

The impact of extrusion processing on fortified breakfast cereals containing β -glucan-enriched flour: physical, structural, and nutritional aspects

Sibel Yağcı,^{a*} Nazlı Şahin,^b Ayşegül Çalışkan^b and İncinur Hasbay^c



Abstract

BACKGROUND: This study evaluated the effects of various factors on breakfast cereals containing β -glucan-enriched flour fraction (BEFF). A central composite design (CCD) was used to examine the effects of BEFF level, screw speed, and die temperature on the physical, textural, microstructural, and nutritional properties of the cereals.

RESULTS: The addition of BEFF initially increased the L^* value by 5.1%, followed by a decrease at higher levels. Porosity increased linearly, whereas starch digestibility decreased. Screw speed was the most significant independent variable affecting the physical and structural properties of the cereals, followed by die temperature. The optimum processing conditions were 5.1% BEFF, 435 rpm screw speed, and 170 °C die temperature (with the other barrel zones maintained at 40, 50, 70, 90, and 100 °C), yielding β -glucan content of 14.3 g kg⁻¹ and starch digestibility of 196.46 mg maltose g⁻¹. β -Glucan loss was limited to 42% at most when compared with the unextruded blend.

CONCLUSION: Extrusion increased starch digestibility and reduced β -glucan content, and the enthalpy of starch gelatinization tended to decrease with BEFF incorporation into the control sample. All breakfast cereal products in this study met Food and Drug Administration (FDA) requirements, containing at least 0.75 g of β -glucan per serving portion.

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Supporting information may be found in the online version of this article.

Keywords: β -glucan; breakfast cereal; extrusion; microstructure; starch digestibility

INTRODUCTION

The breakfast cereal industry has improved considerably, driven by increased demand for ready-to-eat foods due to urbanization, greater awareness of healthy nutrition, advances in grain processing technologies, and evolving marketing techniques. The global breakfast cereal products market, valued at around USD 35 billion, is predicted to reach 40 billion by 2030.¹ Breakfast cereals are often high in sugar, sodium, saturated fat, and energy, while providing limited nutritional and functional benefits.² Research has demonstrated that extrusion can enhance dietary fiber intake, which is associated with multiple health benefits.³⁻⁵

Barley is a rich source of dietary fiber, with β -glucan content typically ranging from 20 to 100 g kg⁻¹ dry basis (db), although certain genotypes can reach 110–170 g kg⁻¹.³ It has been studied extensively for its potential health benefits. Recent findings indicate that the health benefits of β -glucan appear when at least 0.75 g is consumed in a serving portion, contributing to a total daily intake of 3 g to achieve a cholesterol-lowering effect.^{4,6} β -Glucan can be extracted and purified using enzymatic, hot water, alkali, and solvent methods, with the choice of method affecting the texture of the food, depending on the β -glucan source.⁷ In this study, the aqueous extraction method, the most

commonly used technique, was used to extract β -glucan from barley. This approach yielded high concentrations of β -glucan while minimizing the use of chemicals.⁷

Studies have shown that incorporating β -glucan-rich ingredients (e.g., barley or oat fractions) into starch-based formulations can produce extruded food products such as snacks and breakfast cereals. During extrusion, β -glucan in cereals can undergo structural modifications that increase extractability and solubility, contributing to a softer texture. It also increases dietary fiber content, reduces snack hardness, and mitigates potential glycemic responses.⁴ Research demonstrated that consumption of a granola meal containing 3.2 g of cereal β -glucan improved glycemic

* Correspondence to: S Yağcı, Balıkesir University Department of Food Engineering, 10145, Balıkesir Turkey, E-mail: sibelyagci@balikesir.edu.tr (Yağcı)

a Balıkesir University Department of Food Engineering, Balıkesir, Turkey

b Karamanoğlu Mehmetbey University Department of Food Engineering, Karaman, Turkey

c TUBITAK Marmara Research Center, Food Institute, Kocaeli, Turkey

regulation in healthy individuals after only 3 days.⁸ The type of barley used affects the quality of the extruded products significantly.³

It is important to regulate processing conditions in order to achieve satisfactory physicochemical and structural characteristics that influence sensory and nutritional properties.^{3,4} The objectives of this study were: (i) to investigate the incorporation of high-level BEFF from barley into a breakfast cereal model system, focusing on physical, textural, microstructural, and nutritional characteristics; (ii) to optimize extrusion processing conditions (die temperature, screw speed, and BEFF level) using response surface methodology (RSM); and (iii) to determine the chemical composition, and nutritional and structural properties of the BEFF-incorporated optimum sample.

MATERIALS AND METHODS

Materials

Corn semolina, corn starch, and wheat semolina were obtained from Gesaş Food Industry Inc. (Karaman, Türkiye) and Golda Food Industry Inc. (Karaman, Türkiye). During preliminary research, the levels of β -glucan were evaluated in five different barley cultivars. Two research institutes provided five cultivars for the study: two hullless cultivars (Yalin and Ozen) were provided by the Ministry of Agriculture and Forestry Field Crops Central Research Institute and three hulled cultivars (Larende, Kral 97 and Karatay 94) were provided by Bahri Dagdas International Agricultural Research Institute. After analysis, all cultivars had β -glucan levels ranging from 41.6 to 45.9 g kg⁻¹. However, the Yalin cultivar had the highest level, 45.9 g kg⁻¹, and was chosen for further investigation. Grain samples for analysis were milled and the fraction that passed through a 500 μ m sieve was collected and used (Retsch ZM1, Haan, Germany).

