1 **Modeling the effects of microfluidization conditions on properties of corn bran**

2 Fuli He, Tao Wang, Si Zhu, Guibing Chen^{*}

3
4 Center for Excellence in Post-Harvest Technologies, North Carolina A&T State University, The
5 North Carolina Research Campus, 500 Laureate Way, Kannapolis, NC 28081, U.S.A.

6
7 ^{*}Corresponding author:

8 Tel.: 1-704-2505701; Fax: 1-704-2505709

9 E-mail: gchen@ncat.edu

Abstract

Corn bran was microfluidized through a 200-micron channel in the pressure range of 124.1 to 158.7 MPa for 1-5 passes following the central composite experimental design. Physicochemical properties and antioxidant properties of microfluidized bran samples were measured and fitted to the second order polynomial model. The response surface equations obtained showed that all the properties examined had a positive linear relationship with pressure and a negative quadratic relationship with number of passes except for ABTS radical scavenging activity which was quadratically related to both processing parameters. The number of passes generally had a more pronounced effect on the examined properties compared with pressure. Within the experimental range, the maximum values of swelling capacity, water-holding capacity, and oil-holding capacity were respectively 10.62 ml/g d.w. (at 158.7 MPa), 5.49 g water/g d.w. (at 158.7 MPa), and 4.61 g oil/g d.w. (at 124.1MPa); the maximum values of surface reactive phenolic content, DPPH and ABTS radical scavenging activities were 148.80 mg/FAE g d.w. (at 158.7MPa), 50.02 $\mu\text{mol TE/g d.w.}$ (at 158.7 MPa), and 47.90 $\mu\text{mol TE/g d.w.}$ (at 145.9MPa), respectively. All maximum values of the properties occurred at 5 passes.

Keywords: Microfluidization; corn bran; pressure; number of passes

41 **Abbreviations**

42	ABTS	2,2'-azino-bis(3-ethylbenzothiazoline-6-sulphonic acid)
43	AP	adequate precision
44	CS	calculated specific surface area
45	CV	coefficient of variation
46	DPPH	1,1-diphenyl-2-picrylhydrazyl radical
47	FAE	ferulic acid equivalents
48	MV	mean volume diameter
49	OHC	oil-holding capacity
50	PSD	particle size distribution
51	SC	swelling capacity
52	SRPC	surface reactive phenolic contents
53	SS	sum of squares
54	TE	Trolox equivalents
55	TEAC	Trolox equivalent antioxidant capacity
56	TROLEX	6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid
57	WHC	water-holding capacity

1. Introduction

Corn bran is an ideal source of dietary fiber and phenolic antioxidants for human diets. Numerous studies support that sufficient intake of dietary fiber plays a protective role against obesity (Slavin, 2005) and many other chronic diseases (Anderson et al., 2009). Corn bran contains the highest amount of ferulic acid, a potent phenolic antioxidant, compared to other tested grains (Vitaglione et al., 2008; Zhao and Moghadasian, 2008). Phenolic antioxidants can modulate cellular oxidative status and prevent biologically important molecules such as DNA, proteins, and membrane lipids from oxidative damage (Yu et al., 2002). In addition, unlike other cereal brans, high-fiber diets containing corn bran are less likely to cause discomfort due to its lower level of intestinal gas excretion (Marthinsen and Fleming, 1982).

It has been shown that suitable processing technologies could effectively improve physiochemical and health-related properties including hydration properties, oil holding capacity, antioxidant activity, etc., of various types of dietary fiber and cereal bran (Chau et al., 2006; Chou et al., 2008; Wang et al., 2013, 2014a). Such enhancement is of great interest for a few reasons. First, the same health benefits may be achieved by consuming less amount of dietary fiber. This is favorable because consuming large quantities of dietary fiber increases gas production and also retards intestinal gas transit (Gonlachanvit et al., 2004). Second, it facilitates development of foods fortified with fiber ingredients because adding a high level of dietary fiber to foods adversely affects color, texture, flavor and taste of the supplemented foods (Onwulata, 2008; Robin et al., 2012). Finally, most ferulic acids in cereal brans are bound to the fiber matrix, leading to poor bioaccessibility (Saulnier et al., 1995). Suitable processing technologies can reduce particle size and/or loosen the microstructure of cereal bran and thus increase bioaccessibility of bound phenolic compounds (Wang et al., 2014b).

Microfluidization is a process that applies a high pressure to drive a liquid stream carrying solid particles (suspensions) or liquid droplets (emulsions) through a microchannel in the interaction chamber of a microfluidizer. During the process, the carried particles or droplets are subjected to extremely high shear stress, impact force, and instantaneous pressure release, resulting in particle size reduction and loosened microstructure of solid particles. Microfluidization process has been used in pharmaceutical industry for preparing emulsions and encapsulation and in food industry for homogenization and deagglomeration (An et al., 2012; Guraya and James, 2002; Han et al., 2014). Recently, we demonstrated that this process dramatically improved physiochemical properties and antioxidant activity of corn bran primarily because it reduced bran particle size and loosened microstructure of bran matrix, making more reactive groups accessible to reagents present in an aqueous medium and more internal pore spaces in bran particles available for holding liquids such as water and oil (Wang et al., 2013, 2014a). Since processing pressure and number of passes may contribute differently to particle size reduction and porosity increase, they may affect properties of corn bran in different ways during a microfluidization process. Such information is important in optimizing a processing condition to achieve desired properties of corn bran with minimum operating costs.

