

Effect of Extrusion Process Parameters on the Quality of Buckwheat Flour Mixes¹

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ABSTRACT

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A response surface analysis using a second-order central composite design was used to study the effect of extrusion process parameters on the extrudate quality of three blends containing buckwheat flour. The extrudates were prepared as three blends. Blend 1 was a 55:40:5 (w/w) mix of light buckwheat flour, wheat flour, and nonfat dry milk (NFDm). Blend 2 was a 40:55:5 mix of light buckwheat flour, corn meal, and NFDm. Blend 3 was a 30:60:10 mix of light buckwheat flour, corn meal, and NFDm. The blends were processed in a twin-screw extruder with factorial combinations of the parameters including: process temperatures of 95–150°C, dough moisture of 15–22%, and screw speeds of 260–390 rpm. The linear components alone significantly explained most of the variation of expansion index, bulk density, water absorption, and break-

ing strength. The greatest amount of variability was explained by process temperature for blend 1. Dough moisture accounted for the greatest amount of variation for blends 2 and 3. Maximum predicted expansion index values and high water absorption percentages were obtained at low dough moisture levels. Dough moisture and process temperatures were the most important factors predicting bulk density. Sensory evaluation of texture, color, flavor, and general acceptability scores of selected samples ranked blend 3 > blend 2 > blend 1. The *in vitro* protein digestibility values ranked blend 1 > blend 2 > blend 3. An increase of up to 9.5% units in the protein digestibility values was observed when compared to the nonextruded raw blends.

The nutritional and organoleptic properties of buckwheat (*Fagopyrum esculentum* Moench) make this grain a suitable candidate for enhanced processing and marketing opportunities by the food industry. About 3 million hectares of buckwheat are harvested annually throughout the world (Opperer 1985). The leading buckwheat producers include Russia, China, Japan, Poland, Canada, Brazil, and the United States (Chung and Pomeranz 1985).

Buckwheat has been grown in North America since colonial days (Pomeranz 1983). Today, buckwheat is a minor grain crop in the United States. During the 1980s, Minnesota and North Dakota became the main buckwheat-producing states (Robinson 1980, Edwardson 1991). About two-thirds of the buckwheat grain produced in the United States (25–35 million pounds/year) is estimated to be used for feeding livestock and poultry, and the remainder is milled into flour (Chung and Pomeranz 1985; S. Edwardson, *personal communication*). Per capita consumption of buckwheat in the United States is <0.1 lb, compared with well over 100 lb for wheat, 56 lb for corn, and 8 lb for rice (Chung and Pomeranz 1985).

Interest in buckwheat as a diversified and “natural” food has increased (Fornal 1985, Javornik 1986). The proteins in buckwheat are one of the best known sources of high biological value (BV) in the plant kingdom, with 92.3% of the BV of nonfat dried milk, and 81.5% of the BV of dried whole egg (Sure 1955). Amino acid composition of buckwheat is well-balanced and nutritionally superior to that of true cereal grains (Pomeranz and Robbins 1972). Buckwheat proteins have a higher percentage of lysine (6.1%) than the cereal grain crops (2.4–4.0%) (Eggum et al 1981, Marshall and Pomeranz 1982). Due to the high content of crude fiber and tannins, the true protein digestibility in whole buckwheat grain is relatively low, <80% (Eggum et al 1981, Ikeda et al 1986). However, dehulling buckwheat grain increased true digestibility to 89% (Javornik 1986). Buckwheat proteins are deficient in methi-

onine, but when mixed with corn proteins or casein, their biological value increases (Stahl 1970). Trypsin inhibitors are present in buckwheat grain, and their activity disappears during seed germination (Ikeda et al 1984). Ikeda et al (1983) reported the presence of trypsin and chymotrypsin inhibitors in three forms, two of which were thermostable and one which was thermolabile.

Buckwheat flour in the United States and Europe is mixed with cereal flours and used for pancakes, biscuits, bread, noodles, spaghetti, macaroni, and ready-to-eat breakfast cereals (Joshi and Rana 1995). Haber (1980) substituted up to 50% of wheat flour with buckwheat flour in breadmaking. Bread baked with up to 25% of buckwheat flour, with a formulation that included vital gluten, whey, or sour milk, was acceptable.

In Japan, buckwheat flour is used for making *soba* noodles from 100% buckwheat or a mixture of 10–50% wheat flour. Some buckwheat grain is utilized as roasted groats (*kasha*), particularly in Eastern European countries. Groats and farina made from groats are used for breakfast food and porridge and as thickening agents (Marshall and Pomeranz 1982).

Extrusion cooking is a versatile process that improves organoleptic and nutritional qualities in foods (O'Connor 1987). The cost-to-benefit ratio of extrusion technology gives producers, processors, and consumers more choices by increasing the variety of ingredients used in cereal-based products, including buckwheat.

Smietana et al (1985, 1988) reported the influence of extrusion temperature on the chemical and physicochemical properties of starches and formation of starch-protein and starch-lipid complexes in extruded mixtures containing buckwheat flour, buckwheat, barley, and corn starches, and 25% milk proteins. Starch-protein complexes formed during extrusion play an important role in the stability of the porous structure in the extruded products containing buckwheat starch.

Kozikowski et al (1989) studied the influence of the extrusion process (single-screw type) on the nutritive value of proteins from buckwheat flour. Extrusion at 100 and 120°C led to a slight, but statistically significant ($P < 0.05$), increase in apparent and true digestibility of buckwheat protein.

Papotto et al (1990) described the production of corn-buckwheat flakes by extrusion cooking. Formulations with addition of 18, 22, and 30% buckwheat flour were used. The addition of 30% buckwheat flour gave the best organoleptic properties.

The objective of this study was to assess the effects of temperature, dough moisture, and screw speed on the quality of buckwheat-based mixtures produced with a twin-screw extruder using a response surface design.