The BEFF was obtained from the Yalin barley cultivar using a commonly applied aqueous extraction technique, which yields high β -glucan concentrations with minimal chemical use. To deactivate β -glucanase, the barley flour was first refluxed with ethanol before extraction.⁷ The extraction was conducted in a glass reactor (AGI, Kumamoto, Japan) at 200 rpm, 70 °C, for 3 h, using 150 g of barley flour to 3.75 L water (barley flour and water ratio, 40 g L⁻¹). After extraction, solid particles were removed by centrifugation at 9700 \times g for 15 min at 4 °C, and proteins in the supernatant were precipitated by adjusting the pH with 5 mol L⁻¹ HCl, followed by centrifugation at 2994 \times g for 15 min, 4 °C using a rotor (Centrifuge 5840R; Eppendorf SE, Hamburg, Germany).

Extrusion process

Breakfast cereal was produced using a computer-controlled laboratory-type, intermeshing, and co-rotating twin-screw extruder (Rondol Technology Ltd., Staffordshire, UK). The extruder barrel was connected to a smart cooling system (Labtech, Bucharest, Romania) to control each zone temperatures at 40, 50, 70, 90, and 100 °C. Barrel diameter, length-to-diameter ratio (L/D), and feed rate were 21 mm, 40:1, and 2 kg h⁻¹, respectively. The cereal flour formulation – 475 g kg⁻¹ corn semolina, 475 g kg⁻¹ wheat semolina, and 50 g kg⁻¹ corn starch (db) – and moisture content (170 g kg⁻¹) were kept constant. The level of BEFF in the cereal formulation varied from 0 to 83.6 g kg⁻¹ (w w⁻¹, db) in all experimental runs and was calculated over the cereal formulation into the breakfast cereal. The samples were shaped using a circular die with a 4 mm diameter, dried in a

cabinet dryer (Biyosan Chemistry and Laboratory Devices, Ankara, Türkiye) at 45 °C to 50–60 g kg⁻¹ moisture content, and stored in polyethylene bags at 10 °C for further analysis.

Experimental design and statistical analysis

The experiment employed a central composite design (CCD) in Design Expert 11.0 (Minneapolis, MN, USA). The CCD comprised three independent variables and five levels. The variables were die temperature (120–170 °C), screw speed (300–500 rpm), and BEFF level (30–70 g kg⁻¹, w w⁻¹). The Supporting Information, Table S1, shows the physical, textural, microstructural, and nutritional response variables of the experiment. Mathematical models for each response were developed using multiple regression with backward stepwise analysis, including only variables significant at $P < 0.05$. The models' goodness-of-fit was evaluated using R^2 , adjusted R^2 , and predicted R^2 , based on predicted residual sum of squares (PRESS), F-test results, and analysis of residual plots.

The physical properties of breakfast cereal products, such as color, texture, and density, are the most critical characteristics influencing consumer acceptance. Optimal conditions for numerical optimization were maximum lightness (L^*) and expansion ratio values and minimal hardness. The Pearson correlation test in the SPSS 16 program (SPSS Inc., Chicago, IL, USA) was used to determine the statistical relationships between the experimental parameters.

Chemical analysis of raw materials and unextruded blends

The moisture (AOAC 934.06), fat (AOAC 954.02), crude fiber (AOAC 950.02), protein (AACC, 46-30), total starch, and total ash (AOAC 942.06) content of the corn starch, BEFF, corn semolina, wheat semolina, and unextruded blends were analyzed using the standardized procedures outlined by the Association of Official Analytical Chemists (AOAC)⁹ and American Association of Cereal Chemists (AACC).¹⁰

Analyses of breakfast cereals

Physical and textural properties

Sample diameter was measured with a digital caliper and the expansion ratio was calculated as the ratio of sample diameter to die diameter. True density was determined using a gas pycnometer (Accupyc 1340, Micromeritics Instrument Corporation, Norcross, GA, USA). Apparent density was measured by the glass bead displacement method.¹¹

The porosity and textural characteristics of the breakfast cereals were determined following the methods described by Yağcı *et al.*⁵ and Veillard *et al.*,¹² respectively. Textural measurements were performed using a TA.XT2i texture analyzer (Stable Micro Systems Ltd., Godalming, UK) equipped with a Kramer shear cell, 50 kg load cell, and a 5 g trigger force. Sample lengths were adjusted to 20–25 mm. Force–deformation curves obtained during testing were analyzed with Texture Exponent 32 (version 2.03). Crispiness (mm⁻¹) was calculated following the method described by Samuel *et al.*¹³ as the total number of force peaks (fracture events) divided by the deformation distance, representing the frequency of structural breakage per millimeter of probe movement. The hardness was defined as the maximum force (N) recorded during the test.

Color was expressed using L^* (lightness), a^* (redness–greenness), and b^* (yellowness–blueness) indices, measured with a Hunter Lab Colorimeter (Hunter Associates Laboratory Inc., Reston, VA, USA).

Structural analysis

The pore structure and size of the two samples, the control sample (CS) and the optimum sample (OS), were determined using an Olympus SZ61 camera microscope. The CS consisted solely of a cereal grain mixture and was produced under ideal extrusion conditions (screw speed: 435 rpm; die temperature: 170 °C). The OS contained 5.1% BEFF and was produced under the same conditions. Circular 1 cm cross-sections were taken and inked with blue China ink. The cross-sections were captured with the camera microscope and the images were analyzed with Kameram 21 CCD software (Argenit Company, Ankara, Türkiye). The average pore area and number were calculated using the method described by Barrett and Peleg,¹⁴ considering pores larger than 0.04 mm² (Fig. 1).

For scanning electron microscopy (SEM), cross-sectional images were obtained to assess cell structure and wall thickness. Samples were carefully cut with a razor blade mounted on SEM stubs, and coated with 15 nm of gold–platinum.¹⁵ Imaging was performed using a Hitachi scanning electron microscope (Hitachi, Tokyo, Japan) operated at 5 kV.

