

EFFECT OF EXTRUSION ON THE TOTAL ANTIOXIDANT CAPACITY AND FREE PHENOLIC COMPOUNDS OF WHEAT BRAN BY RESPONSE SURFACE METHODOLOGY

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ABSTRACT

There are some antioxidants in wheat bran provides health benefits. But the influence of extrusion pre-treatment on the antioxidant capacity and free phenolic compounds of wheat bran is not clear. Herein, it was investigated by response surface methodology (RSM). Within the experimental range, free phenolic compounds (FPC) increased gradually with feed moisture and extrusion temperature. And the total antioxidant capacity of extruded wheat bran increased gradually with rising feed moisture and screw speed. The optimized extrusion parameters were extrusion temperature at 86°C, feed moisture at 22% and screw speed at 160 rpm. The total FPA reached 3136.9 µg GAE g⁻¹ and the ferulic acid content was 93.4 µg.g⁻¹. Extrusion treatment for wheat bran significantly improved the antioxidant properties and increased the concentration of gallic acid and ferulic acid. The effect of extrusion temperature on total free phenol content is extremely significant.

Keywords: wheat bran, extrusion, response surface methodology, Trolox equivalent antioxidant capacity, free phenolic compounds

1. INTRODUCTION

Wheat bran accounts for approximately 14% of the whole wheat grain. It is consisted of multiple layers, including aleurone layer, the nucellar epidermis, the inner pericarp, and the outer pericarp (from inside to outside) (MATEO *et al.*, 2012), (PANDEY and RIZVI, 2009; PERALES-SÁNCHEZ *et al.*, 2014). Numerous literatures have been found to make a thorough inquiry species of the phenolic compounds exist in wheat, especially in wheat bran fraction (NEACSU *et al.*, 2017; ROSICKA-KACZMAREK *et al.*, 2018). In plants, phenolics compounds incorporate a wide variety of compounds including flavonoids, tannins, coumarins, and phenolic acids (HOSENEY, 2010). What's more, phenolics compounds including a benzene ring bearing one or lots of hydroxyl groups and phenolic acids that derivatives of either hydroxybenzoic or hydroxycinnamic acid are usually being all cereals (KIM *et al.*, 2006; ZHENG *et al.*, 2015). Generally, many beneficial compounds in cereals dedicate to their antioxidant characteristics, such as tocopherols, carotenoids, polyphenols, flavonoids, anthocyanins, lignans. The comprehensive antioxidant ability of all antioxidants in the cereals is generally represents as total antioxidant capacity (TAC) (RE *et al.*, 1999; RICE-EVANS *et al.*, 1999).

Phenolic acids in wheat bran usually can be divided into three type of existing forms: soluble free phenolic acids, conjugated phenolic acids, and insoluble-bound phenolic acids (ROSICKA-KACZMAREK *et al.*, 2018). The free and conjugated phenolic acids make up only a small section, while most of the phenolic acids are bound phenolic compounds by ester and ether linkages with cell wall components such as arabinoxylans and lignin (LIU *et al.*, 2016). The aleurone layer is mostly composed of arabinoxylan with a high content of ferulic acid (FA) monomers and low levels of FA dimers (RAMOS-ENRÍQUEZ *et al.*, 2018a). In fact, different form of phenolic acid worked on various impacts on human health. When the dietary contain bound forms were intake by human, it would be useful in the precaution of colon cancer and other cancers (LEI *et al.*, 2012; RAMOS-ENRÍQUEZ *et al.*, 2018b). However, the intake of soluble free and conjugated forms is attributed to quickly absorption in the stomach and small intestine as well as distribution throughout the body.

Extrusion technology is a combination of mechanical shearing action, pressure action and thermal energy, which causes the material to be suddenly released from the high temperature and huge pressure state to the normal temperature and pressure and the internal structure and physical and chemical properties of the extruded material would be changed and extrusion technology also is a kind of processing methods to force materials at a predetermined feed rate, to flow through materials and through certain die holes to obtain products of different shapes and properties, and the food is called extruded food (AAM *et al.*, 2017). As a high-tech in the field of food processing, extrusion technology opens -up to a new way of production that is simple, mechanized and highly automated for the development of convenience food. Besides, it has a high efficiency, and low cost in processing, as well as the products are easy to digest, keep the nutrient at a maximum degree, and is conducive to long-term storage.