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MATERIALS AND METHODS

Raw Material

Light buckwheat flour was obtained from Minn-Dak Growers Ltd. (Grand Forks, ND). Corn meal #35 was obtained from Illinois Cereal Mills, Inc. (Paris, IL). Wheat flour (Dakota Champion) was obtained from the North Dakota Mill (Grand Forks, ND). Grade A nonfat dry milk (NFDm) was obtained from Cass-Clay Creamery Inc. (Fargo, ND).

Blends for Extrusion

Preliminary experimental extrusion trials were conducted with mixtures including 30–100% light buckwheat flour, 25–60% corn meal, 40–60% wheat flour, and 5–10% NFDm. The three main selection criteria used in the preliminary trials were expansion index, bulk density, and sensory properties. NFDm was added to improve the nutritional quality and flavor as well as the functionality of the blends, as reported by Kozikowski et al (1989). Three blends were selected: blend 1, 55:40:5 (w/w) mixture of light buckwheat flour, wheat flour, and NFDm; blend 2, 40:55:5 mixture of light buckwheat flour, corn meal, and NFDm; blend 3, 30:60:10 light buckwheat flour, corn meal, and NFDm. Before extrusion, the blends were mixed for 20 min in a twin-shell dry blender (Patterson-Kelley Co., East Stroudsburg, PA).

Extrusion

A corotating, intermeshing twin-screw extruder (model TX-52 Wenger Mfg., Sabetha, KS) was used. The extruder was equipped with a volumetric feeder calibrated to deliver ≈90 kg/hr of raw material. The water added to the process was injected into the extruder barrel through a positive displacement pump (Bran-Lubbe, Wheeling, IL). The extruder barrel configuration consisted of six head sections with an extruder profile for expanded breakfast cereals and snacks. The L/D ratio for the trials was ≈15:1. The initial 2.5 head sections of the profile consisted of 52-mm dia. conveying and kneading screws; the next 2.5 head sections consisted of a combination of 45° forward-pitched lobe-shaped shearlocks and 90° oriented shearlocks and conveying and kneading elements. The final head element was conical-shaped and had “cut” flights for more effective mechanical energy development. Temperatures were recorded for all head sections (except the inlet head) by thermocouples that were machined into the extruder head sections and were flush with the interior of the extruder barrel.

The die design consisted of two separate dies to increase the head pressure before the die and to allow a more uniform flow. The secondary or backup die had a Y-shaped channel. At the en-

trance of this initial die, there were two openings of ≈6.7 mm, positioned in front of the center of each extruder shaft. The exit of the secondary or backup die had one opening of ≈8.7 mm dia. in the center. The final die had three exit ports of ≈4 mm dia.

The extruder operation conditions were selected from factorial combinations of the parameters: process temperature (95–150°C), dough moisture (15–22%), and screw speed (260–390 rpm). The extrusion parameters and their levels in the experimental design are presented in Table I. After extrusion, material was dried in a batch dryer (Standard Industries, Fargo, ND) with forced air at 50°C for 18 hr. Extruded material was cooled to room temperature and stored at 20°C in plastic bags for further analysis.

Dough Moisture and Protein Content

Dough moisture, protein ($N \times 6.25$), crude fat, and ash content were determined according to Approved Methods (AACC 1995).

Expansion Index

The diameters of extrudates were measured with a caliper. Diametral expansion index was calculated by dividing the diameter of extrudate by the diameter of die nozzle. Each value was an average of 10 readings.

Bulk Density

Bulk density of the extrudates was determined with a plastic cube of 3375 cm³ volume. The cube was filled with the extruded material by gravity from a height of 20 cm until overfilled. Contents of the cube were leveled off without compressing the extrudates by using a plastic sheet and sliding it over the rim of the cube. The weight of the cube and contents was recorded and bulk density was calculated (g/cm³). Each value was an average of three independent measurements. Bulk density was expressed on a dry basis.

Water Absorption

Water absorption (WA), defined as the weight of gel obtained per gram of dry sample at room temperature, was determined in raw material and extruded blends based on the method described by Rutkowski and Kozłowska (1982). Each sample was analyzed in triplicate.

Breaking Strength

The force required to shear the extrudate was recorded with an universal testing machine (model 1000, Instron Corp., Canton, MA). Capacity of load cell was 50 kg, range of forces was 0–5 kg, cross-head speed was 2 cm/min. Extrudates were subjected to a snap test by a custom-made single plastic blade, and peak force

TABLE I
Central Composite Design Used in Extrusion Cooking of Blends (B1, B2, B3)^a

| Treatment | | Process Temperature (°C) | | | Dough Moisture (%) | | | Screw Speed (rpm) | | |
|-----------|------|--------------------------|-----|-----|--------------------|----|----|-------------------|-----|-----|
| B1 | B2,3 | B1 | B2 | B3 | B1 | B2 | B3 | B1 | B2 | B3 |
| 3 | 3 | 105 | 120 | 110 | 19 | 16 | 16 | 280 | 330 | 280 |
| 2 | 2 | 105 | 120 | 110 | 19 | 16 | 16 | 320 | 370 | 320 |
| 4 | 4 | 105 | 120 | 110 | 21 | 18 | 18 | 280 | 330 | 280 |
| 5 | 5 | 105 | 120 | 110 | 21 | 18 | 18 | 320 | 370 | 320 |
| 13 | 13 | 125 | 140 | 130 | 19 | 16 | 16 | 280 | 330 | 280 |
| 12 | 12 | 125 | 140 | 130 | 19 | 16 | 16 | 320 | 370 | 320 |
| 14 | 14 | 125 | 140 | 130 | 21 | 18 | 18 | 280 | 330 | 280 |
| 15 | 15 | 125 | 140 | 130 | 21 | 18 | 18 | 320 | 370 | 320 |
| 6 | 6 | 115 | 130 | 120 | 20 | 17 | 17 | 300 | 350 | 300 |
| 9 | 9 | 115 | 130 | 120 | 20 | 17 | 17 | 300 | 350 | 300 |
| 1 | 1 | 95 | 110 | 100 | 20 | 17 | 17 | 300 | 350 | 300 |
| 16 | 16 | 135 | 150 | 140 | 20 | 17 | 17 | 300 | 350 | 300 |
| 11 | 7 | 115 | 130 | 120 | 18 | 15 | 15 | 300 | 350 | 300 |
| 10 | 8 | 115 | 130 | 120 | 22 | 19 | 19 | 300 | 350 | 300 |
| 7 | 10 | 115 | 130 | 120 | 20 | 17 | 17 | 260 | 310 | 260 |
| 8 | 11 | 115 | 130 | 120 | 20 | 17 | 17 | 340 | 390 | 340 |

^a Levels used for each design variable in original units.

was measured. Adjusted extrudate breaking strength (N/cm²) was calculated by dividing the shear force by the total cross-sectional area of the extrudate sheared. Each value was an average of 12 independent measurements.

