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Effect of extrusion on folic acid concentration and mineral element dialyzability in Great Northern beans (*Phaseolus vulgaris* L.)

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ABSTRACT

Great Northern beans (GNB) contain appreciable magnesium (Mg), potassium (K), phosphorus (P), and iron (Fe), together with the heat-labile vitamin, folate, and the anti-nutritional compound phytate. Thus, the objective was to increase dialyzability of essential mineral elements while degrading phytate and minimizing destruction of folate through extrusion of GNB. Extrusion resulted in significant ($p < 0.05$) increases in dialyzability of Mg, P, K, and Fe by as much as 50%, 30%, 5%, and 79%, respectively, while decreasing cadmium (Cd) dialyzability. Screw speed (SS) had a significant quadratic effect on dialyzability of all elements. Low MC resulted in a significant reduction (46%) in phytate, although this was accompanied by as much as 24% destruction of folate. In conclusion, low barrel temperature, medium MC and high SS were identified as the optimum conditions to maximize essential mineral element dialyzability and folate retention while minimizing phytate and dialyzable Cd.

1. Introduction

Despite the availability of food together with fortification, supplementation, and enrichment, there are several micronutrients, including magnesium (Mg), potassium (K), choline, calcium (Ca), iron (Fe), and vitamins A, D, E, and C, that are under-consumed in the US (U.S. Department of Health and Human Services, 2015). Additionally, while folate deficiency is nearly non-existent in the US, the major source of folate is fortified, processed food, which can have a negative connotation (Odewole et al., 2013). Individuals with chronic moderate deficiencies of these essential nutrients can develop conditions like hypertension, coronary heart disease, diabetes, and metabolic syndromes that are common in the US (Long & Romani, 2015). Thus, it is important to promote the consumption of food sources that are naturally rich in under-consumed micronutrients to tackle nutrient deficiencies and encourage healthy eating habits.

Typically, dry beans (*Phaseolus vulgaris* L.), such as Great Northern beans (GNB), are valued from a nutritional standpoint for their high protein and dietary fiber contents. However, they are also good sources (i.e., > 10% of the US Daily Value) of folate and the essential minerals Mg, Fe, K, and phosphorus (P) (Office of Dietary Supplements and National Library of Medicine, 2018; U.S. Department of Agriculture, 2018), making dry beans an excellent source of many under-consumed nutrients in the US diet.

However, the availability of these nutrients for absorption is not only dependent on their concentrations, but also on factors like anti-nutritional compounds, processing techniques, and physicochemical state of the nutrient (Fairweather-Tait, 1993). For example, phytate is a naturally occurring compound present in grains and legumes, including GNB, and can form insoluble complexes with essential elements and reduce their absorption (Thompson, 1993). Different processing techniques like germination, pressure-cooking, and extrusion have been shown to reduce phytate, but the extent of reduction depends on the raw material and processing technique (Nergiz & Gokgoz, 2007).

Unfortunately, the techniques adapted for reducing these anti-nutritional factors and increasing mineral element bioavailability can adversely affect other important nutrients like the heat labile folates. Additionally, while heavy metals are typically not abundant in GNB, the same processes that increased bioavailability of essential mineral elements may also increase bioavailability of toxic heavy metals like cadmium (Cd) (Watzke, 1998). Thus, it is important to identify processing techniques that can enhance the bioavailability of essential mineral elements while minimizing loss in important labile vitamins and reducing bioavailability of heavy metals. One such processing technique may be extrusion.

Extrusion has shown promising results in improving the bioavailability of mineral elements, and reducing anti-nutritional compounds in legumes and cereal products (Alonso, Rubio, Muzquiz, & Marzo, 2001;

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Hazell & Johnson, 1989). However, no research has simultaneously focused on the effect of extrusion on vitamins and bioavailability of essential mineral elements and heavy metals in dry beans.

Bioavailability is a complex process that is defined as the amount of an ingested nutrient that is absorbed and is available for physiological function while bio-accessibility is the amount of an ingested nutrient that has the potential to be absorbed and utilized (Etcheverry, Grusak, & Fleige, 2012). *In vitro* dialyzability measures the proportion of the total elements that diffuse through a membrane during food matrix digestion (Miller, Schricker, Rasmussen, & VanCampen, 1981) and is commonly employed to measure the bio-accessibility of mineral elements. This method can also be used as a screening tool to assess if a certain process would have an effect on bioavailability when moved to more complex models.

Thus, the objective of this study was to determine the effect of extrusion on degradation of folate and dialyzability of Mg, Fe, K, P, Fe, and Cd from GNB flour and identify conditions that achieve maximum dialyzability of the essential mineral elements while minimizing dialyzability of heavy metals and degradation of folate. The results from this study can serve as the basis for increasing bioavailability of essential nutrients from processed GNB flour to promote dry beans in the diet.

2. Materials and methods

2.1. Materials

Great Northern beans (GNB) were obtained from FNJ Inc. (Alta Loma, CA, USA) and milled using a pilot scale hammer mill (20SSHMBD, C.S. Bell, Tiffin, OH, USA) with screen size of 0.7 mm. The flour was analyzed for moisture, fat, and ash using approved methods (AACC International, 2018). Protein content was analyzed using a nitrogen analyzer (FP 528, Leco, St. Joseph, MI, USA) with a protein factor of 6.25. Total starch content was analyzed using a total starch assay kit (K-TSTA, Megazyme, Bray, Ireland) following the KOH format. GNB flour was stored at 4 °C until extrusion.