The objective of this study was to correlate physiochemical and antioxidant properties of corn bran with processing pressure and number of passes during microfluidization processes, and to determine processing conditions for the maximum values of these properties within the range of experimental conditions.

2. Materials and Methods

2.1. Materials and chemicals

Fine wet milled corn bran samples comprising 3.5% fat, 82.4% total dietary fiber, 9.3% water, and 4.8% others were purchased from Cargill Dry Corn Ingredients, Inc. (Paris, IL, USA). All the chemicals and reagents were of analytical grade and purchased from Sigma-Aldrich (St. Louis, MO, USA).

2.2. Preparation of microfluidized corn bran

An M-110P Microfluidizer Processor (Microfluidics, Newton, MA) was used for the process. Raw corn bran was repeatedly ground using a Waring laboratory blender (model MX-LB10S) until passing through a US standard No. 35 sieve with a nominal opening of 500 μm (Fisher Scientific Co. TX, USA). Ground corn bran was dispersed in distilled water at a ratio of corn bran: water 1:9 (wt/wt). The suspension was then processed through a 200 μm interaction chamber, at room temperature, 124.1 - 158.7 MPa pressure, and for 1-5 passes according to the experiment design. The processed bran samples were collected by filtration and freeze-dried using Labconco FreeZone Freeze Dry System (Kansas City, MO, USA). Dry samples were sealed in air-tight glass containers and stored at -30 °C for analysis. All experiments were performed in triplicate. About 150 grams of raw corn bran were processed per run.

2.3. Experimental design

In order to investigate the effects of microfluidization conditions on properties of corn bran, a total of 13 experimental runs with different combination of processing pressure (X_1 , 124.1 - 158.7 MPa) and number of passes (X_2 , 1-5) were conducted based on the central composite design (CCD) (Table 1). The predicted responses of properties examined were calculated using a second-order polynomial model shown by Eq. (1):

$$Y = b_0 + \sum_{i=1}^2 b_i X_i + \sum_{i=1}^2 b_{ii} X_i^2 + \sum_{i=1}^2 \sum_{j=i+1}^2 b_{ij} X_i X_j \quad (1)$$

Where Y is the response variable, b_0 , b_i , b_{ii} , and b_{ij} are the constant, linear, quadratic, and cross-product regression coefficients, respectively, and X_i and X_j are the coded independent variables of X_1 and X_2 , respectively.

Software Design Expert 9.0.3 (Stat Ease, USA) was used for analysis of variance (ANOVA), regression analysis of the data, and plotting of response surface figures.

2.4. Determination of particle size distribution

Particle size distribution (PSD) was analyzed using a Bluewave Laser Particle Analyzer (Microtrac, Montgomeryville, PA, USA). The values of mean volume diameter (MV) and calculated specific surface area (CS) were recorded.

2.5. Measurement of bulk density

Five hundred mg of corn bran sample was weighed and carefully transferred into a calibrated 10 ml graduated cylinder. Then the cylinder was gently tapped on a bench-top until no further decrease in sample volume. The bulk density (g/ml) was calculated as weight (g) per volume of sample (ml).

2.6. Swelling capacity

Swelling capacity (SC) was determined using a reported method (Robertson et al., 2000). Weighed dry bran sample (0.500 ± 0.001 g) was added into a 20 ml graduated cylinder. 10 ml distilled water was poured into the cylinder and gently mixed well with the bran sample. The cylinder was then covered with parafilm and let to stand overnight at room temperature. The volume occupied by the settled sample was recorded. The SC was expressed as volume of swollen sample per gram dry sample weight (ml/g d.w.).

2.7. Water-holding capacity

Water-holding capacity (WHC) was determined according to a reported method (Robertson et al., 2000) with some modifications. In brief, corn bran sample (0.500 ± 0.001 g) was weighed in a 50 ml centrifuge tube; 20 ml of distilled water was added and allowed to hydrate at room temperature for 24 h. Then samples were centrifuged (3,000 g) for 10 min and the supernatant was carefully removed. WHC was expressed as the amount of water retained per gram dry sample weight (g water/g d.w.).

2.8. Oil-holding capacity

Oil-holding capacity (OHC) was determined by following the procedure for WHC test described above with water being replaced by corn oil and expressed as: g oil/g d.w.

2.9. Surface reactive phenolic contents

Surface reactive phenolic content (SRPC) of ground raw and microfluidized corn bran was assessed using a direct procedure described in our previous study (Wang et al., 2014a). Briefly, Folin-Ciocalteu reagent (2.5ml, 10% v/v) was added to 2.5 mg of corn bran sample, and sodium carbonate (2 ml, 7.5% w/v) was added after 5min. The mixture was incubated for 2h at room temperature, being vortexed several times during the incubation, and then centrifuged at 10,000 rpm for 15 min. The absorbance of the supernatant was measured at 725 nm wave length using a UV-Vis spectrophotometer (model 2500, Shimadzu Co. Ltd., Kyoto, Japan). Trans-ferulic acid was used as a standard and results were expressed as mg of trans-ferulic acid equivalents per gram of dry weight (mg FAE/g d.w.).