Color

Samples were ground in a Brinkmann Retch Mill type ZM1 (GmbH & Co. KG, Haan, Germany) with a 0.5-mm screen. Color was measured with a color difference meter (model CR-310, Mi-

olta, Japan). Lightness (*L*), redness (*a*), and yellowness (*b*) values were recorded. Each value was an average of three independent measurements.

Protein Digestibility

The in vitro protein digestibility was measured for extruded and nonextruded samples using the method described by Satterlee and Kendrick (1980) and AOAC (1990). Each sample was analyzed in duplicate.

Sensory Evaluation

The hedonic scaling method coupled to a verbal concept nine-point scale (9 = like extremely, 1 = dislike extremely) was used to estimate quality (color, flavor, texture, and general acceptability) of the extruded products (Larmond 1977). Three treatments per blend with the best product characteristics (expansion index, bulk density, water absorption, and shear stress) were selected for sensory evaluation. A taste panel consisted of 23 untrained panelists, male (14) and female (9) students, and staff members of the Department of Cereal Science at North Dakota State University. The age range of the panelists was 28–46 years. The panelists received three samples from the same blend at the time, in a random order. The procedure test was duplicated on different days.

TABLE II
Proximate Analysis of Raw Material^a

| Sample | Moisture (%) | Protein (%) ^b | Oil (%) ^c | Ash (%) |
|--------------------------|--------------|--------------------------|----------------------|-----------------|
| Light buckwheat flour | 11.02 | 10.03 | 2.02 | 1.64 |
| Corn meal | 11.43 | 8.37 | 1.02 | 0.60 |
| Wheat flour ^d | 11.81 | 13.21 | 0.93 | 0.47 |
| Nonfat dry milk | 3.04 | 35.82 | 0.42 | nd ^e |

^a All data expressed on as-is basis.

^b Buckwheat and corn meal (N × 6.25), wheat flour (N × 5.70), nonfat dry milk (N × 6.38).

^c Crude fat.

^d Hard red spring wheat flour.

^e Not determined.

TABLE III
Summary of Models Fit to the Expansion Index (EI), Bulk Density (BD), Water Absorption (WA), and Breaking Strength (BS) Quality Parameters of Buckwheat Flour Blend Extrudates^a

| Response | Model ^b | R ² | Adj R ² | R ² Pred | PRESS Residual | SS | |
|----------|--------------------|----------------------|--------------------|---------------------|----------------|----------|----------|
| EI | Blend 1 | 1st order | 0.8303 | 0.7913 | 0.6772 | 0.477097 | |
| | | 2nd order | 0.9573 | 0.8933 | 0.7008 | 0.442197 | |
| | | *M,S,T,MT,TT | 0.9497 | 0.9245 | 0.8116 | 0.278397 | 1.478027 |
| EI | Blend 2 | 1st order | 0.5875 | 0.4844 | 0.1892 | 0.287343 | |
| | | 2nd order | 0.8870 | 0.7174 | 0.0723 | 0.328768 | |
| | | *M,T,TT | 0.7373 | 0.6717 | 0.4553 | 0.193044 | |
| EI | Blend 3 | M,S,T,MM,SS,TT,MS | 0.8623 | 0.7419 | 0.2499 | 0.265843 | 0.354395 |
| | | 1st order | 0.8644 | 0.8306 | 0.7533 | 0.088729 | |
| | | *2nd order | 0.9899 | 0.9748 | 0.9339 | 0.023775 | |
| BD | Blend 1 | M,S,T,MM,SS,TT,MS,ST | 0.9891 | 0.9765 | 0.9413 | 0.021102 | 0.359666 |
| | | 1st order | 0.9135 | 0.8919 | 0.8347 | 0.001106 | |
| | | 2nd order | 0.9776 | 0.9441 | 0.8355 | 0.001101 | |
| BD | Blend 2 | *M,S,T,SS,TT,MT | 0.9729 | 0.9549 | 0.9020 | 0.000656 | 0.006692 |
| | | 1st order | 0.9561 | 0.9451 | 0.9150 | 0.000273 | |
| | | *2nd order | 0.9933 | 0.9831 | 0.9474 | 0.000169 | |
| BD | Blend 3 | M,S,T,SS,TT,MS,ST | 0.9927 | 0.9862 | 0.9627 | 0.000120 | 0.003213 |
| | | 1st order | 0.9636 | 0.9546 | 0.9386 | 0.000934 | |
| | | 2nd order | 0.9852 | 0.9631 | 0.8833 | 0.001774 | |
| WA | Blend 1 | *M,S,T,MM,TT,MT | 0.9844 | 0.9740 | 0.9278 | 0.001098 | 0.015201 |
| | | M,S,T,MM,MT,MS | 0.9784 | 0.9640 | 0.9233 | 0.001167 | |
| | | *1st order | 0.6094 | 0.5117 | 0.2358 | 1.696455 | |
| WA | Blend 2 | 2nd order | 0.6707 | 0.1767 | -1.7900 | 6.193810 | |
| | | M,S,T,MM | 0.6490 | 0.5214 | -0.0569 | 2.346385 | 2.219964 |
| | | 1st order | 0.3374 | 0.1718 | -0.0764 | 0.584487 | |
| WA | Blend 3 | 2nd order | 0.6924 | 0.2311 | -1.2845 | 1.240524 | |
| | | *M,T,TT | 0.5235 | 0.4044 | 0.0342 | 0.524478 | |
| | | M,T,TT,MT | 0.5240 | 0.3509 | -0.1198 | 0.608090 | 0.543024 |
| BS | Blend 1 | *1st order | 0.9144 | 0.8930 | 0.8672 | 0.211949 | |
| | | 2nd order | 0.9488 | 0.8721 | 0.5827 | 0.665912 | |
| | | M,S,MM,SS | 0.9178 | 0.8880 | 0.7191 | 0.448297 | 1.595822 |
| BS | Blend 2 | 1st order | 0.5493 | 0.4366 | 0.1664 | 4.036374 | |
| | | 2nd order | 0.8994 | 0.7485 | 0.1708 | 4.014978 | |
| | | *M,S,T,MM,SS, MS,MT | 0.8886 | 0.7911 | 0.3409 | 3.191521 | 4.842141 |
| BS | Blend 3 | *1st order | 0.7188 | 0.6485 | 0.4742 | 0.690485 | |
| | | 2nd order | 0.8337 | 0.5844 | -0.3891 | 1.824325 | |
| | | M,S,T,MM | 0.8129 | 0.7448 | 0.3764 | 0.818939 | 1.313308 |
| BS | Blend 3 | 1st order | 0.7858 | 0.7323 | 0.6200 | 1.473666 | |
| | | 2nd order | 0.9650 | 0.9124 | 0.7421 | 1.054742 | |
| | | M,S,T,MM,MS | 0.9528 | 0.9292 | 0.8738 | 0.489283 | |
| | | *M,S,T,MM | 0.9361 | 0.9202 | 0.8770 | 0.477090 | 3.878173 |

