectively. Feed rate of the flour into the extruder was controlled at 30 g/min (dry basis) using a feeder (K-TRON K2M, and twin-screw volumetric feeder, CK-TRON, Pittman, NJ, USA), and screw speeds of the extruder were 150, 200, or 250 rpm. Total moisture content of the flour sample in the extruder was adjusted to 20%, 40%, or 60% using a water injector (Brook Crompton E2 Metripump, Hudders Field, England). Extrudates were cut (approx. 20 cm lengths), cooled to room temperature, and stored for 0, 7, or 14 days at 4 °C. The stored samples were cut (approx. 5 mm), dried at 50 °C for 16 h, and milled using a Micro-mill (Bel-Art Products, Pequannock, NJ, USA). The milled samples were stored at −20 °C prior to analysis.

Table 1
Screw configuration for twin-screw extruder

Zone	Type[a]	Flight angle (°)
I	5D Twin	–
II	3D Twin	–
	7 FP	30
III	5D Twin	–
	3D Twin	–
IV	3 FP	60
	3 RP	30
	2D Single	–
	4 FP	60
V	3 RP	30
	2D Single	–
	Die	–

[a] D, screw diameter (19 mm); P, kneading paddle (0.25 D); FP, forwarding paddle; RP, reversing paddle; Twin, twin lead screw; Single, single lead screw; Die: 3 mm single round type exit die.

2.2. Moisture, ash, and total nitrogen contents

Moisture and ash contents of pastry wheat flour and ground extrudates were measured by AACC methods (AACC, 2000). Total nitrogen content of pastry wheat flour was determined with a nitrogen and protein determinator (FP-428, Leco Co., St. Joseph, MI, USA) using 150–160 mg of each sample. All determinations were done in triplicate.

2.3. Resistant starch content

Resistant starch (RS) contents of pastry wheat flour and the extrudates were measured according to the analysis procedure provided by the Resistant Starch Assay Kit (Megazyme International Ireland Ltd. Co., Wicklow, Ireland). In brief, to each 100 mg of sample, 4 ml of mixture (pancreatic α-amylase, 10 mg/ml, and amyloglucosidase, 3 U/ml) was added and the whole incubated in a shaking water bath (1024, FOSS Tecator AB, Höganäs, Sweden) with 200 strokes/min for 16 h. After incubation, 4 ml of ethanol (99%) was added and the suspension stirred vigorously (stop reaction), and then centrifuged at 1500g for 10 min. The supernatant was removed carefully and 8 ml of 50% (v/v) ethanol was added to the residue and stirred in; this was followed again by centrifugation and removal of the supernatant. The 50% ethanol-washing step was repeated once. Two millilitre of 2 M KOH was added to the residue, with gentle stirring in an ice water bath to dissolve the residue for 20 min; at which point 1.2 M sodium acetate buffer (8 ml, pH 3.8) and amyloglucosidase (0.1 ml, 3300 U/ml) were added. Samples were incubated in the water bath (FOSS Tecator AB) at 50 °C for 30 min and centrifuged at 1500g for 10 min. To 0.1 ml of the supernatant, 3 ml of glucose oxidase-peroxidase-aminoantipyrine (GOPOD, >12,000 U/l glucose oxidase; >650 U/l peroxidase; 0.4 mM 4-aminoantipyrin) were added and the mixture was incubated in the water bath (FOSS Tecator AB) at 50 °C for 20 min. Absorbance was measured using a spectrophotometer (Spectronic Genesys 5, Spectronic Instruments Inc., Rochester, NY, USA) at 510 nm. Sodium acetate buffer (0.1 M, pH 4.5) and glucose (1 mg/ml in 0.2% benzoic acid) were used as a blank and glucose standard, respectively. The measured absorbance was calculated to % resistant starch using an equation from the kit manual. The analyses were performed in triplicate.

2.4. Thermal properties

Thermal characteristics of pastry wheat flour and extrudates were analyzed using a differential scanning calorimeter (DSC, TA 2910, TA Instruments, Newcastle, DE, USA). Samples of 4 mg were weighed into aluminium pans (TA Instruments) and 8 μl of distilled water were added using a micro-syringe. The sample pans were hermetically sealed and allowed to equilibrate overnight at room temperature. Samples were heated from 20 to 200 °C at the rate of 10 °C/min. A sealed empty pan was used as a reference. Transition onset temperature (T_o), transition peak temperature (T_p), and transition enthalphy (ΔH) were recorded and analyzed using the TA Universal Analysis Software (version 3.6, TA Instruments). All procedures were performed in triplicate.