2.10. Antioxidant activities

The antioxidant activities of the treated corn bran were determined with DPPH and ABTS method.

1,1-diphenyl-2-picrylhydrazyl radical (DPPH) scavenging activity was measured following a modified direct procedure (Wang et al., 2014a). DPPH radical was prepared freshly in 80% of aqueous methanol at 64 μ M. 2 mg of corn bran was mixed with 6 ml DPPH reagent. The absorbance of the supernatant was recorded at 515 nm after 2 h reaction in dark; tubes were centrifugation at 10,000 g for 5 min before reading. The results of antioxidant activity were expressed in terms of 6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid (Trolox) equivalents/gram of corn bran on a dry basis (TEAC, μ mol TE equiv. /g d.w.).

A similar procedure was followed for the measurement of 2,2'-azino-bis(3-ethylbenzothiazoline-6-sulphonic acid) (ABTS) scavenging capacity (Wang et al., 2014a). The ABTS reagent was generated in the dark at room temperature by reacting a 7 mmol/L ABTS aqueous solution with 2.45 mmol/L potassium persulfate for 16 h. The resulting radical solution was diluted with ethanol to give an absorbance reading of 0.7 at 734 nm. 2.5 mg corn bran sample was added and absorbance was measured again after a reaction time of 60 min. The results were also expressed as μ mol TE equiv. /g d.w.

2.11. Sensitivity analysis

The slope of a response surface in the direction of a processing parameter at a given point was calculated by taking the partial derivative of the corresponding response surface equation with respect to the processing variable.

2.12. Statistical analysis

All experimental results were reported as mean values (n=3) with their corresponding standard deviations. ANOVA was carried out to determine any significant differences ($p < 0.05$) by IBM SPSS 19.0.

3. Results and discussion

3.1. Particle size distribution and bulk density

Table 1 listed the mean volume particle size (MV), calculated specific surface area (CS), and bulk density of microfluidized and untreated (control) corn bran. Microfluidization process dramatically reduced MV and bulk density and increased CS of corn bran. This was in a good agreement with our previous findings (Wang et al., 2013). Both processing pressure and number of passes contributed significantly to the changes of MV and CS ($p < 0.05$). However, the value of bulk density was affected only by the number of passes based on response surface analysis (data not shown).

3.2. Model fitting

Physicochemical and antioxidant properties (response variables) of corn bran microfluidized under different processing pressure and number of passes (independent variables) are given in Table 3. Tables 4 and 5 show the results of fitting the second-order polynomial model to the experimental data using ANOVA and regression analysis. In these tables, low variation coefficient (CV) values (1.40 - 3.43%) and high adequate precision (AP) ratios (21.296 - 37.189) indicated high precision and reliability of the experiment (Alizadeh Bahaabad et al., 2014). All models were fitted satisfactorily to the data as indicated by the insignificant lack-of-fit ($p > 0.05$) and were highly significant ($p < 0.0001$). Models for SC, WHC, OHC, and SRPC exhibited excellent predictability ($\text{pred-}R^2 > 0.9$), whereas models for DPPH and ABTS radical scavenging

capacity had reasonably good predictability. For these reasons, the established response surface models were validated and could be used to predict the responses. The models were given in Eqs. (2) - (7) and were also presented as response surface plots in Fig. 1.

$$Y_{SC} = 9.71 + 0.13X_1 + 0.71X_2 - 0.14X_2^2 \quad (2)$$

$$Y_{WHC} = 5.13 + 0.066X_1 + 0.25X_2 - 0.043X_2^2 \quad (3)$$

$$Y_{OHC} = 4.30 + 0.058X_1 + 0.26X_2 - 0.093X_1X_2 - 0.079X_2^2 \quad (4)$$

$$Y_{SRPC} = 130.57 + 4.08X_1 + 11.53X_2 - 1.92X_2^2 \quad (5)$$

$$Y_{DPPH} = 46.03 + 1.09X_1 + 2.51X_2 - 0.55X_2^2 \quad (6)$$

$$Y_{ABTS} = 45.40 + 1.11X_1 + 2.77X_2 - 0.96X_1^2 - 0.87X_2^2 \quad (7)$$

3.3. Response surface analysis of physicochemical properties

The effect of microfluidization conditions on hydration properties (SC and WHC) and OHC of corn bran were depicted by the second-order polynomial Eqs. (2) - (4) as well as by response surface graphs (Figs. 1a – 1c).

Table 2 shows the results for sensitivity analysis of Eqs. (2) - (4) with respect to processing pressure (X_1) and number of passes (X_2), respectively, at five selected points. As can be seen from the table, the absolute value of the slope with respect to number of passes was greater than that with respect to processing pressure at each point examined. This indicated that number of passes exerted a more profound impact on physicochemical properties than pressure within the experimental range.