^a R² = coefficient of determination; Adj R² = adjusted; R² Pred = predicted, PRESS residual = prediction error of sum of squares; SS total = sum of squares total. Because dependent measures were not transformed, SS total values were the same for all models.

^b 1st order model: $y = b_0 + b_1(M) + b_2(S) + b_3(T)$. 2nd order model: $y = b_0 + b_1(M) + b_2(S) + b_3(T) + b_4(MM) + b_5(SS) + b_6(TT) + b_7(MT) + b_8(ST) + b_9(MS)$. Reduced models included effects of process temperature (*T*), dough moisture (*M*), screw speed (*S*), process temperature squared (*TT*), dough moisture by process temperature interaction, etc. (*MT*). * = best models.

Experimental Design and Statistical Analysis

Data from each of the quality measurements (expansion index, bulk density, water absorption, and breaking strength) were related to the extrusion variables (process temperature, feed or dough moisture, and screw speed) using response surface methodology (Box and Draper 1987, Myers and Montgomery 1995). A second-order central composite design was used to collect data at 16 different design points (eight points for the 2^3 factorial cube portion, six points for the star portion, and two replicates at the center point). Each blend was processed at extrusion conditions more appropriate to that particular blend, and the conditions varied among the blends (Table I).

For each quality measurement within each blend, a minimum of three regression models was fit; a simple first-order model including only the linear terms for each extrusion variable, a full second-order model, and at least one reduced model arrived at through the use of all-subsets regression and an analysis of the residual plots. Models were compared using adjusted R^2 values, PRESS statistic, and approximate R^2 for prediction values based on PRESS. The full second-order model was used unless differences of 0.08–0.10 were observed between the full and reduced models for adjusted R^2 prediction values as recommended by Myers and Montgomery (1995). Small and negative R^2 prediction values imply models that do not generalize well to data other than those used to build the model.

Once a final model was selected for each quality measurement within each blend, the regression equation based on the coded values (converted to a range of –1 to 1) of the extrusion variables was used to generate predicted values for the quality measurements. The models were visualized with JMP (SAS Institute, Cary, NC) and Slicer (Fortner Research, Sterling, VA) to portray four-dimensional data and determine regions in the design space where optimal values occur. The values of the independent variables were the coordinates in three-dimensional space. The predicted value of the response variable at each unique point was assigned a color; red, yellow, and blue represented small, moderate, and large values of the dependent measure, respectively.

Statistical analyses were performed with SAS and JMP software. Contour plots and interactive visualization were accomplished using SAS/GRAPH, JMP, and Slicer for the Macintosh. ANOVA and Duncan's Multiple Range Test were used to analyze color, protein digestibility, and sensory evaluation data.

RESULTS AND DISCUSSION

Proximate analysis of the raw material used in the blends (Table II) compared well with the values found in the literature (USDA 1989).

Adjusted R^2 , PRESS, and R^2 for prediction statistics enabling

the comparison of the response surface regression models fit to each of the quality measurements are presented in Table III. Good fitting models were identified for blends 1 and 3 for expansion index and for all three blends for bulk density. Only the blend 3 models for water absorption and breaking strength appeared to fit well, suggesting that either more complex models or different variable values were required.

A single best model (largest R^2 for prediction and smallest PRESS statistic) was chosen for each blend by quality variable combination from Table III. When small differences between the first-order or second-order model and the reduced model were obtained, the reduced model was ignored and the first- or second-order model were selected.

Expansion Index

Observed (actual data) expansion index values for blends 2 and 3 were largely overlapping with ranges of ≈ 2.6 – 3.2 units. Blend 1 had observed values with a broader range of ≈ 2.9 – 3.9 units, indicating more expansion in the blend, which is a desired characteristic. Table II suggests that first-order models (linear components) for blends 1 and 3 account for much of the variability in expansion index with R^2 values ≈ 0.85 . Second-order response surface models appear to provide significant improvement for all three blends with coefficients of determination of 0.957, 0.887, and 0.990, respectively. Although tests for lack-of-fit were nonsignificant for the second-order models of all three blends, the PRESS statistics and R^2 of prediction values suggested that the second-order models for blends 1 and 2 may be more complex, especially when compared to the reduced models obtained using model selection procedures. Significance levels associated with the parameter estimates in the full second-order models for blends 1 and 2 show that the majority of the individual parameter estimates are nonsignificant, even when relaxing α to 0.10 (data not shown). Most of the parameter estimates in the reduced models were significant at $\alpha = 0.05$ (Table IV).

