

Nutritional, Functional and Physical Impact of Fortification of Corn Extrudates with Cocoa Pod Husks and Brewer's Spent Grains: Effect of Extrusion Processing

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The incorporation of brewers' spent grains (BSG) for food applications has been widely explored due to their high protein, dietary fiber, and bioactive compounds. Additionally, pectin has been shown to improve the texture and expansion of fiber-based extrudates. Cocoa pod husk (CPH), a by-product of cocoa production, is a natural source of pectin, antioxidants, and fibers, yet its use in functional food manufacturing remains unexplored. Therefore, this research aims to explore the potential use of BSG and CPH as functional ingredients in corn-based extrudates. The optimized formulation (10.2 wt.% BSG, 9.8 wt.% CPH) extruded at high temperatures and 30% feed moisture resulted in high polyphenol content (1.855 mg GAE/g), antioxidant capacity (0.2542 μ M Trolox eq/g), WSI (15.479%), and WAI (0.8393 g/g). Additionally, the extrudate's low oxalate content (2.25 mg/100 g) ensures its nutritional safety. The combination of these by-products enhanced the functional and physicochemical properties of the extrudate, making it a promising ingredient for the development of health-oriented snacks. These findings highlight the potential of BSG and CPH as functional ingredients, improving the nutritional value of extruded products while contributing to food waste reduction. Future studies should assess sensory attributes and optimal formulations to maximize consumer acceptance.

Keywords: cocoa pod husk, brewer's spent grain, pectin, extrusion cooking, polyphenols, antioxidant capacity

1. Introduction

The brewing sector plays a significant strategic role in the economies of various countries. Approximately 1.89 billion hectoliters of beer are produced per year. However, substantial quantities of brewers' spent grain (BSG) are generated, with an average yield of 20 kg per hectoliter of beer. BSG accounts for approximately 85% of the total solid waste produced in brewing (Lynch et al., 2016). Due to its high moisture content and biological instability, physical and chemical pretreatments are required for storage and transportation, which may limit industrial investments in its processing. Consequently, BSG is primarily used as a low-value animal feed supplement, despite being a rich source of non-digestible polysaccharides with prebiotic potential to enhance microbiota activity (Lao et al., 2020). Furthermore, BSG is a lignocellulosic material with a composition rich in fiber (59.1-73%), proteins (15.2-24.0%), minerals, vitamins, and amino acids (<0.5%) (Bonifácio-Lopes et al., 2020). The development of novel technologies to exploit the nutritional properties of this by-product is crucial for minimizing waste generation in the agro-industry.

Previous studies have demonstrated that BSG can serve as a cost-effective source of bioactive compounds, enhancing the nutritional and rheological properties of functional foods. Schettino et al. (2021) reported increased antioxidant activity against oxidative stress in human colon carcinoma cells following the consumption of pasta fortified with fermented BSG. Similarly, Neylon et al. (2021) found that incorporating BSG and its derivatives into bread formulations strengthened the gluten network, improved dough resistance, and reduced

sugar release during starch digestion due to the beneficial effects of dietary fiber on glucose metabolism and insulin response. However, high levels of BSG adversely affect sensory attributes such as texture, expansion, color, and flavor (Nascimento et al., 2017). The inclusion of soluble fibers, such as pectin, has been proposed to enhance elasticity, thickening, and porosity during product expansion. Aćkar et al. (2018) addressed the problems of expansion and sensory quality of BSG extrudates by the addition of 1 wt.% pectin. Despite these findings, limited research on fiber-based extruded snacks attempts to improve the physical properties of extrudates.

Campos-Vega et al. (2018) identified cocoa pod husk (CPH) as a natural source of pectin, antioxidants, and minerals with potential protective effects against cardiovascular diseases, inflammation, oxidative stress, and cancer. Current research on CPH primarily focuses on optimizing pectin extraction techniques to improve process efficiency and product quality (Sarah et al., 2022). However, no efforts have yet been made to improve sensory attributes of extruded snacks by using CPH pectin.