Some evidence has been reported the activities of lipoxygenase and polyphenol oxidase were greatly reduced, and the shelf life was prolonged compared to the wheat bran, which has not been extruded (PILLI *et al.*, 2010). What's more, the content of free phenolic compound in the product was obviously improved, and the antioxidant capacity was also enhanced. Therefore, it can be used as a excellent material for making whole wheat food or health-benefit products for improving the nutritional value (BRENNAN *et al.*, 2011).

In our previous single factor study, it was found that the antioxidant ability and to phenolic content of extruded wheat bran were higher when the feed moisture was 22-24% and the screw speed was 130-190 rpm at the extrusion temperature of 80-100°C. To improve the antioxidant ability and shelf life of wheat bran, the optimum extrusion condition on the antioxidant capacity and free phenolic compounds of wheat bran was investigated by RSM. And the phenolic compounds extracted from wheat bran before and after extrusion were also identified by ultra-high performance liquid chromatography (UPLC).

2. MATERIALS AND METHODS

2.1. Materials and reagents

Wheat brans were obtained from Hubei Sanjie Agricultural Industrialization Co. Ltd (Hubei province, China); 2,2'-Azino-bis (3-ethylbenzothiazoline-6-sulfonic acid) (ABTS) and 6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid (Trolox) were purchased from Sigma–Aldrich. Other chemicals and solvents for chromatographic grade analysis were obtained from Merck (Darmstadt, Germany). All the other chemicals and solvents were of analytical grade.

2.2. Sample preparation

The wheat brans were milled and sieved to a 60 mesh size. The powder was put in to two-screw extruder (FMHE36-24, Hunan Fuma Branch Food Engineering Technology Co, Ltd) and then the extruded wheat brans were dried at 50°C for 18 hours to cut down the moisture content, and then extruded wheat brans were milled and sieved to a 60 mesh size. The powder of extruded wheat bran was kept in a black laboratory bottle, and the bottles were placed at -20°C in refrigerator.

2.3. Ultrasound extraction

0.5 g of the dried powder of extruded wheat brans were thoroughly mixed with 10 mL of 60% ethanol and placed in a 50 mL amber laboratory bottle (DHANANI *et al.*, 2017). The operating extraction was last for 1.5 hours at 60°C. The supernatants were combined after centrifugation (3622×g, 20 min). After centrifugation the supernatant were evaporated to 2 mL at 45°C and placed in amber laboratory bottle at -20°C until used.

2.4. Determination of Trolox equivalent Antioxidant activity

ABTS assay was implemented the concordat of Pellegrini N (RE *et al.*, 1999) with a little modifications by condition. ABTS^{•+} radical solution was prepared to mix 10 mL of ABTS stock solution (7mM ABTS in water) with 176 µL of potassium persulfate (140 mmol/L), which was kept in darkness 12-16 h at 4°C. The ABTS^{•+} reagent was diluted with anhydrous ethanol to detect the absorbance of 0.700 ±0.02 at 734 nm (Berg *et al.*, 1999).

The 0.1 mL of the sample solution and 3.9 mL of diluted ABTS^{•+} were thoroughly mixed and placed in a 10 mL amber glass tube and shook at room temperature for 6 minutes. The absorbance of the reaction was measured at 734 nm ($A_{734} = A_s$) using glass cuvettes. Then 0.1 mL of sample and 3.9 mL of anhydrous ethanol or 3.9 mL of diluted ABTS^{•+} were operated as the above mention and the absorbance A_{734} remarked as A_s and A_0 respectively.

The calibration curve was set using Trolox at the consistence range of 50–1000 μ mol/L in ultrapure water.

The total antioxidant capacity of wheat bran extruded material was expressed as a TEAC per 1 g of dry matter of a sample (μ mol/g).

$$\% \text{ inhibition rate of ABTS} = \left(1 - \frac{(A_s - A_r)}{A_0}\right) \times 100\% \quad (1)$$

2.5. Determination of extruded wheat bran and raw wheat bran total free phenolic content

The total free phenolic content was analyzed as determined following described previously (LI *et al.*, 2008; VÁZQUEZ *et al.*, 2015a) with some modification. Briefly, a stock solution of gallic acid with pipette gallic acid control solutions was prepared at a concentration of 0.5 mg/ml. With this solution, calibration curve was prepared for the different dilutions. In the dim light, 1 mL of the extract obtained from raw wheat bran and extruded wheat bran were placed in each well of a burette and 9 mL of deionized water, 1 mL of Folin-Ciocalteu reagent, and 2 mL of sodium carbonate solution (w/w=1/4) were added in orderly. All blanks except the extract was also prepared. Then these burettes were put in water bath at 50°C for 0.5 h. Secondly, 12 mL of deionized water was added in the burette and put at room temperature for 0.5 h. Subsequently, the burettes were read on a spectrophotometer at an absorbance of 745 nm (VÁZQUEZ *et al.*, 2015a).