Screw speed and process temperature appeared to be the most important factors in modeling expansion index data for blend 1, whereas dough moisture seemed to be the most important factor in the blend 2 model (Table IV). Blend 3 exhibited significant effects for all three factors. Dough moisture was the most important factor based upon the highly significant F -test and the fact that the three most significant individual terms in the model contained dough moisture (Table IV).

The response surface analysis yielded a saddlepoint as the stationary point for each of the three blends. The use of visualization techniques on the expansion index data for blend 1 suggested that the highest predicted values of expansion index occurred at lower

TABLE IV
P-Values of Best Model Regression Coefficients^a Using Type III Sum of Squares for Quality Parameters of Buckwheat Flour Blend Extrudates

| Parameter/Blend | b_0 | b_1 | b_2 | b_3 | b_4 | b_5 | b_6 | b_7 | b_8 | b_9 |
|-------------------|--------|--------|--------|--------|--------|-------|-------|-------|-------|-------|
| Expansion index | | | | | | | | | | |
| 1 | 0.002 | 0.022 | <0.001 | <0.001 | | | 0.002 | 0.031 | | |
| 2 | <0.001 | <0.001 | | 0.015 | | | 0.017 | | | |
| 3 | 0.139 | 0.009 | 0.822 | 0.066 | <0.001 | 0.131 | 0.247 | 0.499 | 0.077 | 0.018 |
| Bulk density | | | | | | | | | | |
| 1 | 0.168 | 0.036 | 0.069 | 0.007 | | 0.142 | 0.017 | 0.091 | | |
| 2 | 0.229 | 0.025 | 0.814 | 0.587 | 0.801 | 0.186 | 0.050 | 0.521 | 0.087 | 0.012 |
| 3 | 0.024 | 0.027 | <0.001 | 0.045 | 0.017 | | 0.083 | 0.158 | | |
| Water absorption | | | | | | | | | | |
| 1 | 0.001 | 0.122 | 0.122 | 0.004 | | | | | | |
| 2 | 0.326 | 0.026 | | 0.043 | | | 0.038 | | | |
| 3 | <0.001 | <0.001 | 0.134 | 0.134 | | | | | | |
| Breaking strength | | | | | | | | | | |
| 1 | 0.529 | 0.706 | 0.794 | 0.059 | 0.157 | 0.015 | | 0.083 | | 0.012 |
| 2 | 0.244 | <0.001 | 0.063 | 0.089 | | | | | | |
| 3 | <0.001 | <0.001 | 0.200 | 0.013 | <0.001 | | | | | |

^a Model coefficients $b_0 - b_9$ are associated with the extrusion variables: dough moisture (M), screw speed (S), and process temperature (T) in: $y = b_0 + b_1(M) + b_2(S) + b_3(T) + b_4(M^2) + b_5(S^2) + b_6(T^2) + b_7(MT) + b_8(ST) + b_9(MS)$.

dough moisture, low process temperature, and moderate to high screw speed. Visualization of the actual data as well as multiple scatter plots of the actual data supported these results.

The best model for blend 2 was more limited than that for blends 1 and 3, and excluded screw speed entirely. Maximum predicted values for expansion index for blend 2 occurred at low dough moisture and mostly low process temperature, although results also suggested that low feed moisture and high process temperature may also yield high expansion index values.

The best model selected for blend 3 was the second-order response surface. Visualization techniques identified regions of maximum predicted expansion index values at low dough moisture and screw speed levels.

Contour plots for expansion index for all three blends are shown in Fig. 1. The highest value for screw speed was used in blend 1, the midpoint screw speed for blend 2, and the lowest screw speed for blend 3. In traditional contour plotting, the midpoints of the dimensions not directly plotted on the X and Y axes are typically used. Because some of the plots in Fig. 1 represent peripheral locations in the design space, they may depict greater extrapolations than the traditional contour plots. Consequently, the results should be viewed as guidance for further investigation. The objective of using the contour plots was to portray regions predicted to be optimal based upon models we fit. The optimal regions were identified using both traditional response surface methodology and visualization methods. The visualization methods played an important part in identifying regions in the design space where optimal predicted responses can be found. This is because models with significant interaction terms and saddle points for the stationary point suggest that no clear region for optima exists.

Some authors have reported that dough moisture and temperature play a major role in the extrudate expansion properties (Mercier and Feillet 1975, Kokini et al 1992). Launay and Lisch (1983) proposed that the corn extrudate longitudinal and diametral expansions depended on the melt viscosity and elasticity. They reported that an increased water content or temperature would yield a lower melt viscosity and increased longitudinal expansion; while the melt elasticity would be lowered and a decrease in diametral expansion would be observed. Blends 2 and 3 contain high proportions of corn, and our contour plots suggest that expansion index is reduced at high dough moisture. Our results showed that the influence on expansion index due to process temperature (Fig. 1) seemed to be moderately important for blend 1, not so much for blend 2, and not an important factor for blend 3.

It has been proposed that the volume expansion phenomena are mainly dependent on viscous and elastic properties of melted starch (Launay and Lisch 1983). Buckwheat flour contains an average of 42–58% amylose, ≈ 1.6 –2.0 times the amount expected in corn and wheat flour (Soral-Smietana et al 1984). It is possible that by changing the amylose-amylopectin ratio and introducing different molecular size amylopectin, the molten state of blend 1 produced a system more conducive to expansion at the tested process conditions.