Nutritional analysis

Starch digestibility of extruded samples and unextruded blends was analyzed following the method described by Onyango *et al.*¹⁵ with minor modifications, by quantifying maltose (mg g⁻¹ of sample). This method was described in detail previously by Yağcı *et al.*⁵ The β -glucan content of raw materials, unextruded blends, and all extruded samples was determined using the Megazyme β -Glucan kit (Megazyme Int., Wicklow, Ireland), following the method described by McCleary and Glennie-Holmes.¹⁶ The total dietary fiber (TDF) content of the OS was determined according to AOAC Official Method 991.43.⁹

The extent of starch gelatinization in unextruded blends containing 0% BEFF (CS), 5.1% BEFF (OS), and 8.36% BEFF (C) was determined using differential scanning calorimetry (DSC). Approximately 4 mg (db) of each sample was weighed into a 70 μ L aluminum pan, and 12 μ L of deionized water was added to achieve a water-to-dry-sample ratio of 3:1 (w w⁻¹). The pan was hermetically sealed and analyzed using a DSC instrument (Exstar6000, Seiko Instruments Inc., Tokyo, Japan) equipped with a Polyscience intercooler (Polyscience Inc., Niles, IL, USA). Samples were heated from 10 to 120 °C at 5 °C per min. Transition temperatures were determined from the thermogram, recording onset (T_o), peak (T_p), and end (T_e) temperatures. The enthalpy of gelatinization (ΔH_g), representing the energy required to melt starch crystallites, was calculated from the area of the endothermic peak and expressed as mJ mAt g⁻¹ sample on a dry basis.¹⁷

According to Food and Drug Administration (FDA) guidelines, a serving size of approximately 52.5 g of the optimized breakfast cereal product provides 0.75 g of β -glucan, the minimum effective dose for cardiovascular health benefits.

RESULTS AND DISCUSSION

Proximate composition of raw materials and pre-extrusion blends

Table 1 shows the chemical composition of the raw materials and unextruded blends on a dry-weight basis. Barley-derived BEFF contained 165 g kg⁻¹ protein and 14.8 g kg⁻¹ fat. The protein and fat composition of β -glucans can vary depending on the source and extraction method.³ The protein and ash content of the mixtures increased with the proportion of BEFF. The fat content of the mixtures ranged from 11.84 to 13.37 g kg⁻¹; the crude fiber content from 3.50 to 9.13 g kg⁻¹; the ash content from 8.09 to 11.01 g kg⁻¹, and the total carbohydrate content from 758.1 to 788.0 g kg⁻¹.

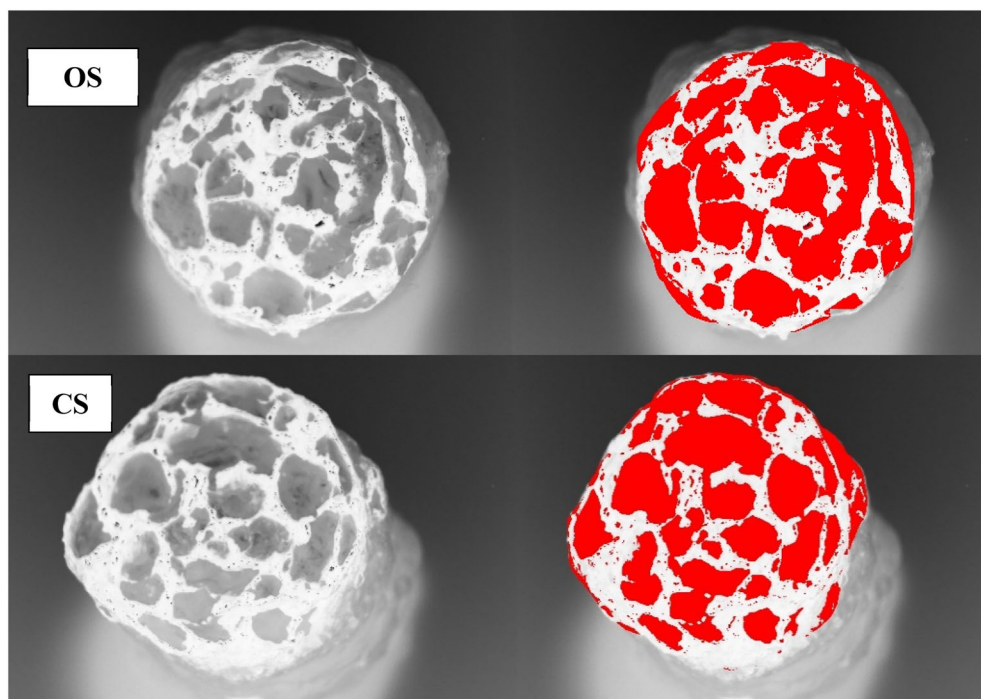


Figure 1. Camera microscope images of breakfast cereals.