Above all results were represented as gallic acid equivalent (mg gallic acid/g of extruded wheat bran material GAE/g of dried sample).

2.6. UPLC analysis

UPLC-PDA was used to determine free phenolic compounds extracted from raw and extruded wheat bran. The chromatographic system was made up an Acquity UPLC (Waters, US) equipped with PDA. Samples were separated using a Waters Column (ACQUITY UPLC@HSS T3 1.8 μ m, 2.1 \times 150 mm Column). The column temperature was maintained at 40°C. Methanol-acetonitrile solution (1:1, v/v) and acetic acid (2.50%, v/v) were used as mobile phase A and B, respectively. The gradient program was as follows: 5-20% A (0-6 min), 20-40% A (6-15 min), 40-70% A (15-18 min), and 70-5% A (18-24 min). Phenolic acids were detected at 280 nm. The phenolic acid content was calculated from the peak area according to the calibration curve by using the external standard method and expressed as μ g/g DW.

2.7. Experimental design

The effect of factors such as extrusion temperature (80°C, 90°C, 100°C), screw speed (130 rpm, 160 rpm, 190 rpm), and feed moisture (20%, 22%, 24%) on free phenolic compounds and Trolox equivalent antioxidant capacity (TEAC) were tested. A three-level-three-factor and seven central point factorial design were employed requiring a total of 19 experiments. The BBD was used to determine the optimal extrusion conditions that maximum of TEAC and FPC of extruded wheat bran.

The three independent variables of extrusion temperature ($^{\circ}$ C, X_1), feed moisture (%), X_2 and screw speed (rpm, X_3) at three levels (-1, 0, +1) were set. The coded and actual values of variables were shown in Table 1.

Table 1. Level of coded and real values for factorial design.

Factors	Level		
	-1	0	1
Extrusion temperature (°C) (X_1)	80	90	100
Feed moisture (%) (X_2)	20	22	24
Screw speed (rpm) (X_3)	130	160	190

2.8. Statistical analysis

All experiments were carried out in triplicates and results were expressed as means \pm standard deviation ($n=3$). ANOVA was carried out to determine any significant differences ($p < 0.05$). Response surface plots were generated using Design-Expert 6.0.

3. RESULTS

3.1. Effect of extrusion condition on TEAC content of extruded wheat bran

The mean values of TEAC content and the content of FPC each of the 19 treatments at the different extrusion conditions were shown in Table 2. The highest of TEAC content of 14.1143 $\mu\text{mol/g}$ was obtained in experimental run number 7 with an extrusion temperature of 90°C, feed moisture of 22% and a screw speed of 160 rpm. While the lowest TEAC content of 12.7929 $\mu\text{mol/g}$ was observed in experimental run number 16 with an extrusion temperature of 90°C, feed moisture of 22% and screw speed at 190 rpm.

In Tables 3 and 4, the estimated regression coefficients and ANOVA of TEAC of extruded wheat bran was observed. The quadratic regression model was extremely significant ($p=0.0004 < 0.001$) and the lack of fit was not significant ($p=0.1609 > 0.05$) at the same time, showing that the model was in good agreement with the experimental data of TEAC content (BANNOUR *et al.*, 2017; Berg *et al.*, 1999; Zhen *et al.*, 2016). The regression coefficient ($R^2 = 0.9264$) suggested the experimental and predicted content data had been a good fit in the experiment. The linear and quadratic of feed moisture showed a significant difference, indicating the effect on TEAC content. Moreover, the interactive variables between feed moisture and screw speed showed a significant difference ($p=0.0286 < 0.05$), suggesting the effect on TEAC content. And then also the interactive variables between extrusion temperature and screw speed showed a significant difference ($p=0.0210 < 0.05$), suggesting the effect on TEAC content.

In order to analyze the effect of interaction of the different variables, the response surface curves were plotted. Meanwhile, for the purpose of determining the optimal extrusion condition of the responses of the independent variables with maximized TEAC content of extruded wheat bran. The concentration of TEAC content of extruded wheat bran increased with increasing feed moisture and screw speed. However, when the feed moisture was added to nearly 22% and the screw speed added to 160 rpm, the TEAC content slowly dropped, as described in Fig. 1C. TEAC values also enhanced with the enhanced of extrusion temperature and feed moisture data as shown in Fig. 1A. Fig. 1C

revealed the increasing TEAC content with an increase of screw speed and extrusion temperature.