Therefore, this study aims to assess the feasibility of incorporating BSG and CPH as functional ingredients in the formulation of a nutritionally enhanced extruded snack with acceptable sensory attributes. Extrusion technology represents a sustainable and economically viable method for processing flour and agro-industrial by-products, as it effectively reduces anti-nutrient content and biological risks while preserving the nutritional and functional quality of food products. This research evaluates the impact of extrusion on the physical and chemical properties of the final product to determine optimal processing conditions. The findings will provide a foundation for future studies on alternative formulations, sensory evaluations, and antioxidant stability during storage.

2. Materials and methods

Food by-products were added up to 20 wt.% on a corn grits basis, as an increased fiber content may lead to greater stiffness and reduced consumer acceptance (Korkerd et al., 2016). The overall process is illustrated in Figure 1.

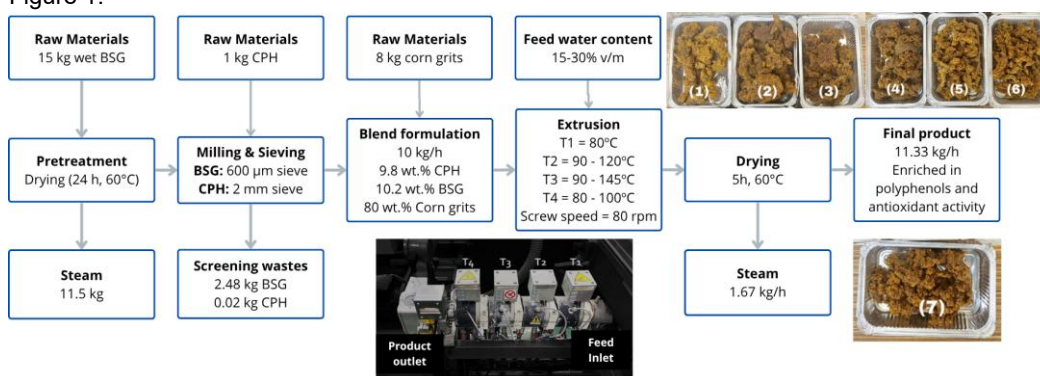


Figure 1: Schematic diagram of the proposed process.

2.1 Sample preparation

Cocoa pod husks (moisture 5.0 %), corn grits (moisture 10.95 %) and brewer's spent grains (moisture 77.7 %) were sourced from local suppliers in Ecuador. The percentages of BSG and CPH were chosen based on previous studies to avoid extrusion issues caused by excess fibre. Cocoa was added at 1% due to its pectin content, which supports cohesion during processing. Prior to extrusion, BSG was dried at 60°C in a tray dryer for 24 hours until reaching a moisture content of 3.3%. The CPH and BSG were ground using a Brabender Break Mill SM3. Subsequently, the CPH was sieved using an AdvanTech Dura Tap vibrating sieve with a 2 mm mesh, while the BSG was sieved using a 600 µm mesh to obtain fine particles in each case. All ingredients were stored at 19°C in sealed airtight bags to prevent moisture absorption before extrusion processing.

2.2 Extrusion processing

The sample formulation consisted of 9.8 wt.% CPH, 10.2 wt.% BSG, and 80 wt.% corn grits. Ingredients were homogenized before extrusion. A semi-industrial single screw extruder (Mapimpianti, Pavan Group, Galliera Veneta-PD, Italy) with a L/D ratio of 20:1 and a screw diameter of 55.7 mm was used. The extruder has a production capacity of 20 kg/h and is equipped with a water filter and an air compressor for pneumatic control and extrudate shaping. The system consists of four-barrel zones, a cooling section, and a die section, each with individual temperature control (Figure 2).