Hydration properties including SC and WHC are among the most studied properties of dietary fiber. They are influenced by the chemical structure of the component polysaccharides and other factors, including particle size, surface area and porosity (Fernández-López et al., 2009; Mora et al., 2013). Corn bran with high hydration capacity may produce the effect of satiety and increased fecal bolus (Kuan and Liong, 2008), and can also improve moisture retention and slow down starch retrogradation in formulated foods especially bakery products (Abdul Aziz et al., 2012; de Escalada Pla et al., 2007; Wani et al., 2013). Our previous study demonstrated that the microfluidization process increased SC and WHC of corn bran because the process increased surface area of bran particles and loosened microstructure of bran matrix, making more polar groups and other water binding sites of the treated corn bran accessible to water molecules (Wang et al., 2013). As can be seen from Eqs. (2) and (3), increasing only pressure resulted in a linear increase in SC and WHC; however, SC and WHC had a negative quadratic relationship with number of passes, implying a hill-shaped response surface as shown in Figs. 1a and 1b. Eqs. (2) and (3) also showed that there was no interaction between processing pressure and number of passes. Within the experimental range, the maximum values of SC and WHC were respectively 10.62 ml/g d.w. and 5.49 g water/g d.w. which occurred at 158.6 MPa, the system's maximum operating pressure, and 5 passes, the largest number of passes examined. It was noted that higher values of SC and WHC could be obtained if the number of passes was further increased. However, increase in SC and WHC became significantly slow at a large number of passes as shown by the small slope of the response surfaces in the direction of number of passes (Figs. 1a and 1b). For this reason, it is advisable to choose a suitable number of passes for a microfluidization process by balancing benefits and operating costs.

OHC is another important property of dietary fiber. It depends on the chemical structure of plant polysaccharides as well as the surface properties, overall charge density, and hydrophobic nature of the fiber particles (Femenia et al., 1997; Fernández-López et al., 2009). Corn bran with higher OHC is favorable for flavor retention and product yield of high-fat foods such as cooked meat products which normally lose fat during cooking (Thebaudin et al., 1997). Based on our previous report (Wang et al., 2013), the microfluidization process improved OHC of corn bran because it increased porosity and surface area of bran particles and thus enhanced physical entrapment of oil by capillary attraction. The present study further revealed that both processing pressure and number of passes were critical factors affecting this change. As shown in Eq. (4), OHC had a positive linear relationship with pressure but a negative quadratic relationship with number of passes, and there was a negative interaction between the two processing parameters. The resulting response surface from these relationships was given in Fig. 1c. The figure showed that OHC increased with increasing pressure at a low number of passes, whereas decreased with increasing pressure at a high number of passes. This could be because extensive microfluidization treatment such as a high pressure combined with a large number of passes broke lipophilic groups or other oil binding sites of corn bran and thus led to a decrease in OHC. Therefore, a relatively low pressure and a large number of passes are favorable for increasing OHC. Within the experimental range, the maximum value of OHC was 4.61 g oil/g d.w. which occurred at 124.1 MPa and 5 passes.

3.4. Response surface analysis of antioxidant properties

The effect of microfluidization conditions on antioxidant properties of corn bran was depicted by the second-order polynomial Eqs. (5) - (7) as well as by response surface graphs (Figs. 1d – 1f).

Table 2 shows the results for sensitivity analysis of Eqs. (5) - (7) with respect to processing pressure (X_1) and number of passes (X_2), respectively, at five selected points. Again, number of passes showed a more profound impact on all antioxidant properties than processing pressure within the experimental range because the absolute value of the slope with respect to number of passes was generally greater than that with respect to pressure at each point examined.

Eqs. (5) - (6) showed that SRPC and DPPH scavenging activity had a positive linear relationship with pressure and a negative quadratic relationship with number of passes. These relationships indicated that values of SRPC and DPPH increased with increasing pressure but followed a hill-shaped curve in the direction of number of passes. On the other hand, Eq. (7) demonstrated that ABTS scavenging activity had a negative quadratic relationship with both processing parameters, implying the value of ABTS followed a hill-shaped curve in each direction of the two processing parameters. There was no interaction effect between the two processing parameters. Based on Eqs. (5) - (7), the maximum values of SRPC (148.80 mg/FAE g d.w.) and DPPH (50.02 μ mol TE/g d.w.) occurred at 158.7 MPa and 5 passes, while the maximum value of ABTS (47.90 μ mol TE/g d.w.) was reached at 145.9 MPa and 5 passes. It was noted that maximum values of antioxidant activities were obtained at the largest number of passes examined. Further increase in number of passes may result in higher values of antioxidant activities. However, Figs. 1d – 1f showed that increase in antioxidant activities became significantly slow at a large number of passes.

DPPH and ABTS assays are frequently used to measure antioxidant capacity in various food materials (Floegel et al., 2011). DPPH and ABTS radical scavenging activity of corn bran is primarily determined by the content of SRPC (Wang et al., 2014a) because about 69% of phenolics present in corn bran are insoluble and bound to the fiber matrix of the bran (Inglett and

Chen, 2011). The present study also confirmed the strong positive correlation between the SRPC value and values of DPPH ($R^2 = 0.846$) and ABTS ($R^2 = 0.817$) based on Pearson correlation analysis. The microfluidization treatment effectively increases antioxidant capacities of corn bran because it increases the accessibility of phenolic compounds either bound to or inside the bran matrix (Wang et al., 2014a).