2.5. Pasting properties

Pasting properties of pastry wheat flour and extrudates were analyzed using the Rapid Visco Analyzer (RVA Model 4, Newport Scientific Inc., Warriewood, Australia). For each sample, 3.5 g of flour was added to 25 ml (for pastry wheat flour) or 25.3 ml (for extrudates) of distilled water. The profile for analysis was Standard Method 1 according to AACC Method (AACC, 2000) and data from the RVA were processed by the software (Thermocline version 1.2, Newport Scientific Inc.). All procedures were performed in triplicate.

2.6. Statistical analysis

The experiments were designed as a 3 (injected total moisture) × 3 (screw speed) × 3 (storage period) factorial. The data were analyzed by ANOVA using SAS (version 8.2, SAS Institute Inc., Cary, NC, USA). The differences among the mean values for resistant starch content, thermal and pasting properties were processed by the Student–Newman–Keuls multiple range test, and significance was defined at $P < 0.05$. Pearson's correlation coefficients between the RS contents and extrusion conditions or storage periods were analyzed, and significance was defined at the $P < 0.05$ and <0.01 levels.

3. Results and discussion

3.1. Resistant starch formation

The contents of moisture, ash, crude protein, and RS in the pastry wheat flour used in this study were 12.56, 0.45, 8.03 and 0.38% (w/w), respectively. The RS content of extruded pastry wheat flour stored at 4 °C is shown in Table 2. Compared to the raw pastry wheat flour (0.38% RS, dry weight basis), the RS content increased from 1.3- to 7-fold right after extrusion (day 0), and increased from 3- to 11-fold during storage (7 or 14 days). The RS contents of most samples increased significantly with increases of extrusion feed moisture (FM) and storage periods ($P < 0.05$). The FM had a significant effect on RS formation during storage. Especially, when the FM was 60%, the RS was relatively higher than that of 20% or 40% FM samples. It is known that moisture acts as a plasticizer for retrogradation of starch and the retrogradation can be maximized in the range of 30–60% of moisture (Fennema, 1996; Jang & Pyun, 2004). Accordingly, extrusion under 60% FM conditions might have increased the RS formation due to the optimum moisture level for retrogradation.

The effects of screw speed, within the same FM level, were not significant right after extrusion (0 day). However, after 7 and 14 days storage, some samples produced at 250 rpm showed higher RS contents than did samples extruded at lower screw speeds. Significant correlations of RS formation were obtained with feed moisture (0.833, $P < 0.01$) and storage period (0.339, $P < 0.05$), but not with screw speed (0.018). These results were in agreement with Sievert and Pomeranz (1989) and Kim and Lee (1998), who reported that high moisture (up to 67%) resulted in the highest yield (up to 38.4%) of RS from high amylose corn starch extrusion. Unlu and Faller (1998) observed 2.13% formation of RS from a mixture of corn, potato and wheat starch by extrusion and reported a small relationship between RS formation and screw speed. Shin et al. (2002) also reported the highest RS formation yield (14.2–15.5%) from corn starch under the extrusion conditions of 110 °C barrel temperature, 150 rpm screw speed, and approximately 30% moisture content. Huth et al.

Table 2
Changes during storage periods at 4 °C in the resistant starch contents of extruded pastry wheat flour made under different extrusion conditions (unit: %, dry weight basis)

Feed moisture (%)	Screw speed (rpm)	Storage period (days)			SEM[a]
		0	7	14	
20	150	0.52by[b]	1.25fx	1.24dx	0.152
	200	0.50bz	1.35fx	1.20dy	0.156
	250	0.48by	1.21fx	1.18dx	0.150
40	150	0.65by	1.63ex	1.56cx	0.200
	200	0.63by	1.52ex	1.58cx	0.195
	250	0.67bz	1.86dx	1.71by	0.238
60	150	2.54ay	4.07bx	4.03ax	0.320
	200	2.59az	3.55cy	4.01ax	0.265
	250	2.65ay	4.25ax	3.98ax	0.314
	SEM[c]	0.231	0.291	0.300	

[a] SEM: Standard error of the means ($n = 9$).
[b] Different letters (a–f) within the same column, and different letters (x–z) within the same row differ significantly ($P < 0.05$).
[c] SEM: Standard error of the means ($n = 27$).