Table 2. Values for TEAC and FPC of extruded wheat bran extracts under different extrusion conditions.

Run number	Factorial design			Determination	
	X ₁	X ₂	X ₃	Total free phenolic content (mg GAE/g)	TEAC (μmol/g)
	Extrusion temperature (°C)	Feed moisture (%)	Screw speed (rpm)	Experimental	Experimental
1	90	24	130	3.04±0.06	13.03±0.01
2	100	22	130	2.83±0.05	13.23±0.01
3	90	22	160	3.14±0.04	13.80±0.01
4	90	20	130	2.94±0.01	13.03±0.05
5	90	24	190	2.78±0.06	13.55±0.02
6	80	24	160	3.04±0.02	13.56±0.02
7	90	22	160	3.18±0.02	14.11±0.01
8	80	22	130	3.14±0.03	13.49±0.01
9	90	22	160	3.17±0.03	13.80±0.01
10	100	22	190	2.99±0.01	13.64±0.03
11	90	22	160	3.15±0.03	13.88±0.04
12	90	22	160	3.21±0.04	13.84±0.01
13	100	20	160	2.88±0.04	13.08±0.01
14	90	22	160	3.21±0.01	13.91±0.07
15	80	20	160	3.07±0.01	13.23±0.01
16	90	20	190	2.92±0.01	12.79±0.01
17	100	24	160	2.90±0.01	13.21±0.03
18	80	22	190	2.92±0.02	13.08±0.05
19	90	22	160	3.13±0.05	13.76±0.02

Table 3. ANOVA.

Source	TEAC (R ² =0.9264)					Total FPC (R ² =0.9579)				
	SS	DF	MS	F-value	p-value	SS	DF	MS	F-value	p-value
Model	2.40	9	0.27	12.58	0.0004	0.00	9	0.00	22.74	< 0.0001
Lack of fit	0.11	3	0.04	2.45	0.1609	0	3	0.00	2.77	0.1329
Pure error	0.09	6	0.01			0	6	0.00		

Table 4. ANOVA for Quadratic model.
Response 1: TEAC of extruded wheat bran.

Source	SS	df	MS	F-value	p-value	
Model	2.40	9	0.2662	12.58	0.0004	significant
A-extrusion temperature	0.0051	1	0.0051	0.2407	0.6354	
B-feed moisture	0.1834	1	0.1834	8.67	0.0164	
C-screw speed	0.0108	1	0.0108	0.5093	0.4935	
AB	0.0107	1	0.0107	0.5035	0.4959	
AC	0.1649	1	0.1649	7.79	0.0210	
BC	0.1433	1	0.1433	6.77	0.0286	
A ²	0.1277	1	0.1277	6.04	0.0364	
B ²	0.8298	1	0.8298	39.21	0.0001	
C ²	0.5207	1	0.5207	24.60	0.0008	
Residual	0.1905	9	0.0212			
Lack of Fit	0.1050	3	0.0350	2.45	0.1609	not significant
Pure Error	0.0855	6	0.0143			
Cor Total	2.59	18				