4. Conclusion

This study clearly demonstrated that the microfluidization process improved physicochemical and antioxidant properties of corn bran and the extent of such improvements depended on processing pressure and number of passes. Response surface analysis showed that swelling capacity, water-holding capacity, and oil-holding capacity, surface reactive phenolic content, and DPPH radical scavenging activity of microfluidized corn bran had a positive linear relationship with pressure and a negative quadratic relationship with number of passes, whereas ABTS radical scavenging activity had a negative quadratic relationship with both processing parameters. Moreover, the analysis showed that the number of passes generally had a more pronounced effect on the examined properties compared with pressure. Within the experimental range, firstly, it seemed that a higher pressure was favorable for swelling capacity, water-holding capacity, surface reactive phenolic content, and DPPH radical scavenging activity, whereas a relatively low pressure is favorable for oil-holding capacity. Secondly, there existed a maximum value for each property indicated by its negative quadratic relationship with number of passes. However, the increase in properties became significantly slow at a large number of passes. Therefore, it is advisable to choose a suitable number of passes for a microfluidization process by balancing benefits and operating costs. Lastly, there was an optimum combination of pressure

319 and number of passes for ABTS radical scavenging activity implied by its negative quadratic
320 relationship with both processing parameters.

321 **Acknowledgments**

322 Thanks to USDA-NIFA for providing the financial support under grant # 2012-38821-20066.

323

324

325

326

327

328

329

330

331

332

333

334

References

- Abdul Aziz, N.A., Wong, L.M., Bhat, R., Cheng, L.H., 2012. Evaluation of processed green and ripe mango peel and pulp flours (*Mangifera indica* var. Chokanan) in terms of chemical composition, antioxidant compounds and functional properties. *Journal of the Science of Food and Agriculture* 92, 557-563.
- Alizadeh Bahaabad, G., Gharibzahedi Seyed Mohammad, T., Esmaili, M., Alizadeh, M., 2014. Response Surface Modeling for Optimization of Textural and Color Characteristics of Dried Grapes. *International Journal of Food Engineering* 10, 493-502.
- An, H.Z., Helgeson, M.E., Doyle, P.S., 2012. Nanoemulsion Composite Microgels for Orthogonal Encapsulation and Release. *Advanced Materials* 24, 3838-3844.
- Anderson, J.W., Baird, P., Davis, R.H., Jr., Ferreri, S., Knudtson, M., Koraym, A., Waters, V., Williams, C.L., 2009. Health Benefits of Dietary Fiber. *Nutr Rev* 67, 188-205.
- Chau, C.-F., Wen, Y.-L., Wang, Y.-T., 2006. Effects of micronisation on the characteristics and physicochemical properties of insoluble fibres. *Journal of the Science of Food and Agriculture* 86, 2380-2386.
- Chou, S.Y., Chien, P.J., Chau, C.F., 2008. Particle size reduction effectively enhances the cholesterol-lowering activities of carrot insoluble fiber and cellulose. *Journal of Agricultural and Food Chemistry* 56, 10994-10998.
- de Escalada Pla, M.F., Ponce, N.M., Stortz, C.A., Gerschenson, L.N., Rojas, A.M., 2007. Composition and functional properties of enriched fiber products obtained from pumpkin (*Cucurbita moschata* Duchesne ex Poiret). *LWT - Food Science and Technology* 40, 1176-1185.

356 Femenia, A., Lefebvre, A.C., Thebaudin, J.Y., Robertson, J.A., Bourgeois, C.M., 1997. Physical
 357 and Sensory Properties of Model Foods Supplemented with Cauliflower Fiber. *Journal of Food*
 358 *Science* 62, 635-639.

359 Fernández-López, J., Sendra-Nadal, E., Navarro, C., Sayas, E., Viuda-Martos, M., Alvarez,
 360 J.A.P., 2009. Storage stability of a high dietary fibre powder from orange by-products.
 361 *International Journal of Food Science and Technology* 44, 748-756.

362 Floegel, A., Kim, D.-O., Chung, S.-J., Koo, S.I., Chun, O.K., 2011. Comparison of ABTS/DPPH
 363 assays to measure antioxidant capacity in popular antioxidant-rich US foods. *Journal of Food*
 364 *Composition and Analysis* 24, 1043-1048.

365 Gonlachanvit, S., Coleski, R., Owyang, C., Hasler, W., 2004. Inhibitory actions of a high fibre
 366 diet on intestinal gas transit in healthy volunteers. *Gut* 53, 1577-1582.

367 Guraya, H.S., James, C., 2002. Deagglomeration of rice starch-protein aggregates by high-
 368 pressure homogenization. *Starch - Stärke* 54, 108-116.

369 Han, L., Li, L., Li, B., Zhao, L., Liu, G., Liu, X., Wang, X., 2014. Effect of high pressure
 370 microfluidization on the crystallization behavior of palm stearin - palm olein blends. *Molecules*
 371 19, 5348-5359.