(2000) produced up to 6% RS from barley extrudates formed with a feed moisture content of approximately 20%, and stored at 4 or −18 °C for 7 days. In contrast, Faraj et al. (2004) reported very low RS formation (only up to 0.06%) from barley flour by extrusion, and concluded that RS in most commercially extruded cereal grain-based foods, is also present at very low levels (0–0.6%) and, additionally, that RS formation can be optimized by post-extrusion conditions, such as storage temperature and periods. Results in the present study indicated that, for pastry wheat flour, the control of extrusion conditions was important for RS formation (up to 11 times higher yield), and that high extrusion FM level and longer storage time can together maximize the formation of RS.

3.2. Thermal properties

All extrudate samples showed similar trends in thermal properties; thus only data from the extrudate samples stored for 14 days with the highest RS levels are presented (Tables 3 and 4). The T_o (onset temperature), T_p (peak temperature), and ΔH (endothermic enthalpy) of raw pastry wheat flour were 60.9 °C, 65.4 °C, and 1.78 J/g, respectively. Extrudates made under 20% FM conditions showed one endothermic transition peak, while samples produced with 40% and 60% FM contents had two endothermic transition peaks. The T_o, T_p, and ΔH of the first small endothermic transitions in samples made from 40% and 60% FM were in the ranges 44.9–46.2 °C, 50.8–53.5 °C, and 0.7–1.0 J/g, respectively (Table 3). The main (the second peak) endothermic transitions of T_o, T_p, and ΔH were in the ranges 122.8–138.2 °C, 144.7–156.6 °C, and 17.8–57.2 J/g, respectively (Table 4). In terms of the main endothermic transition, most T_o values were significantly lower in samples extruded at 200 and 250 rpm than in samples extruded at 150 rpm. Among samples extruded at 150 rpm, the 60% FM samples had significantly lower T_o values than the 20% FM samples. The T_p, in general, decreased with increasing FM and screw speed. These results indicate that extrusions under conditions of lower FM (20%) and screw speed are associated with higher thermal stability of extrudates. Enthalpy decreased with increasing screw speed and decreasing FM levels. Sievert and Pomeranz (1989, 1990) and Gruchala and Pomeranz (1993) observed endothermic transition trends similar to those in this study. Sievert and Pomeranz (1989, 1990) also reported that RS residues enzymatically isolated from seven treated starches, including wheat starch, displayed small endothermic transition between 41 and 67 °C and other prominent peaks between 120 and 177 °C; it is not clear whether the formation of this small transition was from a thermal effect derived from retrograded amylopectin or whether other factors were involved. Gruchala and Pomeranz (1993) also reported two endothermic transitions and values for thermal properties, similar to those in the present study, from wheat and RS blended samples; in their study, the RS was stable and did not interact below 100 °C. However, it is known that the endothermic transition of RS is usually seen at around 120–165 °C (Adamu, 2001; Czuchajowska, Sievert, & Pomeranz, 1991; Haralampu, 2000; Shin et al., 2002; Sievert & Pomeranz, 1989; Szczodrak & Pomeranz, 1992); thus, the second peaks described in the present study were most

Table 3
The first thermal transitions of extruded pastry wheat flour (stored for 14 days at 4 °C) by differential scanning calorimetry

	Feed moisture (%)	Screw speed (rpm)			SEM[a]
		150	200	250	
T_o (°C)	20	–	–	–	–
	40	45.4ax[b]	44.9b	45.6a	0.14
	60	45.0y	45.5	46.2	0.26
	SEM[c]	0.14	0.21	0.27	
T_p (°C)	20	–	–	–	–
	40	51.3a	50.9by	50.8by	0.09
	60	51.8b	53.1ax	53.5ax	0.33
	SEM	0.14	0.18	0.76	
ΔH (J/g)	20	–	–	–	–
	40	0.7	1.0	0.9	0.08
	60	0.9	0.9	0.9	0.02
	SEM	0.11	0.04	0.03	

T_o, onset temperature; T_p, peak temperature; ΔH, enthalpy of transition.
[a] SEM: Standard error of the means ($n = 6$).
[b] Different letter (a–b) within the same row, and different letters (x–y) within the same column differ significantly ($P < 0.05$).
[c] SEM: Standard error of the means ($n = 9$).