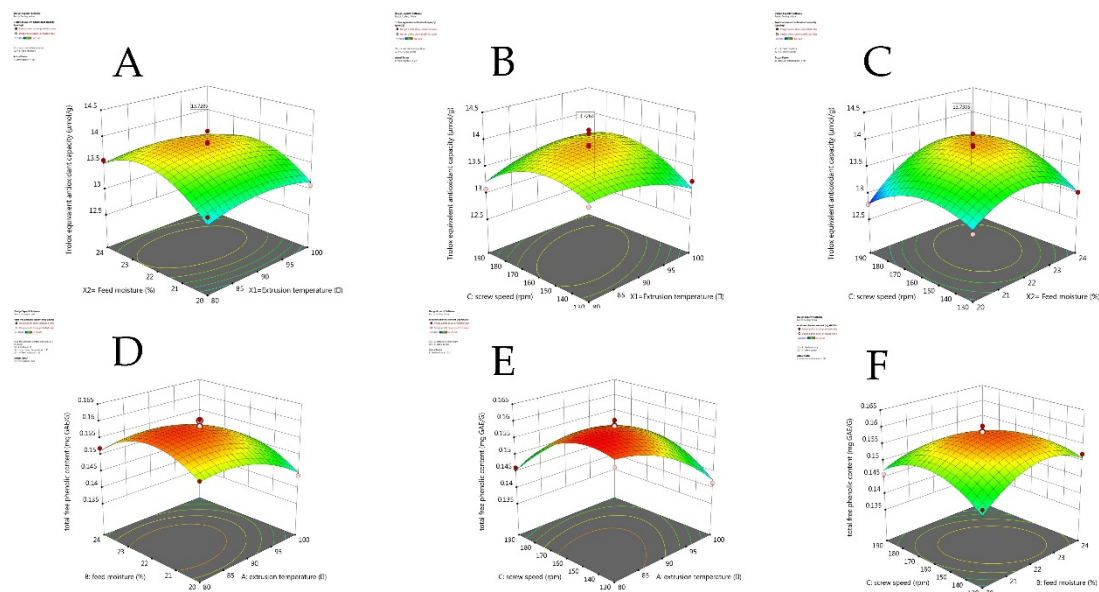


Figure 1. Effect of different extrusion conditions on TEAC and FPC : (A) TEAC effect of extrusion temperature and feed moisture; (B) TEAC effect of extrusion temperature and screw speed; (C) TEAC effect of feed moisture and screw speed; (D) FPC contents effect of extrusion temperature and screw speed for extrusion; (E) FPC contents effect of extrusion temperature and feed moisture; (F) FPC contents effect of screw speed and feed moisture for extrusion.

3.2. Effect of extrusion condition on FPC contents of extruded wheat bran

Table 5 shown that FPC contents of extruded wheat bran quadratic regression model was extremely significant ($p < 0.0001$). What's more, the lack of fit had a p-value higher ($p = 0.1392 > 0.05$). In Table 3, the total FPC value of $R^2 = 0.9579$ demonstrated the model to be a well fit for the experimental data of total FPC of extruded wheat bran.

Individual independent variables extrusion temperature had an extremely significant effect on the total FPC content which was indicated by the linear data model ($p = 0.0006 < 0.01$), and screw speed had major impact effect on the total phenolic content ($p = 0.0133 < 0.05$), but feed moisture didn't show significant effect on the total phenolic content. The interaction between extrusion temperature and feed moisture showed a not significant effect on the total FPC content ($p = 0.5254 > 0.05$), and interaction between feed moisture and screw speed also showed a significant effect on the total phenolic content ($p = 0.0136 < 0.05$). Meanwhile, the interplay between extrusion temperature and screw speed also expressed an extremely significant effect on the total FPC content ($p = 0.001 < 0.01$).

When the extruded temperature was over 100°C , the extruded wheat bran's antioxidant capacity and total free phenolic contents might decrease, which it may be that the phenolic compounds might be degraded. The results showed that the effect of extrusion temperature on total free phenol content is extremely significant ($p = 0.0006 < 0.01$). Meanwhile, screw speed also indicated an obviously effect on the FPC content ($p = 0.0133 < 0.05$). The interaction of the three independent variables of extrusion temperature, feed moisture, and screw speed was used to plot the response surface curves for the total phenolic content as shown in Fig. 1D, Fig. 1E, Fig. 1F. Increasing the extrusion

temperature from 80°C to 100°C at constant feed moisture (Fig. 1D) and screw speed (Fig. 1E) did change total phenolic content. Although, with the extrusion temperature, feed moisture and screw speed increasing, the total FPC contents of extruded wheat bran, as shown in Fig. 1D, Fig. 1E, Fig. 1F. But, when extrusion temperature and screw speed at nearly 100°C and 190 rpm, respectively, a small cut down in total phenolic content was observed (Fig. 1E). This expressed that a portion of phenolic compounds would be degraded by over high extrusion temperature and screw speed in extrusion process.

Table 5. ANOVA for Quadratic model.
Response 2: FPC of extruded wheat bran.

