

# Functionality of fruit powders in extruded corn breakfast cereals

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Received 26 October 2005; received in revised form 10 February 2006; accepted 22 February 2006

## Abstract

Consumer interest in naturally colored foods such as breakfast cereals is growing. Degermed white cornmeal, sucrose, citric acid and dehydrated fruit powder (blueberry, cranberry, Concord grape and raspberry) were mixed in 84.3%:14.3%:0.4%:1.0% proportions, then extruded in a laboratory-scale twin-screw extruder. Feed rate was 255 g/min; water was pumped at a rate of 12.5 g/min; screw speed was 175 rpm. Cooking temperature during extrusion was generally <130 °C. Samples were cut into small spheres and dried to 5% moisture. Cereals were stored at room temperature in opaque bags. The control samples were lighter and less red than the fruit cereals. Soluble phenolics and anthocyanins were higher in the fruit cereals. At three and six weeks of storage, fruit cereals had smaller levels of hexanal, as measured by gas chromatography of headspace of ground cereals. Although anthocyanins from fruit powders survive extrusion and retain some antioxidant activity, the levels used in this study may have been too low. Higher levels of fruit will increase production costs, but the expense may be offset by the more attractive and functional cereals that result.

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**Keywords:** Extrusion; Anthocyanin; Antioxidant activity; Fruit

## 1. Introduction

Anthocyanins provide attractive red and purple colors in plant foods and are also antioxidants. Wrolstad (2004) summarized issues surrounding the use of anthocyanin-rich materials as food colorants. Health benefits of anthocyanin-rich bilberries and blueberries have been reviewed (Camire, 2000), and research suggests that anthocyanins function as antioxidants in vivo. The oxygen radical absorbance capacity (ORAC) test is one measure of the antioxidant activity of a food, and blueberry ORAC values were correlated with anthocyanin content ( $r = 0.77$ ) and total phenols ( $r = 0.92$ ) (Prior et al., 1998). Cyanidin and other anthocyanins with hydroxyl groups in the 3',4' positions in the B ring and conjugation between the A and B rings are more effective antioxidants in vitro than ascorbic acid and vitamin E (Rice-Evans, Miller, Bolwell, Bramley, &

Pridham, 1995). Juices from blackberries, blueberries, cranberries, raspberries and strawberries varied in the ability to inhibit different oxidation initiated by different compounds (Wang & Jiao, 2000). Serum from rats fed a diet containing 2 g/kg cyanidin 3-*O*- $\beta$ -D-glucoside for 14 days exhibited greater resistance to ex vivo oxidation (Tsuda, Horio, & Osawa, 1998). Although it is not clear how processing affects anthocyanins and related compounds, freeze-dried cranberry powder contained flavonol glycosides that are not found in fresh cranberries (Vvedenskaya et al., 2004). The functional significance of such processing-induced changes has yet to be determined. Spray-dried wild blueberry powder had comparable in vitro antiproliferation activity on mouse liver cells as did fresh fruit, but several fractions of a freeze-dried powder were more effective (Schmidt, Erdman, & Lila, 2005). Human volunteers who consumed freeze-dried wild blueberry powder with a high-fat meal had increased serum antioxidant levels as long as four hours after the meal (Kay & Holub, 2002).

Breakfast cereals colored with natural fruit may appeal to consumers interested in healthy foods. Consumer

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demand for natural food colorants has been growing (Gerdes, 2004). The high moisture and sugar content of many fruit products can strain extruder motors, but free-flowing spray-dried powders may be easily incorporated into dry mixes. The purpose of this study was to examine the effects of dehydrated fruit powders as colorants and antioxidants in extruded white cornmeal breakfast cereals.

## 2. Materials and methods

### 2.1. Samples

#### 2.1.1. Materials

White degermed corn meal was purchased from J.R. Short Milling Co., Chicago, IL, USA. Spray-dried fruit powder was supplied by Ocean Spray Ingredient Technology Group, Lakeville-Middleboro, MA. Domino sugar® (Baltimore, MD, USA) was purchased locally. Fruit powder composition is shown in Table 1. Citric acid was purchased from Jungbunzlauer, Newton Center, MA, USA.