The differences in the physical characteristics of the extrudates depend on the intrinsic properties of the solid matrix that is expanded as it exits the die. The solid matrix is affected by amylose-amylopectin ratio and the presence of NFD and buckwheat proteins. Lai and Kokini (1992) reported that under the same extrusion conditions, 70% amylose corn starch gave higher temperature rise due to viscous heating than did high-amylopectin (98%) corn starch. Thus, we attribute extrudates' physical differences, such as expan-

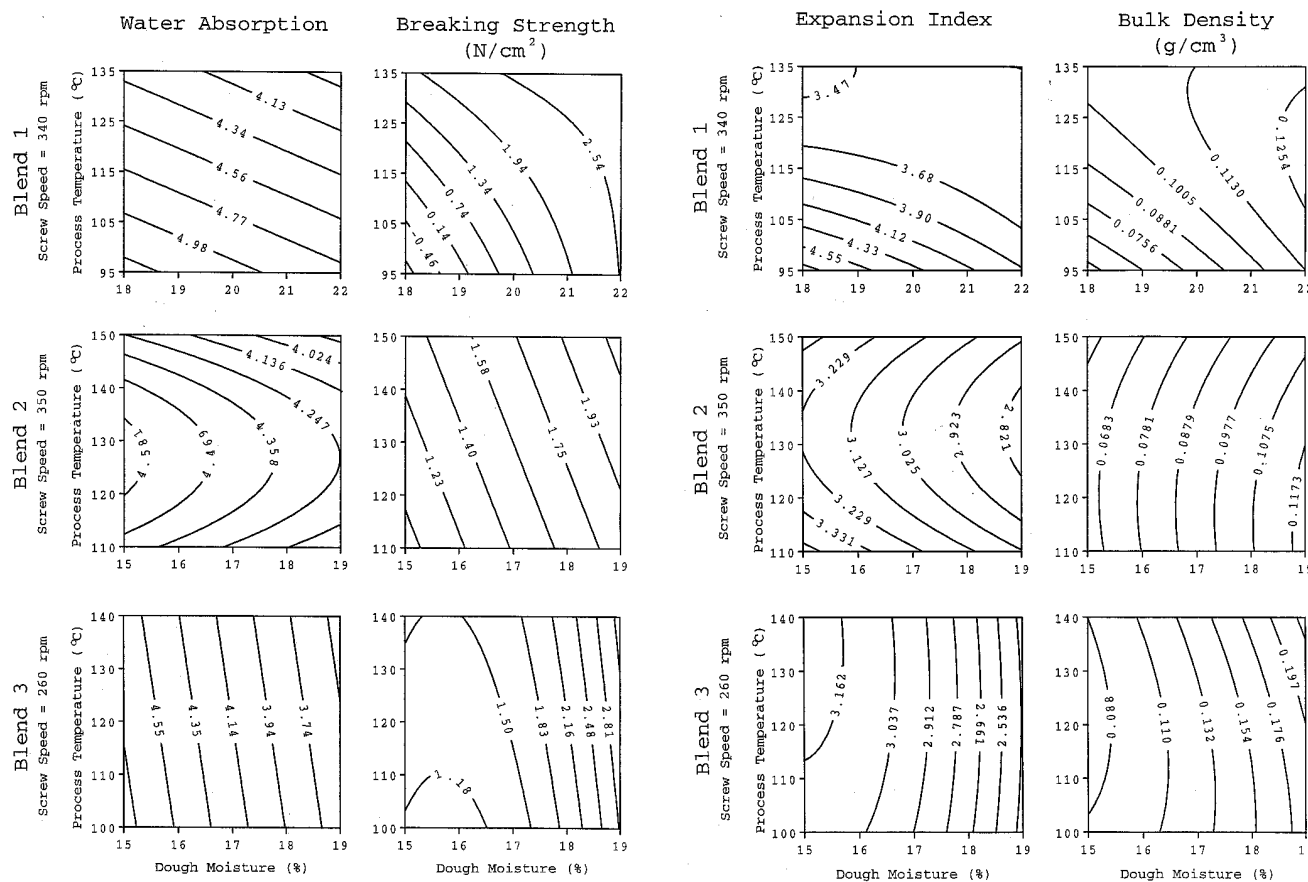


Fig. 1. Contour plots of extrudate quality variables as functions of dough moisture, process temperature, and screw speed for the three buckwheat flour blends. All 12 individual plots have the same orientation (dough moisture on x-axis, process temperature on y-axis) to facilitate comparisons. Individual plots within a row represent each of the quality characteristics for a single blend. Plots within each column represent the blends for a single quality characteristic.

sion, to rheological changes during melting of starch and proteins during the extrusion conditions of the three-component blends of this report.

Bulk Density

Bulk density values for blends 1 and 3 largely overlap with highs of about 0.17 g/cm³ and lows at 0.07–0.09 g/cm³. Blend 2 had a range of ≈0.07 to 0.12 g/cm³ suggesting an overall lighter extrudate, which is a desirable characteristic. Response surface regression models on bulk density yielded excellent fits with *R*² of prediction values for the best models >0.9 for all three blends (Table III). The best model for blend 2 was the full second-order model, whereas the best model for blends 1 and 3 included five of the nine possible terms from the second-order model (Table III). Dough moisture and process temperature appeared to be the most important factors for predicting bulk density for each of the blends as seen by the generally significant statistics for these parameters (Table IV). The preferred lower bulk density values occurred in the same regions as the high predicted values for expansion index (Fig. 1). Correlation coefficients between expansion index and bulk density (Table V) were strong for each blend and supported the contour plot results.

Water Absorption

Water absorption values ranged from 3.8 to 5.18 g of water per gram of dry sample for blend 1. The ranges for blends 2 and 3 were similar (3.98–4.76 and 3.69–4.70, respectively). A direct comparison of water absorption values in the literature is difficult due to differences in processing conditions and raw material used. Water absorption values in the literature range from 4.1 to 6.4 for corn meal (Conway et al 1968, Brenner et al 1986) and from 8 to 11 for wheat starch (Paton and Spratt 1984). Generally, the response surface models applied to water absorption fit poorly, with the best models for blends 1 and 3 being the basic first-order model (Table III). The best model for blend 2 has only three terms with the linear term for screw speed being replaced by the quadratic term for process temperature. Only the blend 3 model appeared to have good predictive capability as measured by the *R*² of prediction value of 0.87. The blend 1 and 2 models for water absorption appeared to be unreliable with *R*² of prediction values that are barely positive (Table III).