372 Inglett, G.E., Chen, D., 2011. Antioxidant activity and phenolic content of air-classified corn
 373 bran. *Cereal Chemistry Journal* 88, 36-40.

374 Kuan, Y.-H., Liong, M.-T., 2008. Chemical and physicochemical characterization of agrowaste
 375 fibrous materials and residues. *Journal of Agricultural and Food Chemistry* 56, 9252-9257.

376 Marthinsen, D., Fleming, S.E., 1982. Excretion of breath and flatus gases by humans consuming
 377 high-fiber diets. *The Journal of Nutrition* 112, 1133-1143.

378 Mora, Y.N., Contreras, J.C., Aguilar, C.N., Meléndez, P., Garza, I.D.I., Rodríguez, R., 2013.
 379 Chemical composition and functional properties from different sources of dietary fiber.
 380 American Journal of Food and Nutrition 1, 27-33.

381 Onwulata, C.I., 2008. Baking properties of milk protein-coated wheat bran. Journal of Food
 382 Processing and Preservation 32, 24-38.

383 Robertson, J.A., de Monredon, F.D., Dysseler, P., Guillon, F., Amado, R., Thibault, J.-F., 2000.
 384 Hydration properties of dietary fibre and resistant starch: a European collaborative study. LWT -
 385 Food Science and Technology 33, 72-79.

386 Robin, F., Schuchmann, H.P., Palzer, S., 2012. Dietary fiber in extruded cereals: Limitations and
 387 opportunities. Trends in Food Science & Technology 28, 23-32.

388 Saulnier, L., Vigouroux, J., Thibault, J.F., 1995. Isolation and partial characterization of
 389 feruloylated oligosaccharides from maize bran. Carbohydrate Research 272, 241-253.

390 Slavin, J.L., 2005. Dietary fiber and body weight. Nutrition 21, 411-418.

391 Thebaudin, J.Y., Lefebvre, A.C., Harrington, M., Bourgeois, C.M., 1997. Dietary fibres:
 392 Nutritional and technological interest. Trends in Food Science and Technology 8, 41-48.

393 Vitaglione, P., Napolitano, A., Fogliano, V., 2008. Cereal dietary fibre: a natural functional
 394 ingredient to deliver phenolic compounds into the gut. Trends in Food Science and Technology
 395 19, 451-463.

396 Wang, T., Sun, X., Raddatz, J., Chen, G., 2013. Effects of microfluidization on microstructure
 397 and physicochemical properties of corn bran. Journal of Cereal Science 58, 355-361.

398 Wang, T., Zhu, Y., Sun, X., Raddatz, J., Zhou, Z., Chen, G., 2014a. Effect of microfluidisation
 399 on antioxidant properties of corn bran. Food Chemistry 152, 37-45.

Wang, T., He, F., Chen, G., 2014b. Improving bioaccessibility and bioavailability of phenolic compounds in cereal grains through processing technologies: A concise review. *Journal of Functional Foods* 7, 101-111.

Wani, I.A., Sogi, D.S., Gill, B.S., 2013. Physicochemical and functional properties of flours from three Black gram (*Phaseolus mungo* L.) cultivars. *International Journal of Food Science and Technology* 48, 771-777.

Yu, L.L., Haley, S., Perret, J., Harris, M., 2002. Antioxidant properties of hard winter wheat extracts. *Food Chemistry* 78, 457-461.

Zhao, Z., Moghadasian, M.H., 2008. Chemistry, natural sources, dietary intake and pharmacokinetic properties of ferulic acid: A review. *Food Chemistry* 109, 691-702.

List of Figure Captions

Fig. 1 Response surface plots of SC (a), WHC (b), OHC (c) SRPC (d), DPPH (e), and ABTS (f) values of microfluidized corn bran as a function of pressure and number of passes.

433

434

435

Figure 1

List of Tables

Table 1 Particle size distribution and bulk density of corn bran with difference microfluidization conditions based on central composite design

Run	Coded levels		Actual levels		MV ^a (μm)	CS ^b (m^2/cm^3)	Bulk Density (g/cm^3)
	X ₁	X ₂	Pressure (MPa)	Number of passes			
1	1	-1	151.7	2	65.84 $\pm$ 1.42	0.18 $\pm$ 0.00	0.29 $\pm$ 0.01
2	1	1	151.7	4	51.55 $\pm$ 0.73	0.22 $\pm$ 0.00	0.28 $\pm$ 0.01
3	0	0	137.9	3	55.48 $\pm$ 0.48	0.20 $\pm$ 0.00	0.28 $\pm$ 0.01
4	-1	-1	124.1	2	77.20 $\pm$ 0.39	0.16 $\pm$ 0.00	0.33 $\pm$ 0.02
5	-1	1	124.1	4	54.63 $\pm$ 0.46	0.20 $\pm$ 0.00	0.28 $\pm$ 0.01
6	-1.414	0	117.2	3	56.34 $\pm$ 0.43	0.19 $\pm$ 0.00	0.28 $\pm$ 0.01
7	1.414	0	158.6	3	56.66 $\pm$ 0.84	0.20 $\pm$ 0.01	0.29 $\pm$ 0.01
8	0	-1.414	137.9	1	100.90 $\pm$ 0.46	0.13 $\pm$ 0.00	0.38 $\pm$ 0.03
9	0	1.414	137.9	5	50.71 $\pm$ 0.70	0.21 $\pm$ 0.00	0.27 $\pm$ 0.02
10	0	0	137.9	3	56.14 $\pm$ 1.07	0.20 $\pm$ 0.00	0.30 $\pm$ 0.02
11	0	0	137.9	3	59.35 $\pm$ 0.83	0.19 $\pm$ 0.01	0.30 $\pm$ 0.03
12	0	0	137.9	3	58.02 $\pm$ 0.80	0.20 $\pm$ 0.00	0.28 $\pm$ 0.01
13	0	0	137.9	3	60.20 $\pm$ 0.25	0.19 $\pm$ 0.00	0.30 $\pm$ 0.01
	Control				230.07 $\pm$ 3.63	0.09 $\pm$ 0.00	0.75 $\pm$ 0.03