#### 2.1.2. Sample preparation

Duplicate batches (5000 g each) of white cornmeal (84.3%, w/w), sucrose (14.3%), citric acid (0.4%) and dehydrated fruit powder (blueberry, cranberry, Concord grape and red raspberry) (1.0%) were mixed in a Hobart mixer (model VCM-25, Hobart Corp., Troy, OH) for 10 min.

A control treatment consisting of 85.3% (w/w) cornmeal, 14.3% sucrose and 0.4% citric acid was also extruded. A Werner-Pfleiderer ZSK-30 twin screw extruder (Ramsey, NJ) was used for processing. Extrusion conditions were based on previous work (Camire, Chaovanalikit, Dougherty, & Briggs, 2002) that resulted in acceptable model breakfast cereal samples. The dry feed rate was 255 g/min; water was pumped at 12.5 g/min; screw speed was 175 rpm. The length and diameter of each screw were 963 and 30 mm, respectively. The high shear screw configuration used in the experiment, from feed end to the die, was: 504 mm of 42 mm pitch forward conveying elements, 56 mm of 28 mm pitch conveying elements, 14 mm of 28 mm narrow pitch conveying element, 260 mm of 20 mm pitch conveying elements, 7 mm of 41 mm pitch Igel (mixing) elements, 14 mm of 45°/5 disc kneading block elements at 671 mm, 20 mm of 45°/5 disc kneading block elements at 739, 813, and 883 mm, 14 mm of 45°/5 disc left-handed kneading block elements at 685 and 759 mm, and

20 mm of 20 mm pitch reverse elements. Barrel temperatures in sections from the feed end to the die were set to: 35, 45, 60, 95, 113, 163 °C. Mass temperature, torque, and die pressure were read directly from the extruder control panel. Residence time was 3.5 min as determined by the addition of food coloring and subsequent timing of the appearance and conclusion of colored extrudate. A 10 min stabilization period was allowed between extrusion runs to allow feed to equilibrate between treatments. A single 9.7 mm long by 4 mm diameter round die was used. Extrudates were cut with a single blade cutter at 937 rpm. The collets were collected onto metal screens, then dried to about 5% moisture in a Hobart DRO Series G (Hobart Corp., Troy, OH) convection air oven at 121 °C. When cooled to room temperature, samples were sealed in tri-laminate bags (Cadill Products, Paris, IL, USA) and stored at room temperature. Specific mechanical energy (SME) was calculated according to Frame's equation (1994).

### 2.2. Physical analyses

#### 2.2.1. Expansion

Cereals were lightly packed into a 1250 mL beaker, weighed, and bulk density recorded as g/mL. Six measurements were made per extrusion run. For diametric expansion, 20 pieces from each extrusion run were measured with a caliper (Monostat Mecamin type 69X1L, KWB, Switzerland). The expansion ratio was calculated as the cross-sectional diameter of an extrudate divided by the diameter of the die opening.

#### 2.2.2. Color

Ground cereals were analyzed for Hunter  $L^*a^*b^*$  color with a Hunter Labscan color meter (Hunter Associate Laboratory Inc., Reston, Virginia) with a 6 cm optical aperture. In each treatment, the reflectance measurement was obtained from the average of three readings; duplicate samples were evaluated.

### 2.3. Chemical analyses

#### 2.3.1. Moisture

Moisture was determined using Approved Method 44-15A (AACC International, 2000). Cereals were ground to pass through a 2 mm screen in a Thomas-Wiley Laboratory Mill Model 4 (Arthur H. Thomas Co., Philadelphia, PA) and were dried 16 h at 105 °C.

#### 2.3.2. Total anthocyanin content

Cereals were ground with a pestle and mortar to pass through an US # 30 sieve. Fruit powders required no further preparation before extraction. The samples were extracted twice for 2 h with 10 mL methanol (GR Anhydrous, EM Sciences, Gibbstown, NJ, USA) containing 1% HCl with constant stirring. The vessels were wrapped in aluminum foil to minimize anthocyanin degradation due to light. Supernatants were combined and roto-evapo-

Table 1  
Characteristics of fruit powders

Fruit powder	Anthocyanins <sup>a</sup> (mg/100 g)	Ascorbic acid <sup>b</sup> (mg/100 g)	Fruit solids <sup>b</sup> (%)
Blueberry	465.0	0	80
Concord grape	234.3	0	76
Cranberry	327.1	4.9	90
Raspberry	173.6	9.0	77

<sup>a</sup> Mean of three determinations.