Contour plots suggested that lower values of water absorption are associated with high dough moisture for all three blends (Fig. 1). Comparison of the expansion index plots to the corresponding water absorption plots suggested that the higher values of expansion index were associated with high water absorptions. This was confirmed by the relatively high correlations between these two quality measurements, especially for blends 1 and 3 (Table V). The contour plots suggest that blend 1 and 3 extrudates with high expansion index were also more porous, as shown by high values of water absorption. The amount of water absorbed by the ground extrudate has been used as an indirect estimation of the porosity of the material (Colonna et al 1989). As the porosity of the extrudate material increases, the water absorption would also increase. In extruded breakfast cereals, a slow rate of water absorption is desirable to maintain crispiness, while in snack products, other texture attributes such as mouthfeel and firmness are also important.

Water absorption of extruded products may be interpreted on the basis of starch-water-protein interactions that govern the solid-phase structure. Water absorption has been generally attributed to the dispersion of starch in excess water, and the dispersion is increased by the degree of starch damage due to gelatinization and extrusion-induced fragmentation, that is, molecular weight reduction of amylose and amylopectin molecules (Colonna et al 1989, Politz et al 1994). In extruded wheat flours, fragmentation of starch occurred to a greater extent than in corn flours (Wen et al 1990, Rodis et al 1993). Amylopectin was the main fraction affected, with a reduction in molecular weight from 10⁷–10⁸ to 10⁵–10⁷

(Politz et al 1994). Among other factors affecting water absorption are the type of proteins, degree of denaturation, and amount of fiber present (Gujska and Khan 1990). The soluble and insoluble fiber content in the raw material used were 1.16 and 2.56% for the buckwheat flour and 0.50 and 1.43% for the wheat flour (P. Rayas-Duarte, unpublished data).

Breaking Strength

Observed values of breaking strength of blends 1 and 3 were relatively similar ranging from ≈1–3 N/cm². Blend 2 has a much narrower range of 1.18–2.04 N/cm². Response surface regression results for breaking strength were similar to those for water absorption in that only the blend 3 model appeared to have considerable predictive capability as measured by the *R*² of prediction of 0.88 (Table III). Blends 1 and 2 models yielded *R*² prediction values <0.5, suggesting that these models may not generalize well to new data. Dough moisture appeared to be the most influential extrusion variable in determining breaking strength for blends 2 and 3, whereas screw speed was more important in blend 1 (Table IV).

High expansion index values for blend 1 occurred at low dough moisture, low process temperature, and relatively high screw speed. The contour plots for blend 1 suggest that this region is associated with low predicted values of breaking strength (Fig. 1). High breaking strength values occurred at high dough moisture and high process temperature. Generally, blend 1 breaking strength values suggested a region of low moisture and temperature with more crispy characteristics. Higher moisture and temperatures generally yielded blend 1 extrudate with increasing crunchy characteristics.

High expansion index values for blend 2 occurred at low moisture and extreme (low and high) process temperatures. Although the equation generating the contour plot had limited predictive capability, blend 2 predicted breaking strength values were relatively low in this region of the design space (Fig. 1). This suggests that the product obtained with high expansion index will be crispy rather than crunchy, which was confirmed by actually testing the extrudates. Blend 3 exhibits a similar pattern with relatively low breaking strength associated with desirable values of expansion index (Fig. 1). The negative association between expansion index and breaking strength is confirmed by the correlation coefficients in Table V.

Breaking strength is reported to be a function of cell structure, that is, cell size and cell wall thickness (Launay and Lisch 1983). High breaking strength values are generally related to large cells with thicker cell walls, creating a crunchy texture. Low breaking strength values are usually related to a large number of small cells per unit area with thinner cell walls, resulting in a crispy texture. Among the factors affecting crispiness is the facility to rupture the cell walls of a product (Launay and Lisch 1983) which is correlated to the overall energy applied (thermal and mechanical). This will govern the shape of the cell walls, that is, amorphous versus

TABLE V
Correlation Coefficients Among the Quality Parameters^a
for Each of Three Blends (*n* = 16)

| Blend | Parameter | EI | BD | WA | BS |
|-------|-----------|--------|---------|---------|---------|
| 1 | EI | 1.0000 | -0.9074 | 0.7917 | -0.3830 |
| | BD | | 1.0000 | -0.7002 | 0.2190 |
| | WA | | | 1.0000 | -0.4414 |
| | BS | | | | 1.0000 |
| 2 | EI | 1.0000 | -0.7145 | 0.3350 | -0.7260 |
| | BD | | 1.0000 | -0.4173 | 0.7451 |
| | WA | | | 1.0000 | -0.3838 |
| | BS | | | | 1.0000 |
| 3 | EI | 1.0000 | -0.9245 | 0.9274 | -0.9546 |
| | BD | | 1.0000 | -0.9766 | 0.9298 |
| | WA | | | 1.0000 | -0.9256 |
| | BS | | | | 1.0000 |

^a EI = expansion index, BD = bulk density, WA = water absorption, BS = breaking strength.

organized cell wall material. In expanded rice products, dough moisture has been reported as the most important factor affecting crispiness, bulk density, hardness, and appearance (Pan et al 1992). Figure 1 shows that, generally, dough moisture had a stronger influence in the expansion index, bulk density, and breaking strength than did process temperature in products containing corn meal (blends 2 and 3).

Protein Digestibility

The dough moisture of samples ranged from 2.70 to 3.80%. The *in vitro* protein digestibility was analyzed in six selected treatments per blend (Table VI). When compared to raw material values, extrusion process generally significantly increased protein digestibility in the selected extrudates from the three blends. Overall means ranked as blend 1 > blend 2 > blend 3, with values of 84.8, 83.3, and 81.7% protein digestibility, respectively. Overall means of protein content were 17.56, 13.62, and 12.97% (db) for blends 1, 2, and 3, respectively. Higher protein and protein digestibility were observed in samples with higher content of light buckwheat flour. Wheat and corn meal have limited nutritional value, lysine and tryptophan limiting amino acids, while buckwheat complements their deficiencies. Metabolic availability is also important and buckwheat has higher biological value than corn meal and wheat flour (Sure 1955).