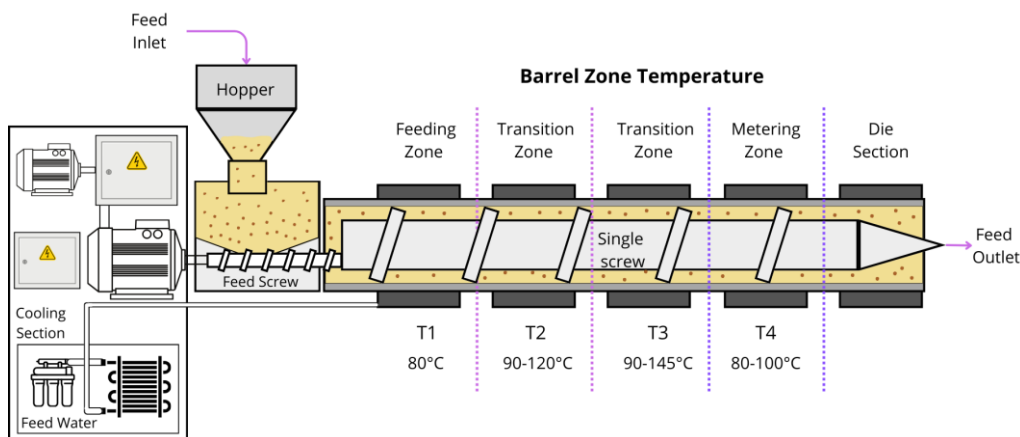


Figure 2: Schematic representation of the single-screw extruder and temperature profile used for processing.

The cooling and die sections were maintained at 70 °C and 100 °C, respectively. Screw speed was kept constant at 80 rpm. The temperature profile and feed water content were manually adjusted in each trial. Seven experimental runs were performed by varying the feed water level from 15 % to 30 %. Section T₁ was maintained at 80 °C across all trials, while T₂ and T₃ varied from 90 °C to 145 °C. Section T₄ was operated at either 80 °C (low temperature) or 100 °C (medium-to-high temperature) based on the trial. Table 1 presents the specific process conditions.

Table 1: Process conditions per experimental run

Samples	Feed water level (%)	Screw speed (rpm)	T ₁ (°C)	T ₂ (°C)	T ₃ (°C)	T ₄ (°C)	Temperature level
S1	15	80	80	90	90	80	Low
S2	30	80	80	90	90	80	Low
S3	15	80	80	90	90	100	Medium
S4	30	80	80	90	90	100	Medium
S5	15	80	80	120	120	100	High
S6	30	80	80	120	120	100	High
S7	30	80	80	120	145	100	Very High

Extrudates were cut using a rotary cutter at 42 Hz as they exited the die. The samples were collected in aluminum trays, dried in an oven for 5 hours, cooled, and stored in sealed containers covered with aluminum foil at 19°C until analysis.

2.3 Physicochemical analysis of samples

The physicochemical properties of the extruded samples were evaluated to assess their functional and nutritional characteristics.

2.3.1. Pectin extraction

CPH pectin was extracted via acid hydrolysis following the methodology described by Jarrín-Chacón et al. (2023). The sample was heated and stirred at 80 °C for 30 minutes. The extracted pectin was dried at 40°C for 48 hours and weighed to determine pectin content. The pectin level in the feed was standardized at 0.6 wt.% in a 10 kg feed blend.

2.3.2. Water absorption index (WAI) and water solubility index (WSI)

Dried and ground samples (~0.8 g) were placed in 15 ml centrifuge tubes with 10 ml distilled water and stirred for 10 minutes at 3000 rpm. The supernatant was collected, dried at 135°C for two hours, and weighed. The precipitate was also dried and weighed. WAI and WSI were calculated using Eq (1) and (2):

$$WAI (g/g) = \frac{\text{dry weight of precipitate}}{\text{dry weight of sample}} \quad (1)$$

$$WSI (\%) = \frac{\text{dry weight of solids in supernatant}}{\text{dry weight of sample}} \times 100 \quad (2)$$

2.3.3. Total phenolic content (TPC) and total antioxidant capacity (TAC)

TPC was determined using the Folin-Ciocalteu (FC) colorimetric method as described by Viteri et al. (2022), expressed as mg Gallic Acid Equivalent (GAE) per g dry sample. Antioxidant activity was assessed using the DPPH (1,1-diphenyl-2-picrylhydrazyl) radical scavenging assay according to Bhat et al. (2019) and expressed as μM Trolox Equivalent per g dry sample.

2.3.4. Oxalate content

Approximately 2 g of sample was mixed with 10 ml of 6 N HCl and 190 ml distilled water, heated in a water bath for 1 hour, and vacuum filtered. The filtrate was heated to 90 °C, and 10 ml of 5 % CaCl_2 solution was added to precipitate insoluble oxalates. The sample was then titrated with 0.05 M standardized KMnO_4 solution.