2.2. Experimental design

The effect of extrusion on GNB flour was studied by varying three extrusion factors: barrel temperature [Temp (90–140 °C)], feed moisture content [MC (17–25%)], and screw speed [SS (156–250 rpm)], while keeping other factors such as feed rate and screw configuration constant. The levels of these factors were determined based on preliminary trials. The commonly used central composite rotatable design (CCRD) was used, except Temp was not randomized during the experiment due to equipment operating constraints. Thus, experiments were conducted in increasing order of Temp while randomizing MC and SS. Temp was tested at three fixed levels: −1, 0, +1 (i.e., 90, 115 and 140 °C), respectively. In order to maintain orthogonality of the experiment, the design space was rotated by 45°, which resulted in new axial (± a = 1.43) and factorial (± b = 0.70) points for MC (+a = 25%; −a = 17%; +b = 22.5%; −b = 19%) and SS (+a = 250 rpm; −a = 156 rpm; +b = 225 rpm; −b = 180 rpm). The new values for MC and SS were calculated based on the ratio of block error variance and experimental error obtained during preliminary experimentation (Draper & John, 1998). Based on the design, extrusion was carried under 18 experimental conditions (MC, SS, Temp): ± a, 0, 0 (2 runs); 0, ± a, 0 (2 runs); ± b, ± b, ± 1 (8 runs); and 0, 0, 0 (6 runs) (Table 1).

2.3. Extrusion process

To adjust the MC of GNB flour, batches (2 kg) representing each experimental run were blended in an upright mixer (H-600-D, Hobart, Troy, OH, USA) at medium speed with the required water to obtain the target moisture content according to the experimental design. The samples were then sealed in polyethylene bags and tempered for 16 h at 4 °C. The GNB flour was then fed into the extruder barrel using a single screw volumetric feeder (FW 40 Plus, C. W. Brabender, Hackensack, NJ, USA) set at a constant delivery rate of 76 g/min.

A laboratory scale co-rotating conical twin screw extruder with mixing zones was used for extrusion (CTSE-V, C.W. Brabender, Hackensack, NJ, USA). The specifications of extruder and operating conditions used were the same as described in Gulati, Weier, Santra, Subbiah, and Rose (2016).

The extrudate sample for each experimental condition was collected after a stable temperature and torque reading was observed. The collected samples were dried in a belt drier (4800 series Wenger, Sabetha, KS, USA) at 100 °C for 10 min and ground using cyclone sample mill (UDY, Fort Collins, CO, USA) with a screen size of 1 mm. The ground extruded samples were stored at 4 °C until analysis.

2.4. Phytic acid content

Phytic acid in GNB flour and extrudates was quantified as phytate phosphorus using 2,2′-bipyridine as described (Haug & Lanstzsch, 1983), with slight modifications. Briefly, phytic acid was extracted from the sample (250 mg) using 0.2 N HCl (10 mL) overnight at 4 °C with gentle shaking. The contents were centrifuged and the supernatant was used for analysis after dilution with distilled water (25 mL). For unprocessed flour, 0.25 mL of the diluted supernatant was mixed with 0.75 mL of 0.2 N HCl; for extrudates, 0.5 mL of the diluted supernatant was mixed 0.5 mL of 0.2 N HCl. One mL of 415 μM $Fe(NH_4)(SO_4)_2$ (Sigma-Aldrich. St. Louis, MO, USA) was then added to diluted extracts and tubes were placed in a boiling water bath for 30 min. The tubes were cooled immediately and contents centrifuged. One mL of the supernatant was mixed with 1.5 mL of 2, 2′-bipyridine. The color was measured at 530 nm. The samples were quantified by means of external calibration using sodium phytate dodecahydrate (Sigma-Aldrich, 71649) which contained 19% phytate phosphorus as measured with inductive coupled plasma mass spectrometry (ICP-MS) as described later (Section 2.7).

2.5. Folic acid content

Total folates were measured using the standard microbiological assay (*L. casei* subsp. Rhamnosus, ATCC no. 7469) with the tri-enzyme extraction technique (AACC method 86-47; DeVries, Keagy, Hudson, & Rader, 2001). The analysis was conducted on replicated sample submissions by a commercial lab (NP Analytical Laboratories, St. Louis, MO, USA).

2.6. In vitro digestion

In order to measure the dialyzability of elements, bean flour was digested under the *in vitro* conditions described by Luten et al. (1996), with some modifications. The modifications were to reduce sample weights and volumes to fit in a 48-well dialysis plate format (Rapid Equilibrium Dialysis Plate, MWCO 8 K Dalton, Thermoscientific, 90006, Waltham, MA, USA). For digestion, 20 mg of sample was weighed in the sample chamber and mixed with 0.2 mL of pepsin (284 units/mg, P7000, Sigma-Aldrich) solution (50 mg/mL in 50 mM HCl). The plate was covered with a sealing tape (15036 ThermoScientific), and incubated at 37 °C for 2 h with gentle shaking at 125 rpm. Pepsin digestion was stopped by adding 0.25 mL of dialysis buffer (0.1 M $NaHCO_3$) in the buffer chamber and the mixture incubated for 55 min under previously described conditions. The amount of dialysis buffer added was pre-determined by titrating the gastric mixture with dialysis buffer until the pH reached 6. Meanwhile a pancreatin-bile solution (0.4 g pancreatin; 2.5 g bile salts) was prepared in 10 mL of 0.1 M $NaHCO_3$. Given the low solubility of pancreatin, the solution was centrifuged and