Table 4
The second thermal transitions of extruded pastry wheat flour (stored for 14 days at 4 °C) by differential scanning calorimetry

	Feed moisture (%)	Screw speed (rpm)			SEM[a]
		150	200	250	
T_o (°C)	20	138.2ax[b]	129.4ab	123.6b	3.12
	40	134.9axy	128.1b	125.2b	2.67
	60	135.6ay	127.2ab	122.8b	2.59
	SEM[c]	0.96	2.30	1.05	
T_p (°C)	20	156.6ax	154.6ax	150.3bx	1.25
	40	150.3ay	144.7by	148.6bxy	1.37
	60	146.8z	145.8y	146.4y	1.16
	SEM	1.07	2.11	0.88	
ΔH (J/g)	20	30.4ay	20.38by	17.8by	2.47
	40	55.2ax	21.9by	21.3bx	8.59
	60	57.2ax	33.9bx	20.9bx	6.91
	SEM	4.20	2.91	4.97	

T_o, onset temperature; T_p, peak temperature; ΔH, enthalpy of transition.
[a] SEM, standard error of the means ($n = 9$).
[b] Different letters (a–b) within the same row, and different letters (x–z) within the same column differ significantly ($P < 0.05$).
[c] SEM, standard error of the means ($n = 9$).

likely from the RS formed during extrusion and subsequent storage.

3.3. Pasting properties

The pasting properties of peak, trough, breakdown, final viscosity, and setback of raw pastry wheat flour were 3068, 1942, 1126, 3741, and 1799 cP, respectively, and those of all studied extrudates stored for 14 days are listed in Table 5. The extrudate samples for each of the storage periods (0, 7, 14 days) showed similar trends in pasting properties; therefore, samples with the highest RS levels (those stored for 14 days) were chosen for presentation in this table. The 20% FM samples showed significantly lower viscosity profile patterns than did other higher FM samples. In addition, most of the pasting values decreased with increasing screw speed. In the 40% FM samples, peak and breakdown values were significantly increased, while other pasting values (trough, final viscosity, and setback) were decreased with increasing screw speed. The 60% FM samples had significantly higher peak, trough, final viscosity, and setback values than other extruded samples. Samples made from higher FM levels and those containing higher levels of RS exhibited higher peak and final viscosity characteristics. The setback values of the 60% FM samples were significantly higher than others. The setback is known as a prediction value for retrogradation of starch in flours (Mazurs, Scoch, & Kite, 1957). Data obtained in the present study confirmed this prediction, i.e., samples with higher levels of RS exhibited higher setback values by RVA. Therefore, measuring RVA properties, especially the setback value, could give an indirect indication of how much RS has been formed via retrogradation of starch.

Table 5
Pasting properties of extruded pastry wheat flour (stored for 14 days at 4 °C) by rapid visco analyzer (unit: cP)

Pasting properties	Feed moisture (%)	Screw speed (rpm)			SEM[a]
		150	200	250	
Peak	20	2274az[b]	2071by	1756cy	95.7
	40	4941cy	5418bx	5526ax	113.9
	60	5582ax	5382bx	5452bx	38.5
	SEM[c]	340.7	401.8	487.2	
Trough	20	939ay	631bz	513cz	80.6
	40	933ay	746by	603cy	61.0
	60	2427bx	2614ax	2345cx	50.9
	SEM	314.4	406.6	377.1	
Breakdown	20	1357az	1420az	1248bz	32.8
	40	4029cx	4690bx	4908ax	167.2
	60	3160ay	2785by	3087ay	73.2
	SEM	497.8	399.8	368.3	
Final viscosity	20	1938ay	1356bz	1148cz	149.6
	40	2010ay	1529by	1327cy	128.1
	60	4923bx	5063ax	4586cx	90.0
	SEM	322.0	463.9	406.7	
Setback	20	1012az	720bz	622cz	74.3
	40	1092ay	801by	715cy	72.4
	60	2496ax	2470ax	2239bx	52.0
	SEM	304.9	360.7	331.6	

[a] SEM, standard error of the means ($n = 9$).
[b] Different letters (a–c) within the same row, and different letters (x–z) within the same column differ significantly ($P < 0.05$).
[c] SEM, standard error of the means ($n = 9$).

4. Conclusion

Feed moisture and longer storage period were the most important factors in the current study for extrusion of pastry wheat flour to produce RS_3. In application, the RS from pastry wheat flour by extrusion might be used to produce RS enriched functional cereal-foods. Studies are in progress to identify optimum extrusion conditions to maximize RS formation, and confirm whether the produced RS can survive further cooking processing.

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