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	0.0008	9	0.0001	22.74	< 0.0001	significant
A-extrusion temperature	0.0001	1	0.0001	26.32	0.0006	
B-feed moisture	5.234E-07	1	5.234E-07	0.1360	0.7208	
C-screw speed	0.0000	1	0.0000	9.45	0.0133	
AB	1.680E-06	1	1.680E-06	0.4365	0.5254	
AC	0.0001	1	0.0001	23.14	0.0010	
BC	0.0000	1	0.0000	9.36	0.0136	
A ²	0.0001	1	0.0001	15.42	0.0035	
B ²	0.0002	1	0.0002	44.71	< 0.0001	
C ²	0.0002	1	0.0002	44.71	< 0.0001	
Residual	0.0000	9	3.848E-06			
Lack of Fit	0.0000	3	6.709E-06	2.77	0.1329	not significant
Pure Error	0.0000	6	2.418E-06			
Cor Total	0.0008	18				

3.3. Verification of predictive optimal extrusion conditions

The predicted extrusion conditions of wheat bran extruded material at 85.85°C of extrusion temperature, 22.19% of feed moisture and 154 rpm of screw speed provided the maximum TEAC content of extruded wheat bran, and the maximum total phenolic content was reached at the predicted conditions of 85.85°C of extrusion temperature, 22.19% of feed moisture and 154 rpm of screw speed. The predicted extruded conditions of wheat bran were the same for the TEAC content and total phenolic content for convenient operation, and considering the experiment in practice the optimal extraction parameters were adjusted to be 86°C of extrusion temperature, 22% of feed moisture and 160 rpm of screw speed at which that the predicted TEAC content was 13.8472 $\mu\text{mol g}^{-1}$

and total phenolic content was 13.19754 mg GAE g⁻¹ DW. This strongly suggests that the model is suitable to predict TEAC content, total phenolic content using extrusion at selected conditions.

3.4 Identification and quantification phenolic compounds of extruded wheat bran and raw wheat bran by UPLC

Table 6 showed results about the concentration of each phenolic compound obtained for the extruded wheat bran and raw wheat bran hydroalcoholic extracts. Two kinds of phenolic compounds (11.39 µg.g⁻¹ gallic acid and 78.40 µg.g⁻¹ ferulic acid) were identified in raw wheat bran and 4 kinds of phenolic compounds (12.58 µg.g⁻¹ gallic acid, 61.06µg.g⁻¹ caffeic acid, 93.40 µg.g⁻¹ ferulic acid, and 183.64µg.g⁻¹ rutin) in the extruded wheat bran. The compounds caffeic acid and rutin were not identified in the raw wheat bran. So those two phenolic compounds became the significant differences between the raw wheat bran and extruded wheat bran. Meanwhile, the raw wheat bran's TEAC was 12.38±0.22 µmol.g⁻¹ and extruded wheat bran's TEAC was 13.91±0.04 µmol.g⁻¹. And then the raw wheat bran's total free phenolic compounds was 2.88±0.09 mg GAE.g⁻¹, while extruded wheat bran's was 3.14±0.07 mg GAE.g⁻¹ the extruded wheat bran. Above all, the reason why that extruded wheat bran antioxidant capacity and free phenolic compounds significantly higher than the raw wheat bran could be explained by these dates.

Table 6. Characterization of extruded wheat bran extracts.

Determination	This study	
	The raw wheat bran	The extruded wheat bran
Total free phenolic compounds (mg GAE.g ⁻¹)	2.88±0.09 ^{Aa}	3.14±0.07 ^{Bb}
TEAC (µmol. g ⁻¹)	12.38±0.22 ^{Aa}	13.91±0.04 ^{Bb}
Ferulic acid (µg.g ⁻¹)	78.40±0.001 ^{Aa}	93.40±0.000 ^{Bb}
Gallic acid (µg.g ⁻¹)	11.39±0.002 ^{Aa}	12.58±0.001 ^{Bb}
Caffeic acid (µg.g ⁻¹)	0	61.06±0.001
Rutin (µg.g ⁻¹)	0	183.64±0.001

The content of gallic acid (12.58 $\mu\text{g g}^{-1}$ extruded wheat bran), ferulic acid (93.4 $\mu\text{g g}^{-1}$ extruded wheat bran) in extruded wheat bran were higher than the raw wheat bran. The extrusion technology helped the wheat bran to break the cell structure of wheat bran and then the phenolic compounds were released by high temperature, strong pressure, and great powerful shear force.

Phenolic compounds have one or more hydroxyl groups conjugated to an aromatic hydrocarbon group, which characterizes the phenolic structure (CHAIYASUT *et al.*, 2017; GUTIÉRREZ-GRIJALVA *et al.*, 2017; HILBIG *et al.*, 2018). The phenolic compounds specially structure bring about these compounds antioxidant activity to a certain degree, which may be higher or lower depending on the position and number of hydroxyls (APEABAH *et al.*, 2017; VÁZQUEZ *et al.*, 2015b). The presence of several phenolic compounds in extruded wheat bran extracts might explicate the antioxidant activity demonstrated for the extruded wheat bran.