Table 1. Chemical composition of raw materials and unextruded blends

Sample	Moisture (g kg ⁻¹)	Fat (g kg ⁻¹) ^a	Crude fiber (g kg ⁻¹) ^a	Protein (g kg ⁻¹) ^a	Ash (g kg ⁻¹) ^a	Total Starch (g kg ⁻¹) ^a	β -glucan content (g kg ⁻¹) ^a	<i>In vitro</i> starch digestibility (mg maltose g ⁻¹ sample)
Corn grit	112.2 ± 4.9	23.12 ± 1.62 a	12.45 ± 0.95 a	70.80 ± 0.26 c	8.69 ± 0.14 b	713.70 ± 7.31 c	6.17 ± 0.07 b	57.96 ± 0.74 c
Wheat grit	127.9 ± 9.7	10.92 ± 0.16 c	6.10 ± 0.64 b	115.01 ± 1.05 b	7.89 ± 0.17 b	748.48 ± 9.79 b	6.39 ± 0.10 b	84.41 ± 0.92 a
Corn starch	113.7 ± 8.2	0.72 ± 0.17 d	1.65 ± 0.87 c	2.71 ± 0.13 d	1.29 ± 0.32 c	808.28 ± 4.14 a	10.7 ± 0.15 b	74.60 ± 0.41 b
BEFF	68.3 ± 9.6	14.80 ± 1.22 b	1.59 ± 0.80 c	165.08 ± 4.17 a	66.77 ± 1.32 a	111.78 ± 5.08 d	333.62 ± 19.08 a	8.36 ± 0.03 d
0% BEFF	95.8 ± 8.4	11.84 ± 0.11 a	9.13 ± 0.99 a	87.11 ± 0.14 d	8.09 ± 0.12 c	783.64 ± 9.45 a	6.47 ± 0.05 b	68.77 ± 1.11 a
3% BEFF	95.2 ± 10.9	10.74 ± 0.16 a	8.42 ± 0.25 a	92.62 ± 0.46 c	8.29 ± 0.45 c	770.00 ± 2.37 a	16.77 ± 0.05 a	67.48 ± 0.27 ab
5% BEFF	104.8 ± 1.4	13.01 ± 1.12 a	7.09 ± 0.58 a	98.21 ± 0.05 b	9.66 ± 0.19 b	764.33 ± 3.75 a	23.25 ± 0.05 a	65.80 ± 0.23 b
7% BEFF	107.8 ± 3.5	13.15 ± 1.58 a	8.06 ± 1.04 a	100.30 ± 0.57 ab	10.39 ± 0.20 ab	762.45 ± 9.85 a	28.63 ± 0.57 a	63.43 ± 0.47 c
8.36 %BEFF	109.6 ± 4.8	13.37 ± 1.11 a	3.50 ± 0.48 b	102.02 ± 1.39 a	11.01 ± 0.22 a	760.90 ± 12.30 a	30.58 ± 1.46 a	62.70 ± 0.24 c
5.1% BEFF	110.4 ± 5.1	13.11 ± 0.75 a	7.39 ± 0.34 a	100.34 ± 0.05 ab	10.65 ± 1.25 ab	762.16 ± 3.01 a	24.47 ± 1.25 a	65.42 ± 0.58 b

Abbreviation: BEFF, β -glucan-enriched flour fraction.

^a Data are expressed on a dry weight basis. Results are presented as means ± SDs. Different lower case letters in the same column indicate significant differences ($P < 0.05$).

Physical and textural properties of the breakfast cereals

The color of extruded breakfast cereals is an important quality attribute. Cereals enriched with β -glucan showed variations in lightness (L^*), redness (a^*), and yellowness (b^*) with ranges of L^* : 48.66–73.65, a^* : 3.15–6.27, and b^* : 25.19–32.96. Table 2 provides analysis of variance (ANOVA) results for the L^* model, as the model did not adequately describe the a^* and b^* data.

Expansion ratio, apparent density, and porosity were measured to evaluate expansion behavior. The expansion ratio is a key parameter for determining consumer preference in extruded products developed as snacks or breakfast cereals. In this study expansion ratios ranged from 2.94 to 4.02, apparent density from 96.2 to 252.7 kg m⁻³, and porosity from 0.840 to 0.935. The total pore number, average pore area, and pore distribution within the extrudate structure are also crucial for product quality.

Average pore area ranged from 2.26 to 8.02 mm² and total pore numbers ranged from 41 to 110.

Altan *et al.*¹⁸ reported that the sectional expansion index of barley flour extrudate was 2.033 and the apparent density was 0.365 g cm⁻³. Textural properties of extruded products are usually expressed as hardness and crispness, with hardness varying between 117.1 and 440.5 N, and crispness between 2.18 and 4.07 mm⁻¹ (Supporting Information, Table S1).

Effects of process variables

Effect of BEFF level on product responses

Table 3 presents the mathematical models for various response parameters. The ANOVA results showed that the BEFF level affected several parameters significantly, including the L^* value, expansion ratio, porosity, hardness, average pore area, β -glucan

Table 2. Analysis of variance results for the response surface methodology (RSM) response parameters

	Physical properties				Textural properties		Microstructure properties		Nutritional properties	
	L^*	Expansion ratio	Apparent density (kg m ⁻³)	Porosity	Hardness (N)	Crispness (mm ⁻¹) ^b	Average pore area (mm ²)	Total pore number	β -glucan content (g kg ⁻¹)	Starch digestibility (mg maltose g ⁻¹ dry sample)
Model <i>P</i> value	0.0001*	0.0001*	0.0001*	0.0001*	0.0001*	0.0001*	0.0001*	0.0001*	0.0001*	0.0002*
R^2	0.9560	0.9755	0.8680	0.8473	0.9098	0.8857	0.8856	0.7175	0.9855	0.8789
R^2_{adj}	0.9303	0.9611	0.8444	0.8066	0.8775	0.8553	0.8447	0.6645	0.9816	0.8356
R^2_{pred}	0.7983	0.9001	0.7295	0.6821	0.7343	0.7307	0.7298	0.5300	0.9735	0.6973
PRESS	298.47	0.152	11 470.91	0.0052	50 197.01	1.40	12.83	3842.51	0.0568	14 277.73
Adequate precision	15.913 ^a	30.660 ^a	20.908 ^a	17.091 ^a	19.114 ^a	20.395 ^a	18.503 ^a	13.559 ^a	63.96 ^b	14.7221 ^b
CV	3.81	1.55	12.8	1.43	14.59	5.86	15.1	15.73	3.33	10.37
Lack of fit	0.2599 ^c	0.0873 ^c	0.3061 ^c	0.1807 ^c	0.1145 ^c	0.0754 ^c	0.1278 ^c	0.4080 ^c	0.3169 ^c	0.5100 ^c

R^2 , coefficient of determination; R^2_{adj} , adjusted R^2 ; R^2_{pred} , predicted R^2 ; PRESS, predicted residual sum of squares; CV, coefficient of variation.