Nutritional value in cereal protein is usually enhanced by mild extrusion cooking conditions, due to an increase in digestibility (Chung and Pomeranz 1985, O'Connor 1987, Kozikowski et al 1989, Stanley 1989). This study suggests that the majority of the extrusion conditions significantly increased the protein digestibility of buckwheat-containing extrudates when compared with the nonextruded raw material. The selected extrusion processing conditions generally yielded similar *in vitro* protein digestibility values within the same blend.

Sensory Evaluation

The three treatments per blend selected for sensory evaluation based on the best product characteristics (expansion index, bulk density, water absorption, and shear stress) are presented in Table VII.

TABLE VI
Protein Content and *In Vitro* Protein Digestibility of Buckwheat Flour Blend Extrudates

| Blend/Treatment ^a | Protein (% dwb) | Protein Digestibility (%) |
|------------------------------|---------------------|---------------------------|
| Blend 1 | | |
| Raw material | 17.63b ^b | 77.31d |
| 20.0/300/94 | 16.89d | 83.80bc |
| 19.1/320/105 | 17.16c | 85.04a-c |
| 20.0/340/115 | 17.50b | 82.67c |
| 18.0/300/115 | 17.73b | 86.62a |
| 19.1/320/125 | 18.04a | 85.83ab |
| 20.0/300/135 | 18.06a | 84.71a-c |
| Blend 2 | | |
| Raw material | 13.76b | 75.00c |
| 17.2/350/110 | 13.32cd | 82.90ab |
| 16.1/370/120 | 13.27cd | 84.48a |
| 16.1/330/120 | 13.10d | 83.35ab |
| 14.9/350/130 | 14.21d | 84.25ab |
| 16.1/370/142 | 14.33a | 83.57ab |
| 17.2/350/151 | 13.52bc | 81.20b |
| Blend 3 | | |
| Raw material | 12.39d | 77.25b |
| 17.0/300/100 | 12.76c | 83.01a |
| 16.2/320/110 | 12.85bc | 81.77a |
| 16.2/280/110 | 12.94bc | 80.53ab |
| 14.8/300/120 | 12.99b | 81.99a |
| 15.9/320/130 | 13.03ab | 81.43a |
| 17.0/300/140 | 13.24a | 81.54a |

^a Dough moisture (%) / screw speed (rpm) / temperature (°C).

^b Values followed by the same letter in the same column for each blend are not significantly different ($\alpha = 0.05$) ($n = 2$) using Duncan's multiple range test.

There were no significant differences ($\alpha = 0.05$) in general acceptability and flavor in selected extrudates from each treatment for respective blends and in texture of treatments of blends 2 and 3.

Blend 1 extrudates from treatments 2 and 8 differed significantly in color and texture. There were significant differences in color for treatments 7 and 12 (blend 2) and 2 and 7 (blend 3). Blend 1 extrudates received lower acceptability scores (dislike slightly or moderately) than did blends 2 and 3 containing corn meal. This corresponded to overall less crunchy texture with lower breaking strength values of blend 1. Treatments 2 and 7 of blend 3 (30:60:10 mixtures of buckwheat, corn, and NFDM) received the highest scores. Overall, these extrudates were more crunchy than the other blends.

Color Analysis

In general, the range of *L* (lightness) value obtained for blend 1 (78.82–71.56, the higher the value the brighter the product) represented lower values than that for blend 3 (79.96–74.50). Extrudates processed in the range of 104–110°C, 19% dough moisture, and 295–304 rpm gave the highest values of *b* (yellowness) and *a* (redness) for blend 1 (values obtained from contour maps not shown). The highest *b* values in blend 3 were obtained at 140°C and 300–340 rpm at all dough moistures tested (14–19%). The data suggest that blend 3 gave a lighter (higher *L* values) and yellower (higher *b* values) extrudate product as compared to blends 1 and 2.

CONCLUSION

The influence of three process variables (process temperature, dough moisture, and screw speed) in the extrusion quality of three blends containing buckwheat showed that the linear term component of these factors explained most of the variations observed. The greatest amount of variability on the response variables in blend 1 was the process temperature, while in blends 2 and 3 the variability was due mainly to dough moisture. An interaction of dough moisture by screw speed significantly affected the breaking strength of blend 1, bulk density of blend 2, and expansion index of blend 3. Although blend 1 had the highest protein content and protein digestibility, the sensory evaluation scores and objective color measurements indicated the overall acceptability of the extrudates ranked blend 3 > blend 2 > blend 1.

The use of buckwheat flour in extruded snacks and ready-to-eat breakfast cereals products offers a desirable variation in flavor and can take advantage of the nutritional quality of buckwheat. This study suggested that expansion index was the most important indicator of the overall buckwheat-containing extrudate quality.

TABLE VII
Sensory Evaluation of Selected Buckwheat Flour Blend Extrudates

| Blend/Treatment ^b | Sensory Evaluation Scores ^a | | | |
|------------------------------|--|--------|---------|-----------------------|
| | Color | Flavor | Texture | General Acceptability |
| Blend 1 | | | | |
| 19/320/105 | 4.19a ^c | 4.27a | 4.11a | 4.00a |
| 20/340/115 | 3.43b | 4.04a | 3.22b | 3.44a |
| 19/320/125 | 3.69ab | 4.09a | 3.67ab | 3.80a |
| Blend 2 | | | | |
| 16/370/120 | 6.00ab | 5.33a | 6.33a | 5.67a |
| 14/350/130 | 5.63b | 5.35a | 6.46a | 5.56a |
| 16/370/142 | 6.28a | 5.02a | 6.28a | 5.46a |
| Blend 3 | | | | |
| 16/320/110 | 6.39a | 5.61a | 6.43a | 5.85a |
| 14/300/120 | 5.74b | 5.59a | 6.56a | 5.76a |
| 17/300/140 | 6.09ab | 5.39a | 6.63a | 5.71a |

^a Ratings for each sample are scored as "like extremely" (9) to "dislike extremely" (1).

^b Dough moisture (%) / screw speed (rpm) / temperature (°C).

^c Values followed by the same letter in the same column for each blend are not significantly different ($\alpha = 0.05$) ($n = 2$) using Duncan's multiple range test.

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