2.4 Statistical analysis

Experimental data on bioactive compound quantification and antioxidant activity were analyzed using a one-way ANOVA ($p < 0.05$), followed by Tukey's HSD test for mean comparisons. Different lowercase letters indicate statistically significant differences between groups. Analyses were conducted in RStudio (v4.4.1) using the *Agricolae* package.

3. Results and discussion

The pectin extracted from CPH before extrusion was used to determine the appropriate CPH level in the formulation, ensuring a pectin content of 6%. The pectin yield was determined as 6.12 % using hydrochloric acid. These findings align with Priyangini et al. (2018), who reported yields of 2 - 5.2 % using ascorbic acid. Higher yields were observed in chemical extractions, such as Hennessey-Ramos et al. (2021) with 8.08% using citric acid. These comparisons validate the extraction conditions on pectin yield used in this study.

3.1 Effects of extrusion on WAI and WSI

The WAI and WSI of the extruded samples were evaluated immediately after extrusion, using a control sample of corn grits and the raw mixture as references. The non-extruded corn grits exhibited a WSI of 13.45 % (± 1.28), while the extruded samples showed values ranging from 14.75 (± 0.03) to 16.90 % (± 0.79) (Figure 3a). The highest WSI was observed in sample S6, processed at a moisture content of 30 % and a high temperature (120 °C). This increase is likely due to the disruption of the starch crystalline structure by mechanical shear at elevated temperatures, facilitating the release of low-molecular-weight soluble polysaccharides bound to the fibrous matrix. Previous studies on extruded products containing 10 wt.% BSG reported a WSI of 6.8% (Ainsworth et al., 2007). This might indicate that the inclusion of CPH in the extruded blend may enhance soluble fiber content, improving water compatibility (Ačkar et al., 2018).

WAI analysis revealed that corn grits alone had a value of 0.701 g/g (± 0.067), whereas extruded samples exhibited higher values, ranging from 0.8082 (± 0.03) to 0.9193 (± 0.03) g/g (Figure 3b). Samples S4, S6, and S7 displayed the highest WAI, suggesting greater water-holding capacity at these processing conditions. This behavior is attributed to starch gelatinization, which enhances water interaction with amylose and amylopectin chains. However, these findings may also correlate with variations in viscosity and elasticity in expanded product formation, which is beyond the scope of this study. The influence of temperature on WAI values suggests that lower temperatures favor water retention, whereas higher temperatures may induce excessive starch gelatinization and expansion, increasing porosity and reducing water absorption capacity. As shown in Figure 3, no statistically significant differences were observed between treatments ($p > 0.05$).

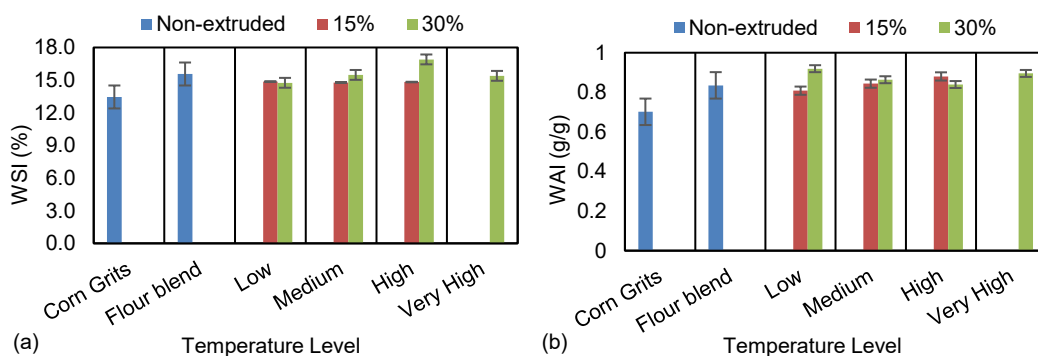


Figure 3: a) Water solubility index (WSI) and b) water absorption index (WAI) of non-extruded and extruded samples.

3.2 Effect of extrusion on TPC and TAC

The non-extruded corn grits contained a low phenolic content of 0.485 mg GAE/g, which aligns with values reported by Jozinović et al. (2021), whose result (0.6138 mg GAE/g) decreased when the raw material was processed at very high temperatures (over 170°C) due to mechanical shearing action (0.4839 mg GAE/g). As shown in Table 2, the non-extruded blend containing CPH exhibited a higher TPC (2.05 mg GAE/g). However, after extrusion, TPC decreased by 21.1 - 27.7 %, likely due to thermal degradation and structural alterations of phenolic compounds, reducing their antioxidant activity. The TAC of the extruded samples ranged from 0.2218 to 0.2888 μM Trolox eq/g.