^a A ratio greater than 4 is desirable.

^b Crispness (expressed as mm⁻¹) representing the frequency of structural breakage per millimeter of probe movement.

^c Lack of fit should be non-significant at $P < 0.05$.

* $P < 0.01$; significant.

Table 3. Predicted regression equations for physical, textural, microstructural, and nutritional properties
$$L^* = -264.44 + 7.52x_1 + 0.61x_2 + 2.64x_3 - 0.002x_2x_3 - 0.76x_1^2 - 0.0006x_2^2 - 0.007x_3^2$$

$$\text{Expansion ratio} = -1.22 - 0.065x_1 + 0.012x_2 + 0.026x_3 + 0.00059x_1x_2 - 0.000043x_2x_3 - 0.0012x_1x_3 - 0.0000069x_2^2$$

$$\text{Apparent density} = +773.34 - 2.30x_2 - 0.67x_3 + 0.0024x_2^2$$

$$\text{Porosity} = 0.53 + 0.0048x_1 + 0.0013x_2 + 0.00027x_3 - 0.0000013x_2^2$$

$$\text{Hardness} = 1561.15 - 29.77x_1 - 4.67x_2 - 1.18x_3 + 3.27x_1^2 + 0.0046x_2^2$$

$$\text{Crispness} = -5.14 + 0.039x_2 + 0.0087x_3 - 0.000036x_2^2$$

$$\text{Pore area}_{\text{average}} = -5.83 + 0.21x_1 - 0.0049x_2 + 0.171x_3 - 0.00044x_2x_3 + 0.00007x_2^2$$

$$\text{Pore number}_{\text{total}} = -452.23 + 0.137x_2 + 6.35x_3 - 0.021x_3^2$$

$$\beta\text{-glucan content} = 8.3 + 2.42x_1 - 0.0008x_2 - 0.0082x_3 - 0.085x_1^2$$

$$\text{Starch digestibility} = 102.88 - 39.93x_1 + 0.011x_2 + 0.765x_3 + 0.095x_1x_2$$

x_1 , BEFF level (%); x_2 , screw speed (rpm); x_3 , temperature ($^{\circ}\text{C}$).

content, and starch digestibility. The BEFF level showed a linear effect on porosity and both linear and quadratic effects on hardness but no significant effect on apparent density, crispness, or total pore number ($P > .05$). An increase in BEFF in the formulation led to a slight increase in the L^* value at 5.1%, followed by a decrease (Supporting Information, Fig. S1(a)). The expansion ratio also decreased with higher BEFF substitution (Supporting Information, Fig. S1(b)) and the magnitude of this effect depended on screw speed and die temperature. At lower screw speeds, the expansion ratio decreased as the BEFF level increased whereas it increased at higher screw speeds (Supporting Information, Fig. S1(c), (d)).

Recent studies have shown that high die temperatures combined with high screw speeds can enhance product expansion through rapid water evaporation acting as a physical blowing agent and creating shear-induced structural disruption.¹⁹ This is consistent with previous reports on extrudates with added fiber, such as tomato pomace-added barley-based extrudates by Altan *et al.*²⁰ and carrot pomace-incorporated barley extrudates by Lotfi Shirazi *et al.*²¹

Fiber content influences the expansion properties of the extruded cereals, which depend on the molecular weight and structure of the hydrocolloids.²² Increasing the BEFF level increases the viscosity of the blends, which may hinder moisture evaporation during extrusion and result in reduced expansion. The expansion behavior of different fiber types is influenced by their impact on mechanistic steps such as starch transformation, bubble nucleation, bubble growth, and bubble collapse.²³

The level of BEFF had a significant effect ($P < 0.05$) on porosity and hardness, with a linear effect on porosity and both linear and quadratic effects on hardness (Table 3). Incorporating BEFF into breakfast cereals resulted in more porous cereals (Supporting Information, Fig. S1(e)) with brighter colors, improving the textural properties of extrudates. However, the effects of BEFF on hardness were limited (Supporting Information, Fig. S1(f)). As the level of BEFF increased, the average pore area and β -glucan content increased (Supporting Information, Fig. S1(h), (i)) but starch digestibility decreased (Supporting Information, Fig. S1(j)). The BEFF level affected the average pore area and starch digestibility linearly and β -glucan content through both linear and quadratic effects ($P < 0.05$).

Nikinmaa *et al.*²² attributed the higher extractability of β -glucan to a more open structure in expanded extrudates of oat flour extruded by twin-screw extrusion. They concluded that the conditions that favor expansion yield a more open

structure with thinner cell walls, from which β -glucan is more readily extracted.

Effect of screw speed on product responses

The examination of process variables showed that screw speed influenced all product responses significantly, particularly the physical and textural properties, with both linear and quadratic effects. Supporting Information, Fig. S1(a) demonstrates that the extrudates had a lower L^* value at higher screw speeds. Low screw speed increased the residence time, increasing thermal energy input in a low-shear environment. Even with a short residence time, high temperatures and pressure during extrusion cooking can still produce colored products, as observed by Li *et al.*²⁴

Screw speed correlates with the expansion behavior and textural properties of extrudates. As screw speed increases, expansion ratio, porosity, and crispness values generally increase, whereas hardness and apparent density decrease as expected. A negative correlation was found between the expansion ratio and apparent density (-0.81 , $P < 0.01$), consistent with previous findings by Yağcı *et al.*⁵

Pressure in the extruder increases with screw speed, enhancing expansion and texture due to additional die pressure. Increased energy input from a higher screw shear rate positively affects extrudate expansion by accelerating bubble growth.²³

The effects of changes in screw speed on average pore area and total pore number were both linear and quadratic (Table 2). As screw speed increased to 500 rpm, average pore area decreased and the total pore number increased, indicating a more homogeneous pore distribution inside the extrudate structure. Microstructure analysis showed a positive correlation (0.62, $P < 0.01$) between pore number and porosity measured by the pycnometer, and a negative correlation (-0.62 , $P < 0.01$) with the average pore area.