Each Value is expressed as means $\pm$ S.D. (n=3)

^a MV: Mean volume diameter

^b CS: Calculated specific surface area

448 **Table 2** Sensitivity analysis of response surface Eqs. (2) - (7) with respect to processing pressure (X_1) and number of passes (X_2)

Property	Variable	Point (-1.414,-1.414)	Point (1.141,-1.414)	Point (0,0)	Point (-1.414,1.414)	Point (1.414,1.414)
		Partial derivatives of response surface equations				
SC ^a	X_1	0.130	0.130	0.130	0.130	0.130
	X_2	1.106	1.106	0.710	0.314	0.314
WHC ^b	X_1	0.066	0.066	0.066	0.066	0.066
	X_2	0.372	0.372	0.250	0.128	0.128
OHC ^c	X_1	0.190	0.190	0.058	-0.074	-0.074
	X_2	0.615	0.352	0.260	0.168	-0.095
SRPC ^d	X_1	4.080	4.080	4.080	4.080	4.080
	X_2	16.960	16.960	11.530	6.100	6.100
DPPH	X_1	1.090	1.090	1.090	1.090	1.090
	X_2	4.065	4.065	2.510	0.955	0.955
ABTS	X_1	3.825	-1.605	1.110	3.825	-1.605
	X_2	5.230	5.230	2.770	0.310	0.310

449

450 ^a SC: Swelling capacity

451 ^b WHC: Water-holding capacity

452 ^c OHC: Oil-holding capacity

453 ^d SRPC: Surface reactive phenolic contents

454 **Table 3** Experimental data for 5-level-2-factor response surface analysis

Independent Variables		Response variables					
Pressure (MPa)	Number of passes	SC ^a (ml/g d.w.)	WHC ^b (g water/g d.w.)	OHC ^c (g oil/g d.w.)	SRPC ^d (mg/FAE ^e g d.w.)	DPPH (μ mol TE ^f /g d.w.)	ABTS (μ mol TE/g d.w.)
151.7	2	9.20 $\pm$ 0.05	4.94 $\pm$ 0.05	4.10 $\pm$ 0.04	120.30 $\pm$ 5.08	45.03 $\pm$ 3.66	42.37 $\pm$ 1.93
151.7	4	10.49 $\pm$ 0.15	5.39 $\pm$ 0.08	4.36 $\pm$ 0.04	144.81 $\pm$ 2.50	49.22 $\pm$ 2.57	47.85 $\pm$ 2.18
137.9	3	9.73 $\pm$ 0.16	5.08 $\pm$ 0.05	4.28 $\pm$ 0.06	132.31 $\pm$ 1.12	45.53 $\pm$ 1.46	44.89 $\pm$ 1.94
124.1	2	8.85 $\pm$ 0.28	4.83 $\pm$ 0.04	3.75 $\pm$ 0.08	114.50 $\pm$ 0.44	41.86 $\pm$ 2.67	41.20 $\pm$ 1.18
124.1	4	10.29 $\pm$ 0.19	5.25 $\pm$ 0.01	4.37 $\pm$ 0.04	134.41 $\pm$ 1.76	45.51 $\pm$ 1.00	45.29 $\pm$ 2.83
117.2	3	9.58 $\pm$ 0.17	5.04 $\pm$ 0.08	4.22 $\pm$ 0.05	127.76 $\pm$ 3.09	45.26 $\pm$ 3.52	40.80 $\pm$ 3.56
158.6	3	9.96 $\pm$ 0.06	5.25 $\pm$ 0.03	4.32 $\pm$ 0.03	140.06 $\pm$ 0.60	46.83 $\pm$ 2.33	44.61 $\pm$ 1.08
137.9	1	7.66 $\pm$ 0.19	4.41 $\pm$ 0.03	3.43 $\pm$ 0.02	100.00 $\pm$ 3.99	38.33 $\pm$ 2.94	35.69 $\pm$ 2.23
137.9	5	10.54 $\pm$ 0.16	5.49 $\pm$ 0.01	4.57 $\pm$ 0.10	146.97 $\pm$ 3.09	49.43 $\pm$ 3.85	47.53 $\pm$ 2.05
137.9	3	9.68 $\pm$ 0.12	5.08 $\pm$ 0.01	4.34 $\pm$ 0.01	136.78 $\pm$ 1.47	45.63 $\pm$ 3.23	45.63 $\pm$ 0.69
137.9	3	9.60 $\pm$ 0.28	5.17 $\pm$ 0.09	4.31 $\pm$ 0.08	126.10 $\pm$ 1.25	47.50 $\pm$ 3.42	44.32 $\pm$ 2.28
137.9	3	9.72 $\pm$ 0.15	5.09 $\pm$ 0.08	4.27 $\pm$ 0.04	130.39 $\pm$ 1.89	46.32 $\pm$ 0.97	45.94 $\pm$ 0.54
137.9	3	9.72 $\pm$ 0.07	5.21 $\pm$ 0.10	4.33 $\pm$ 0.06	128.69 $\pm$ 2.26	45.26 $\pm$ 1.87	45.47 $\pm$ 3.03