Table 2: Total phenolic content (TPC) and total antioxidant capacity (TAC) of non-extruded and extruded samples. (Values with different letters within the same column are significantly different at $p < 0.05$)

Sample	TPC (mg GAE/g)	TAC (μM Trolox eq/g)
Non-extruded corn grits	0.485 ^a	0.083 ^a
Non-extruded blend	2.050 ^b	0.349 ^b
S1	1.526 ^c	0.2218 ^c
S2	1.533 ^c	0.2607 ^d
S3	1.482 ^c	0.2548 ^d
S4	1.620 ^{bc}	0.2585 ^d
S5	1.589 ^{bc}	0.2888 ^e
S6	1.525 ^c	0.2634 ^d
S7	1.855 ^{bc}	0.2542 ^d
STD	0,406	0,067

3.3 Effect of extrusion on oxalate content

According to the Academy of Nutrition and Dietetics, individuals with kidney stones should limit their dietary oxalate intake to 40–50 mg per day. In this study, the oxalate content of the extruded mixture was measured, yielding 2.25 mg/100 g of product. This indicates that the extruded flour poses no health risk, as its oxalate contribution remains well below the recommended intake limits. The potential decrease in oxalate content could be influenced by processing conditions such as the high temperature and mechanical shear typical of extrusion, as reported in other studies. Kaur et al. (2015) reported that processing at temperatures between 115°C and 140°C, particularly at moisture levels above 20%, effectively reduces antinutritional factors. Similarly, Nkesiga et al. (2022) found that extruded ready-to-eat baby foods exhibited a notable reduction in oxalate content (0.16 - 0.50 mg/100 g), reinforcing the role of extrusion in producing nutrient-dense foods with lower antinutrient levels. In contrast, Ainsworth et al. (2007) reported that extrusion did not reduce antinutritional factors in extrudates made with BSG, chickpea, and maize flour, despite increased mechanical energy and shear rates. This discrepancy suggests that the effectiveness of extrusion in reducing antinutritional factors may depend on the specific compound and raw material composition. In the present study, a direct comparison of oxalate reduction was not possible due to the absence of pre-extrusion measurements, highlighting the need for further investigation into the influence of raw material composition on the retention or degradation of different antinutritional factors during extrusion.

4. Conclusions

This study demonstrated that the inclusion of BSG (10.2 wt.%) and CPH (9.8 wt.%) in corn grits-based extrusion formulations enhanced the nutritional profile of the final extrudates. The process resulted in a significant increase in polyphenol content (up to 1.855 mg GAE/g) and antioxidant capacity (0.2888 μM Trolox eq/g), surpassing values reported for conventional corn-based products. Additionally, a higher water solubility index (WSI: 15.48 %) and water absorption index (WAI: 0.8393 g/g) were observed due to the presence of CPH, which likely contributed to increased soluble fiber content. The extrusion conditions effectively preserved bioactive compounds, achieving a retention rate of 90.5%, particularly under high moisture content (30%), which mitigated the thermal and mechanical degradation of phenolic compounds. Furthermore, the oxalate content in the extruded product (2.25 mg/100 g) remained well below dietary risk thresholds, ensuring its safety for consumption. Therefore, the recommended processing condition is S6 — 30% moisture, 80 rpm screw speed, and a high-temperature profile of 80/120/120/100 °C in sections T1–T4 —. Future research should focus on sensory analysis to evaluate consumer acceptability and optimize formulations to balance nutritional, textural, and functional properties. Additionally, comprehensive economic and environmental assessments should be conducted to determine the industrial feasibility and sustainability of incorporating these by-products into extruded food products.

Nomenclature

BSG – brewer's spent grain, -

CPH – cocoa pod husk, -

T_i – barrel temperature of section i, °C

TAC – total antioxidant capacity, µM Trolox eq/g

TPC – total phenolic content, mg GAE/g

WAI – water absorption index, g/g

WSI – water solubility index, %

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