At low screw speeds, the pore number was lowest (63) and pore area highest (8.02 mm²). Conversely, at high screw speeds, pore number was highest (110), and pore area lowest (2.98 mm²). Breakfast cereals containing 7% BEFF exhibited the lowest density (0.1363 g cm⁻³) and hardness (253.8 N). In contrast, the highest expansion ratio (4.02) and porosity (0.916) were observed at 500 rpm screw speed and 120 $^{\circ}\text{C}$ die temperature. Karlsson *et al.*²⁵ reported similar trends, showing that higher extrusion temperature and screw speed produced finer and more uniform pore structures, although some initial pore formation was already visible under lower processing conditions.

Effect of die temperature on product responses

Die temperature affected the structure and expansion of the extrudate significantly. Increasing die temperature resulted in a higher expansion ratio and crispness (Supporting Information, Fig. S1(k), (l)), whereas hardness and apparent density decreased (Supporting Information, Fig. S1(l)–(n)). The ANOVA results showed that the BEFF level, screw speed, die temperature, and the interaction between screw speed and temperature exerted linear and quadratic effects on L^* values (Table 2). The BEFF-incorporated extrudates exhibited L^* values of 50.65–71.74, a^* values of 3.15–6.27, and b^* values of 25.19–32.96. Unextruded blends exhibited L^* values of 77.04–78.10, a^* values of 8.70–11.07, and b^* values of 38.55–47.36. Extrudates were darker than unextruded blends due to color loss during twin-screw extrusion resulting from nonenzymic browning reactions that affected product appearance.

The brightness of the extrudates increased as the die temperature increased at low screw speeds. However, at higher screw speeds, the L^* value increased, up to 145 °C die temperature, decreasing slightly beyond that value (Supporting Information, Fig. S1(o)). Greater expansion resulted in brighter colors in extrudates due to air cells, particularly at 300 rpm, whereas Maillard or caramelization reactions caused greater darkening of extruded samples at higher screw speeds (500 rpm) with increasing die temperature.

Increasing die temperature also led to an increase in expansion, attributable to reduced melt viscosity and an increase in superheated steam, which produced a more puffed structure at the die.²⁵ These results are consistent with previous studies, including Zambrano-Zaragoza *et al.*,²⁶ who reported higher expansion ratios for oat flour-based breakfast cereals at higher temperatures (133 °C). When other barrel zone temperatures were constant, higher die temperature increased expansion and total pore number (linear effect: $P < 0.05$; second-order effect: $P < 0.01$), promoting more uniform pore distribution by shrinking pores and enhancing homogeneity.

Model fitting and optimization

Statistical validation of response surface models

The extrusion process for breakfast cereal production was optimized using a CCD design with three independent variables. Table 2 reports statistical values – including P , R^2 (coefficient of determination), R^2_{adj} (adjusted R^2), R^2_{pred} (predicted R^2), and PRESS – obtained from response surface quadratic models fitted to the experimental data. The quadratic model adequately described physical, textural, and nutritional properties, whereas *in vitro* starch digestibility was fitted using two-factor interactions. Higher order interaction terms were excluded as their effects were statistically insignificant. The inclusion of only two-factor interactions ensured model parsimony without compromising prediction quality. This was supported by the regression results (Table 3).

Model equations showed significant probability values with low P -values and nonsignificant lack of fit. Responses exhibited R^2 , R^2_{adj} , and R^2_{pred} values ranging from 0.7175–0.9828, 0.6645–0.9796, and 0.5300–0.9724, respectively. Coefficient of variation and adequate precision ranged from 1.43% to 15.73% and 13.56–67.75, respectively, with adequate precision values >4 considered desirable. Residuals behaved well, and the difference between R^2_{adj} and R^2_{pred} was <0.2 , confirming satisfactory model fit (Table 2). Overall, all coefficients were in reasonable agreement, and the RSM models were deemed acceptable, providing reliable predictions for practical application.

Numerical optimization and experimental model validation

A numerical optimization technique was applied to improve breakfast cereal quality using the desirability function of response surface methodology. The process considered BEFF level, screw speed, and die temperature as independent variables to maximize desirability, aiming for minimum hardness, maximum expansion ratio, and brightness (L^*), as these physical and textural properties strongly influence consumer preference.

Using the desirability function method, the optimal combination of BEFF level, screw speed, and die temperature was 51 g kg⁻¹, 435 rpm, and 170 °C (Supporting Information, Fig. S2). The resulting desirability value was 0.897. Predicted values for L^* value, expansion ratio, and hardness were 66.71, 3.80, and 139.4 N, respectively, closely matching the experimental values of 63.72, 3.71, and 159.8 N, with an average deviation of 6.0% from the predicted values. This model can thus guide extrusion conditions to achieve the required properties in BEFF-incorporated breakfast cereal. Although 187 °C was the highest temperature tested, the corresponding product ($L^* = 68.78$, expansion ratio = 3.71, hardness = 146.4 N, crispness = 4.07 mm⁻¹) did not outperform those at the optimal 170 °C conditions, which provided a more balanced response profile.

These results suggest that temperatures above 170 °C are unlikely to improve product quality and may even have a negative impact on certain properties. Goudar *et al.*²⁷ emphasized the importance of optimizing barley extrusion conditions to increase soluble β -glucan, recommending an extrusion temperature of 150–190 °C and feed moisture content of 14–16% to enhance β -glucan levels. The optimum extrusion conditions for breakfast cereal production in this study were consistent with those reported by Goudar *et al.*²⁷

Microstructural characteristics of BEFF-enriched optimum sample and control sample

The microstructures of both control (CS) and BEFF-containing (OS) breakfast cereal samples were examined using SEM to evaluate the morphological differences resulting from BEFF incorporation. Micrographs showed that the OS, with a higher BEFF and protein content, had a more compact and rough structure than the CS (as shown in Fig. 2). However, pore distribution and structure were more homogeneous and regular. This finding is consistent with Vadukapuram *et al.*,²⁸ who reported that flaxseed-incorporated extruded products had a more compact structure than the CS. In the present study, the OS wall was also thinner than that of the CS (Fig. 2), supporting the observed textural properties.