<sup>b</sup> Information provided by manufacturer.

rated at 40 °C to approximately 1 mL, then 10 mL distilled water was added and the solution was filtered through a PTFE 0.045 Fm cartridge (Fisher Scientific International Inc., Hampton, NH, USA). The sample solution containing the anthocyanins was loaded onto a Sep-Pak C-18 cartridge (Waters Corp., Milford, MA, USA). Anthocyanins were eluted with 1 mL methanol and brought up to 2 mL with methanol, then analyzed by the pH differential method as described by Wrolstad (1976). Individual anthocyanins were not identified.

### 2.3.3. Soluble phenolics

Procedures were adapted from Velioğlu, Mazza, Gao, and Oomah (1998). Samples (10 g) were ground to pass through a 2 mm screen in a Thomas-Wiley Laboratory Mill Model 4. Four hundred milligrams were extracted for 2 h with 5 mL 80% methanol (v/v) containing 1% HCl with constant stirring at room temperature. Samples were centrifuged (Beckman TJ-6R, TA 10 rotor, Beckman Instruments, Palo Alto, CA, USA) at 1000g for 10 min. The supernatant was decanted into 4 mL vials. The pellet was re-extracted under the same conditions. Supernatants were combined. Two hundred microliters of the extract were mixed with 1.5 mL of Folin–Ciocalteu (Sigma, St. Louis, MO, USA) reagent (diluted 1:10 with distilled water) and allowed to stand at room temperature for 5 min, then 1.5 mL of sodium bicarbonate (T.J. Baker Chemical Co., Phillipsburg, NJ, USA) solution (6 g/100 mL) was added. After 90 min at room temperature, the absorbance at 725 nm was read on a Spectronic 20D+ (Spectronic Instruments Inc., Rochester, NY). A standard curve containing 0, 0.1, 1.0, 10 and 100 µg of ferulic acid (Sigma) was used to determine the regression equation. A fresh standard curve was run each day.

### 2.3.4. Headspace analysis

Collets were ground with a pestle and mortar to pass an US # 30 sieve and 10 g were weighed into 22 mL vials with Teflon caps. Six replicates of each treatment were analyzed at three weeks intervals. The procedures used were adapted from Frankel, Hu, and Tappel (1989) and Frankel (1993). A HP 6890 gas chromatograph with Chem Station software (Agilent Technologies, Palo Alto, CA, USA) was equipped with a RESTEK® Stabilwax (Bellefonte, PA, USA) 30 m, 0.32 mm ID, 1.0 µm df column. The oven initial temperature was 50 °C, the initial heating time was 2 min and ramp temperature was 5 °C per min to 65 °C. A split injector (split ratio 100:1) was used and heated to 180 °C. The carrier gas was helium, and a flame ionization detector set to 180 °C was used. A Tekmar-Dohrmann Model 7050 (Cincinnati, OH, USA) headspace analyzer was connected to the GC. A 1 mL sample loop was used, and the platen temperature was 100 °C, with an equilibration time of 10 min. The sample loop temperature was 65 °C.

Hexanal standards were prepared by making a 500 µM hexanal (Sigma) stock solution (50.0 mg hexanal/L) with

distilled water. The stock solution was diluted with distilled water to give concentrations of 50, 25, 12.5 and 6.25 µM. Five milliliters of each of the standards were placed in 22 mL vials and sealed with Teflon caps. The peak area of the standards was used to calculate a regression equation (SYSTAT, Ver. 9). Fresh standards were prepared for each analysis run.

The retention times for pentane (1.48 min), octanal (2.02), nonanal (2.72 min), pentanal (6.13 min) and hexanal (9.90 min) were determined by adding 50 mg of each compound to a vial and analyzing as described.