455

456 Each Value is expressed as means $\pm$ S.D. (n=3)

457 ^a SC: Swelling capacity

458 ^b WHC: Water-holding capacity

459 ^c OHC: Oil-holding capacity

460 ^d SRPC: Surface reactive phenolic contents

461 ^e FAE: Ferulic acid equivalents

462 ^f TE: Trolox equivalent

463

Table 4 ANOVA and regression coefficients of the second-order polynomial model for the effect of processing pressure and passes on SC, WHC, and OHC values

Source	SC ^a			WHC ^b			OHC ^c		
	Coefficient	SS ^d	p Value ^e	Coefficient	SS	p Value	Coefficient	SS	p Value
Block		0.027			1.05E-003			7.79E-003	
Model/Intercept	9.71	20.24	< 0.0001	5.13	2.56	< 0.0001	4.30	3.15	< 0.0001
Linear									
B ₁ -pressure	0.13	0.45	0.0004	0.066	0.11	< 0.0001	0.058	0.086	0.0004
B ₂ -pass	0.71	17.98	< 0.0001	0.25	2.29	< 0.0001	0.26	2.52	< 0.0001
Quadratic									
B ₁ ²	-	0.081	ns	-	3.022E-003	ns	-	0.018	ns
B ₂ ²	-0.14	1.41	<0.0001	-0.043	0.13	< 0.0001	-0.079	0.44	<0.0001
Interaction									
B ₁₂	-	0.016	ns	-	6.668E-004	ns	-0.093	0.10	0.0001
Residual		0.91			0.16			0.17	
Lack-of-Fit		0.62	ns		0.063	ns		0.13	ns
Pure error		0.30			0.095			0.039	
Total		21.18			2.71			3.33	
R ²	0.9570			0.9417			0.9487		
Adj-R ²	0.9500			0.9324			0.9404		
Pred-R ²	0.9303			0.9139			0.9134		
C.V. (%)	1.78			1.40			1.76		
PRESS	1.47			0.23			0.29		
Adequate precision	37.189			31.596			32.552		

^a SC: Swelling capacity

^b WHC: Water-holding capacity

^c OHC: Oil-holding capacity

^d SS: Sum of squares

^e (p<0.05): Significant; (p<0.0001): Highly significant; ns: Not significant

Table 5 ANOVA and regression coefficients of the second-order polynomial model for the effect of processing pressure and passes on SRPC, DPPH, and ABTS values

Source	SRPC ^a			DPPH			ABTS		
	Coefficient	SS ^b	p Value ^c	Coefficient	SS	p Value	Coefficient	SS	p Value
Block		2.65			144.69			81.59	
Model/Intercept	130.57	5572.25	< 0.0001	46.03	278.21	< 0.0001	45.40	370.00	< 0.0001
Linear									
B ₁ -pressure	4.08	423.92	< 0.0001	1.09	30.03	0.0014	1.11	31.50	0.0002
B ₂ -pass	11.53	4785.35	< 0.0001	2.51	225.92	< 0.0001	2.77	276.05	< 0.0001
Quadratic									
B ₁ ²	-	25.04	ns	-	9.161E-003	ns	-0.96	21.93	0.0012
B ₂ ²	-1.92	255.85	< 0.0001	-0.55	20.73	0.0066	-0.87	52.75	< 0.0001
Interaction									
B ₁₂	-	15.91	ns	-	0.22	ns	-	1.45	ns
Residual		389.80			75.71			53.44	
Lack-of-Fit		176.29	ns		47.67	ns		31.86	ns
Pure error		213.51			28.03			21.58	
Total		5964.71			498.61			505.04	
R ²	0.9346			0.7861			0.8738		
Adj-R ²	0.9241			0.7516			0.8534		
Pred-R ²	0.9010			0.6456			0.8050		
C.V. (%)	2.74			3.43			2.99		
PRESS	590.05			125.41			82.58		
Adequate precision	29.404			21.296			25.137		

^a SRPC: Surface reactive phenolic contents

^b SS: Sum of squares

^c (p<0.05): Significant; (p<0.0001): Highly significant; ns: Not significant