These results suggest that incorporating BEFF into breakfast cereals leads to a structure that is less hard and more porous. The increased fiber level may also have resulted in fewer but larger pores, indicated by reduced total pore number and increased average pore area, possibly due to the fiber's effect on nucleation behavior.²³ Previous studies reported that blending 30% oats with rice produced extrudates with higher porosity (91%), larger mean pore diameter (1267 μm), and thinner cell walls (63 μm), consistent with the current results. Extractable β -glucan content of oat-based extrudates was 11.4 g kg⁻¹.²²

Nutritional properties of the breakfast cereals

Tables 2 and 3 present the statistical and regression parameters for β -glucan content. Breakfast cereals contained 3.84 to 18.56 g kg⁻¹ of β -glucan (Supporting Information, Table S1). The equation

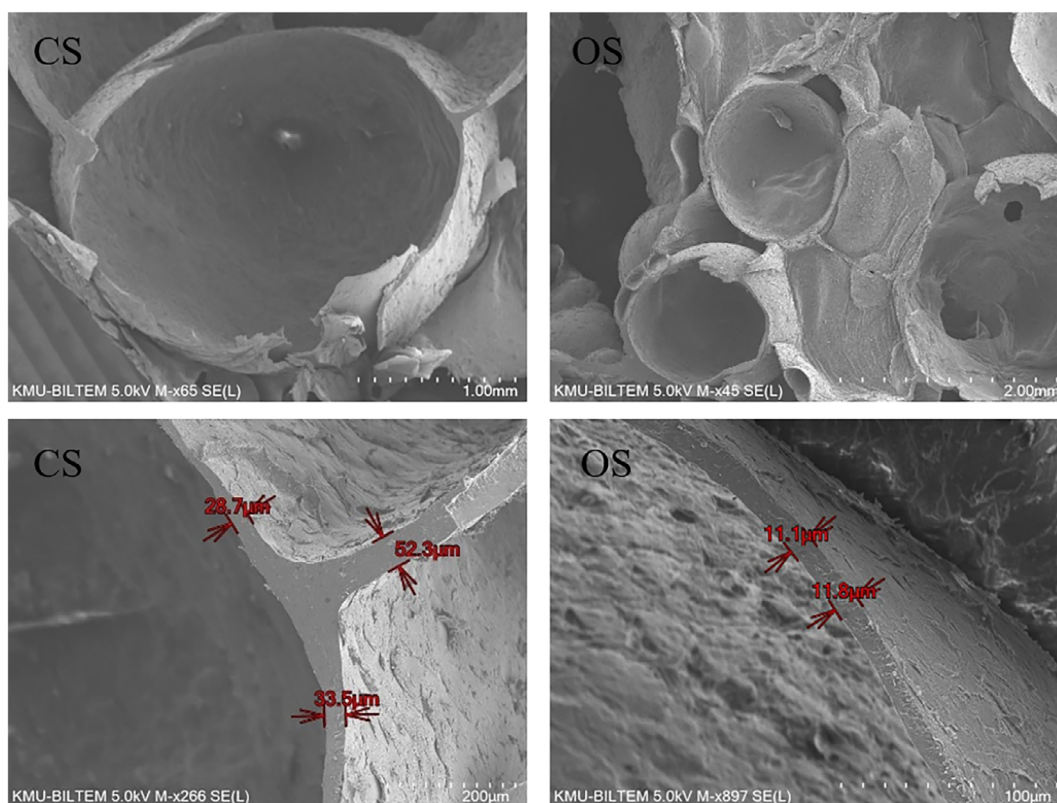


Figure 2. Scanning electron microscopy micrographs of the control sample (CS) and the optimum sample (OS).

in Table 2 indicates that BEFF exerts a positive linear effect on β -glucan, whereas screw speed and die temperature have a linear negative effect. Extrudates with higher screw speeds have a lower β -glucan content (Supporting Information, Fig. S1(p)). Unextruded blends also had a higher β -glucan content, ranging from 16.77 to 30.58 g kg⁻¹, than extrudates (Table 1). On average, β -glucan loss during extrusion was approximately 40%, with the highest loss at 568 rpm (46.81%) and the lowest at 300 rpm (32.05%). The reduction in β -glucan is attributed to shear-induced bond cleavage and elevated temperature during extrusion, which together decrease total β -glucan content.²⁷

This decrease may result from the breakdown of carbohydrate polymers and their conversion into other compounds. Processing conditions such as temperature, moisture content, and screw speed significantly influence β -glucan fragmentation.^{3,29} Although the initial blend moisture content was fixed at 170 g kg⁻¹ (db) in all formulations, fiber–moisture interactions during extrusion may have altered water distribution within the matrix, potentially affecting β -glucan degradation. Ge *et al.*²⁹ observed that β -glucan content in extruded highland barley flour depended strongly on the interactions of extrusion process parameters like feed moisture content and barrel temperature. These interactions were linked closely to shear forces, which affected the solubility of β -glucan and facilitated its dispersion.