### 2.3.5. Antioxidant activity

Five grams of cereals were ground with a pestle and mortar to pass through a US # 30 sieve. One gram was extracted with methanol for 1 h at room temperature with constant stirring. Samples were centrifuged (TJ-6R, TA 10 rotor) at 1000g for 10 min. The supernatant was decanted into 4 mL vials. The pellet was re-extracted under the same conditions. Supernatants were combined. A Total Antioxidant Status Kit (Randox Laboratories, San Francisco, CA, USA) was used to measure the ABTS cation assay according to the method of Re et al. (1999).

## 2.4. Statistical analyses

All data were analyzed by the general linear model hypothesis program (SYSTAT 9.0). Most analyses were analyzed with the treatment (type of fruit or control) as the independent factor. For the headspace data, each treatment at each week was compared to the other treatments at the same time period. Fisher's LSD test was used to separate means at  $P \leq 0.05$ .

## 3. Results and discussion

### 3.1. Extrusion

There were no significant differences among treatments for cook temperature (about 128 °C), melt temperature (170 °C), torque (41%) and die pressure (0.96 MPa) (data not shown). SME was an average of 304 kJ/kg, with no significant differences among treatments. Extrusion was steady, with no surging or other problems. Pieces were somewhat sticky as they were cut at the extruder die face.

### 3.2. Physical properties

Diametric expansion ranged 190–193%, and the average bulk density was 0.2 g/mL. Both characteristics were similar among treatments (data not shown). Extruded corn cereals containing blueberry or grape juice concentrates had lower bulk density than a control product that was sweetened with corn syrup instead of juice (Camire et al., 2002). The control cereal was lighter and less red than the fruit cereals (Table 2). Blueberry cereal was darker than all other samples. Hunter 'a' values varied in the following

Table 2  
Hunter color of extruded white cornmeal cereals containing fruit powder<sup>a</sup>

Treatment	<i>L</i>	<i>a</i>	<i>b</i>
Blueberry	70.897 ± 0.708a	5.822 ± 0.150c	6.993 ± 0.660a
Concord grape	76.047 ± 0.480c	3.652 ± 0.129b	10.133 ± 0.794b
Control	81.715 ± 0.547d	0.343 ± 0.618a	12.215 ± 1.196c
Cranberry	73.858 ± 0.776b	6.545 ± 0.092d	10.483 ± 0.527b
Red raspberry	73.208 ± 0.776b	7.453 ± 0.397e	10.103 ± 0.226b

<sup>a</sup> Mean of six values. Values within columns followed by different letters are significantly different (Fisher's LSD test,  $P < 0.05$ ). Hunter color scales are *L*0, black; 100, white; +*a*, red; −*a*, green; +*b*, yellow; −*b*, blue.

order: red raspberry > cranberry > blueberry > Concord grape > control. Hunter '*b*' was highest in the control and lowest (more blue) in the blueberry.

### 3.3. Chemical composition

Blueberry cereal moisture content was higher than that of the Concord grape and raspberry samples (Table 3). The control cereal also had more moisture than did the grape cereal. Soluble phenolics, as ferulic acid equivalents (FAE), were higher in blueberry and cranberry cereals than in the control sample. Although phenolics were not measured in the individual ingredients and mixtures prior to extrusion, significant loss of phenolics occurred during extrusion of oat cereals (Viscidi, Dougherty, Briggs, & Camire, 2004).

Anthocyanins are heat-labile, therefore some loss of the pigments was expected during extrusion. Concord grape and red raspberry were similar in anthocyanin content, but cranberry contained more anthocyanins than did the grape (Table 3). Blueberry cereal had the highest concentration of anthocyanins. Considering the anthocyanin content of the fruit powders (Table 1) used at a level of 1% of the dry ingredients, there is an apparent loss of about 90% of the pigments in all fruit except raspberry. These losses are comparable to those reported for corn cereals with added blueberry juice concentrate (Camire et al., 2002; Chaovanalikit, Dougherty, Camire, & Briggs, 2003). The minimal amount of anthocyanins recovered from the control sample is indicative of some contamination with fruit powder during processing or analysis, but most replicates of that treatment were negative for anthocyanin content.