4. CONCLUSION

In the present work, BBD was successfully carried out to set the extrusion conditions optimized parameters for the antioxidant capacity and free phenolic compounds of extruded wheat bran. In comparison to raw wheat bran, the extruded wheat bran resulted in the higher recoveries of both total free phenolic compounds and Trolox equivalent antioxidant capacity, which showed that extrusion could enhance the antioxidant capacity and free phenolic compounds of wheat bran.

Moreover, both the raw wheat bran and extruded wheat bran, yielded the same phenolic compounds, namely gallic acid, ferulic acid caffeic acid and rutin, were determined by UPLC. The study suggested that the use of extrusion pre-treatment in enhancing contents the of desired bioactive components from food industry by-product was a numerous potential extraction technology. From the data of the response surface, the extruded technology is efficient, economic and environmental process technology. Because the antioxidant capacity and free phenolic compounds were significantly improved by extrusion treatment. Thus, the extruded wheat brans would be a good source of natural antioxidants.

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ABBREVIATIONS

RSM	response surface methodology
FPC	free phenolic compounds
GAE	gallic acid equivalent
FA	ferulic acid
LDL	low-density lipoprotein
PDA	photo-diode array
UPLC	ultra performance liquid chromatography
TEAC	Trolox equivalent antioxidant capacity
BBD	Box-Behnken design

DW	dry weight
ANOVA	analysis of variance
ABTS	2,2'-Azino-bis (3-ethylbenzothiazoline-6-sulfonic acid)
SS	sum of squares
DF	degree of freedom
MS	mean square

REFERENCES

- Aam A., Andersson R., Jonsäll A., Andersson J. *et al.* 2017. Effect of Different Extrusion Parameters on Dietary Fiber in Wheat Bran and Rye Bran. *Journal of Food Science*, 82(6):1344.
- Apeabah F.B., Serem J.C., Bester M.J. and Duodu K.G. 2017. Phenolic composition and antioxidant properties of koose, a deep-fat fried cowpea cake. *Food Chemistry*, 237, S0308814617309111.
- Bannour M., Fellah B., Rocchetti G., Ashi-Smiti S. *et al.* 2017. Phenolic profiling and antioxidant capacity of Calligonum azel Maire, a Tunisian desert plant. *Food Research International*, 101:148-154.
- Berg R.V.D., Haenen G.R.M.M., Berg H.V.D. and Bast A. 1999. Applicability of an improved Trolox equivalent antioxidant capacity (TEAC) assay for evaluation of antioxidant capacity measurements of mixtures. *Food Chemistry*, 66(4):511-517.
- Brennan C., Brennan M., Derbyshire E. and Tiwari B.K. 2011. Effects of extrusion on the polyphenols, vitamins and antioxidant activity of foods. *Trends in Food Science & Technology*, 22(10):570-575.
- Chaiyasut C., Pengkumsri N., Sirilun S., Peerajan S. *et al.* 2017. Assessment of changes in the content of anthocyanins, phenolic acids, and antioxidant property of *Saccharomyces cerevisiae* mediated fermented black rice bran. *AMB Express*, 7(1):114.
- Dhanani T., Shah S., Gajbhiye N. and Kumar S. 2017. Effect of extraction methods on yield, phytochemical constituents and antioxidant activity of *Withania somnifera*. *Arabian Journal of Chemistry*, 10, S1193-S1199.
- Gutiérrez-Grijalva E.P., Angulo-Escalante, M.A., León-Félix J. and Heredia J.B. 2017. Effect of In Vitro Digestion on the Total Antioxidant Capacity and Phenolic Content of 3 Species of Oregano (*Hedeoma patens*, *Lippia graveolens*, *Lippia palmeri*). *Journal of Food Science*, 82(12).
- Hilbig J., Alves V.R., Müller C.M.O., Micke G.A. *et al.* 2018. Ultrasonic-assisted extraction combined with sample preparation and analysis using LC-ESI-MS/MS allowed the identification of 24 new phenolic compounds in pecan nut shell [*Carya illinoensis* (Wangenh) C. Koch] extracts. *Food Research International*, 106:549-557.
- Hoseney R.C. 2010. Analysis of Bioactive Components in Small Grain Cereals. In: Kim K.-H., Tsao, R., Yang, R., & Cui, S. W. 2006. Phenolic acid profiles and antioxidant activities of wheat bran extracts and the effect of hydrolysis conditions. *Food Chemistry*, 95(3):466-473.
- Lei L., Winter K.M., Stevenson L., Morris C. *et al.* 2012. Wheat bran lipophilic compounds with in vitro anticancer effects. *Food Chemistry*, 130(1):156-164.
- Li J., Nie J., Li H. and Xu G. *et al.* 2008. On determination conditions for total polyphenols in fruits and its derived products by Folin-phenol methods. *Journal of Fruit Science*, 25(1):126-131.
- Liu L., Zhao M., Liu X., Zhong K. *et al.* 2016. Effect of steam explosion-assisted extraction on phenolic acid profiles and antioxidant properties of wheat bran. *Journal of the Science of Food & Agriculture*, 96(10):3484-3491.
- Mateo A.N., Hemery Y.M., Bast A. and Haenen G.R. 2012. Optimizing the bioactive potential of wheat bran by processing. *Food & Function*, 3(4):362-375.
- Neacsu M., McMonagle J., Fletcher R.J., Hulshof T. *et al.* 2017. Availability and dose response of phytophenols from a wheat bran rich cereal product in healthy human volunteers. *Molecular Nutrition & Food Research*, 61(3):1600202.
- Pandey K.B. and Rizvi S.I. 2009. Plant polyphenols as dietary antioxidants in human health and disease. *Oxidative Medicine & Cellular Longevity*, 2(5):270-278.