Extrusion cooking subjects ingredients to high temperature and pressure, leading to complete starch gelatinization and greater availability to digestive enzymes.³⁰ The gelatinization properties of unextruded blends were assessed using DSC (Table 4). The control sample, containing only corn and wheat semolina, had a gelatinization temperature (T_p) of 78.46 °C, whereas samples with BEFF showed lower T_p values of 61.54–66.06 °C compared

with CS. This result is consistent with Altan *et al.*'s²⁰ report of a 68.5 °C gelatinization temperature for barley flour. In extruded samples, 100% starch gelatinization was observed, with no endothermic peak observed. The enthalpy of gelatinization (ΔH_g) decreased with BEFF incorporation into CS, from 0.59 mJ mg⁻¹ (0% BEFF) to 0.46 mJ mg⁻¹ (5.1% BEFF) and 0.25 mJ mg⁻¹ (8.36% BEFF). These findings align with previous research showing that certain polysaccharides reduce starch granule gelatinization, as reported by Altan *et al.*²⁰ and Ge *et al.*²⁹ Similarly, Symons and Brennan³¹ found that corn starch with 5% added barley β -glucan exhibited lower gelatinization than the control, and Tudorică *et al.*³⁰ observed that increasing fiber concentration reduced the enthalpy of the flour–fiber complex.

Starch digestibility in raw materials and unextruded blends varied significantly, ranging from 8.37 to 74.60 mg maltose g⁻¹ dry sample. In contrast, extruded samples showed a marked increase

Table 4. Differential scanning calorimetry (DSC) results of unextruded blends containing various BEFF levels

	T_o (°C)	T_p (°C)	T_e (°C)	ΔH_g (mJ mg ⁻¹)
CS	77.52	78.46	78.75	0.59
OS	65.25	66.06	66.23	0.46
C	60.51	61.14	61.54	0.25

Control sample (CS): 0% BEFF. Optimum sample (OS): 5.1% BEFF. Sample containing highest level of BEFF (C): 8.36% BEFF; T_o , onset temperature; T_p , peak temperature; T_e , endset temperature; ΔH , enthalpy change (mJ mg⁻¹).

Table 5. The chemical composition of the optimum sample

Chemical composition ^a	Measured values
Protein (g kg ⁻¹)	92.32 ± 0.03
Fat (g kg ⁻¹)	2.28 ± 0.13
Crude fiber (g kg ⁻¹)	6.13 ± 0.17
Ash (g kg ⁻¹)	10.41 ± 1.04
Moisture (g kg ⁻¹)	43.74 ± 5.12
Total starch (g kg ⁻¹)	675.90 ± 4.60
β-glucan content (g kg ⁻¹)	14.29 ± 0.18
Total dietary fiber (g kg ⁻¹)	52.4
Starch digestibility (<i>in vitro</i> , mg maltose g ⁻¹)	196.46 ± 0.19

^a All data are expressed on a dry weight basis. Results are given as means ± SDs.

in *in vitro* digestibility, ranging from 117.88 to 256.46 mg maltose g⁻¹ sample. According to Altan *et al.*,¹⁸ extrusion cooking substantially enhances the *in vitro* digestibility of barley flour. Regression analyses indicated significant linear effects of BEFF level, screw speed, and die temperature on *in vitro* starch digestibility ($P < 0.01$, Supporting Information, Fig. S1(s), (t)), along with a significant BEFF–screw speed interaction ($P < 0.05$; Table 3). Extrusion reduces starch crystallinity and increases enzyme availability, facilitating amyolytic degradation of starch molecules.^{15,32} The values obtained in the current study (170 °C, 400 rpm, Supporting Information, Fig. S1(s)) are consistent with previous findings that higher extrusion temperature and screw speed above 90 °C and 410 rpm cause greater damage to the starch structure.³² Higher screw speed increases shear rate, promotes starch swelling, and encourages starch gelatinization during extrusion.²³

Table 5 presents the composition of the OS, showing significant ($P < 0.05$) decreases in protein, fat, crude fiber, total starch, and β-glucan, accompanied by a significant ($P < 0.05$) increase in starch digestibility. The OS contains 52.4 g kg⁻¹ total dietary fiber, and its total starch content decreased with BEFF incorporation, reflecting the formation of insoluble fiber fractions during extrusion. The β-glucan content of the OS is 14.29 g kg⁻¹, representing a 42% loss during extrusion. Nevertheless, all breakfast cereal products in this study satisfied the FDA's requirement of 0.75 g of β-glucan per serving. Whole oat products such as oat bran, oat flour, and rolled oats can provide up to 3 g of soluble fiber daily, which can help reduce the risk of heart disease when consumed with a low-fat, low-cholesterol diet.^{4,6} Based on the β-glucan content (14.29 g kg⁻¹), a 52.5 g serving of the product provides 0.75 g of β-glucan.

CONCLUSIONS

This study aimed to optimize the extrusion process for breakfast cereals enriched with β-glucan. The independent variables in the five-level CCD were BEFF level, screw speed, and die temperature. Modeling was performed for L^* value, expansion ratio, and hardness, all of which affect consumer preferences. Optimization was carried out through variable testing and statistical analysis, yielding reliable models with accurate predictive power. The optimum process conditions were 5.1% BEFF, 435 rpm screw speed, and 170 °C die temperature. The screw speed had the greatest effect, followed by die temperature. Under these conditions, the

optimum product contained 92.3 g kg⁻¹ protein, 2.3 g kg⁻¹ fat, 6.1 g kg⁻¹ crude fiber, 10.4 g kg⁻¹ ash, 43.7 g kg⁻¹ moisture, 675.9 g kg⁻¹ total starch, 14.3 g kg⁻¹ β-glucan content, 14.3 g kg⁻¹ total dietary fiber, and 196.46 mg maltose g⁻¹ *in vitro*. All breakfast cereal products produced in this study had sufficient β-glucan content to meet the FDA requirement of 0.75 g of β-glucan per serving.

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DATA AVAILABILITY STATEMENT

All data generated or analyzed during this study are included in this published article and its Supporting Information files.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

SUPPORTING INFORMATION

Supporting information may be found in the online version of this article.

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