Table 3  
Moisture (%), total phenolics (ppm) and anthocyanins (mg/100 g) in extruded white cornmeal cereals with fruit powder<sup>a</sup>

Treatment	Moisture ( <i>n</i> = 6)	Phenolics ( <i>n</i> = 6)	Anthocyanins ( <i>n</i> = 6)
Blueberry	5.20 ± 0.91b	138.5 ± 16.0b	0.46 ± 0.12d
Concord grape	4.03 ± 0.37a	118.4 ± 21.8a,b	0.21 ± 0.05b
Control	5.03 ± 1.26b	102.6 ± 12.4a	0.03 ± 0.05a
Cranberry	4.43 ± 0.10a,b	132.6 ± 15.7b	0.36 ± 0.07c
Red raspberry	4.29 ± 0.22a	124.2 ± 17.7a,b	0.29 ± 0.09b,c

<sup>a</sup> Values within columns followed by different letters are significantly different (Fisher's LSD test,  $P < 0.05$ ).

### 3.4. Antioxidant activity

Antioxidant activity as measured by the ABTS method was highest in the control and cranberry samples; other fruit cereals were not different from each other (62–64% inhibition) (Table 4). Antioxidant activity was not significantly correlated with either anthocyanin or phenolic content (Table 6). One possible explanation for this finding is that Maillard browning during extrusion and/or storage was suppressed by the fruit powders, thus reducing one source of antioxidants. The antioxidants in the cranberry powder may have been more stable to heat and shear than those in the other fruits. The low lipid content of the corn cereals was not suited to typical measures of lipid oxidation such as peroxide value, because large quantities of material would be needed to obtain sufficient free lipid for analysis. Headspace volatiles were monitored to assess oxidation during storage. Technical problems prevented us from collecting headspace data immediately after extrusion, but realistically few consumers eat breakfast cereals less than three weeks post-processing due to shipping and other handling.

The amount of pentane in the control sample headspace was significantly greater in week 11 than values for cranberry and red raspberry (Table 5). Pentanal was highest in the control sample throughout the study, and levels rose in blueberry cereals to a comparable level. At week 11 of storage, hexanal in the headspace was significantly less in Concord grape and red raspberry than blueberry; and Concord grape and red raspberry were significantly less than the control cereal (Table 5). The octanal present in week 3 was significantly greater in red raspberry than in cranberry, Concord grape and control. At week 11 blueberry octanal content was greater than Concord grape and control; red raspberry was greater than control and Concord grape. The nonanal in the headspace of the control at week 3 of storage was significantly greater than for Concord grape. At week 11 control and blueberry cereals had significantly greater nonanal levels than did Concord grape (Table 5). Decanal values were lowest in the control and raspberry samples. Total volatiles during the sixth week of storage in the headspace of red raspberry and Concord grape cereals were significantly lower than that for blueberry. At week 11, the trend continued and Concord grape was less than red raspberry. Since some of these volatile

Table 4  
Antioxidant activity as measured by ABTS ( $\mu\text{M}$  Trolox equivalents/g sample) in extruded cornmeal cereals containing fruit powder<sup>a</sup>

Treatment	Antioxidant activity ( <i>n</i> = 6)
Blueberry	24 ± 4b,c
Concord grape	19 ± 3a
Control	28 ± d
Cranberry	27 ± 3c,d
Red raspberry	21 ± 2a,b

<sup>a</sup> Values within columns followed by different letters are significantly different (Fisher's LSD test,  $P < 0.05$ ).

Table 5  
Headspace volatile compounds from extruded corn cereals containing fruit powders stored at 25 °C<sup>a</sup>

Treatment	Week	Pentane	Pentanal	Hexanal	Octanal	Nonanal	Decanal	Total volatiles
Blueberry	3	4.38a,b	5.23a,b	10.60a,b	29.95d	28.94c,d	44.22e	160.05d
	6	4.35a,b	5.96b,c	11.43a,b	25.18c,d	24.14b	35.42d	145.65d
	11	4.56b,c	8.51c	18.33c	27.40c,d	29.86d	38.60d,e	163.02d
Concord grape	3	3.20a	3.28a,b	5.34a	25.37c,d	14.02a,b	8.77b	96.49b,c
	6	3.07a	3.15a,b	6.45a	23.52c,d	13.63a,b	8.11a,b	73.66a,b
	11	2.43a	2.63a	4.69a	13.72a	8.75a	4.64a,b	44.33a
Control	3	5.40c	6.29c	16.68c	23.82c,d	32.20d	2.26a	133.59c
	6	5.35c	6.67c	15.98b,c	21.06b,c	30.36d	1.81a	116.96c
	11	5.95c	8.19c	15.99b,c	16.73a,b	29.17c,d	1.82a	97.84b,c
Cranberry	3	3.76a,b	4.76a,b	7.84a,b	24.87c,d	17.88a,b,c	23.27c	131.86c,d
	6	3.67a,b	4.87a,b	8.64a,b	22.95b,c	16.82a,b,c	19.54c	99.64b,c
	11	3.63a,b	5.46a,b	11.40a,b	21.56b,c	17.58a,b,c	20.37c	107.17b,c
Raspberry	3	3.22a,b	3.43a,b	6.11a	37.32e	16.36a,b	2.28a	105.97b,c
	6	2.67a	2.87a,b	5.60a	27.14c,d	12.81a,b	1.88a	67.72a,b
	11	2.67a	2.88a,b	5.96a	24.88c,d	12.51a,b	1.85a	67.88a,b

<sup>a</sup> Means of six values. Values followed by different letters within columns are significantly different ( $P < 0.05$ , Fisher's LSD test).

Table 6  
Pearson correlation matrix for selected physical and chemical properties of extruded cornmeal cereals containing fruit powder

Property	Bulk density	Hunter <i>L</i>	Hunter <i>a</i>	Hunter <i>b</i>	Anthocyanins	Phenolics	Antioxidant activity
Bulk density	1.000						
Hunter <i>L</i>	0.273	1.000					
Hunter <i>a</i>	-0.258	-0.884 <sup>a</sup>	1.000				
Hunter <i>b</i>	0.073	0.776 <sup>a</sup>	-0.488	1.000			
Anthocyanins	-0.154	-0.850 <sup>a</sup>	0.717 <sup>a</sup>	-0.714 <sup>a</sup>	1.000		
Phenolics	-0.519	-0.578 <sup>a</sup>	0.558 <sup>a</sup>	-0.508	0.503	1.000	
Antioxidant activity	0.277	0.316	-0.261	0.250	-0.049	-0.187	1.000

<sup>a</sup> Correlation coefficients have a Bonferroni probability  $\leq 0.05$ .

compounds also contribute to fruit aroma, they may not be the most sensitive measures of lipid oxidation in cereals containing fruit products. Total volatiles were higher than those found for extruded yellow corn containing added antioxidant-rich food ingredients (Camire, Dougherty, & Briggs, 2005).

Whole yellow corn is a good source of phenolic compounds that may serve as antioxidants in processed foods (Adom & Liu, 2002). Milling may remove some endogenous antioxidants such as phenolics (Zielinski & Kozłowska, 2000) and thus addition of other antioxidants to corn should improve shelf-life. The antioxidant status of white corn has not been reported in the literature, but Awika, Rooney, Wu, Prior, and Cisneros-Zevallos (2003) reported that white sorghum had lower antioxidant activity than did brown and black sorghums.

#### 4. Conclusions

Although anthocyanins from fruit powders survived extrusion and retained some antioxidant activity, we concluded the levels used in this study were too low (1% w/w). Anthocyanin and phenolic levels were lower in the final cereal products than has been reported in previous work; residual levels should be 10-fold or more. Higher levels of

fruit powder will increase production costs, but the expense may be off set by the more attractive and functional cereals that result. Methods to increase retention of these pigments during processing are needed. Additional research is necessary to determine optimal levels of fruit powders for extruded cereals that increase antioxidant activity while providing acceptable sensory quality. Regulatory approval of purified anthocyanin products, as suggested by Wrolstad (2004), would provide food manufacturers with more concentrated sources of color and antioxidants compared with the powders tested in this study.

#### Acknowledgments

We gratefully acknowledge the assistance of Laurel Regan in the laboratory. We thank Denise I. Skonberg, Michael G. Murphy, Beth L. Calder and the staff at the Natick Soldier Center for their assistance during extrusion.

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