- Perales-Sánchez J.X.K., Cuauhtémoc R.M., Gómez-Favela M.A., Jorge M.C. *et al.* 2014. Increasing the antioxidant activity, total phenolic and flavonoid contents by optimizing the germination conditions of amaranth seeds. *Plant Foods for Human Nutrition*, 69(3):196-202.
- Pilli T.D., Legrand J., Derossi A. and Severini C. 2010. Study on interaction among extrusion-cooking process variables and enzyme activity: evaluation of extrudate structure. *Journal of Food Process Engineering*, 33(1):65-82.
- Ramos-Enríquez J.R., Ramírez-Wong B., Robles-Sánchez R.M., Robles-Zepeda R.E. *et al.* 2018a. Effect of Extrusion Conditions and the Optimization of Phenolic Compound Content and Antioxidant Activity of Wheat Bran Using Response Surface Methodology. *Plant Foods for Human Nutrition*, 73(5):1-7.
- Ramos-Enríquez J.R., Ramírez-Wong B., Robles-Sánchez R.M., Robles-Zepeda R.E. *et al.* 2018b. Effect of Extrusion Conditions and the Optimization of Phenolic Compound Content and Antioxidant Activity of Wheat Bran Using Response Surface Methodology. *Plant Foods for Human Nutrition*, 73(3):228-234.
- Re R., Pellegrini N., Proteggente A., Pannala A. *et al.* 1999. Antioxidant activity applying an improved ABTS radical cation decolorization assay. *Free radical biology and medicine*, 26(9-10):1231-1237.
- Rice-Evans C., Miller N. and Papaganda G. 1999. Antioxidant activity applying an improved ABTS radical cation decolorization assay. *Free Radic Biol Med*, 26:1231-1237.
- Rosicka-Kaczmarek J., Komisarczyk A. and Nebesny E. 2018. Heteropolysaccharide preparations from rye and wheat bran as sources of antioxidants. *Journal of Cereal Science*, 81:37-43.
- Vázquez C.V., Rojas M.G.V., Ramírez C.A., Chávez-Servín J.L. *et al.* 2015a. Total phenolic compounds in milk from different species. Design of an extraction technique for quantification using the Folin-Ciocalteu method. *Food Chemistry*, 176:480-486.
- Vázquez C.V., Rojas M.G.V., Ramírez C.A., Chávez-Servín J.L. *et al.* 2015b. Total phenolic compounds in milk from different species. Design of an extraction technique for quantification using the Folin-Ciocalteu method. *Food Chemistry*, 176(8):480-486.
- Zhen J., Villani T.S., Guo Y., Qi Y. *et al.* 2016. Phytochemistry, antioxidant capacity, total phenolic content and anti-inflammatory activity of *Hibiscus sabdariffa* leaves. *Food Chemistry*, 190(2016):673-680.
- Zheng X., Zhang R. and Liu C. 2015. Extraction and antioxidant activity of phenolic compounds from wheat bran treated by steam explosion. *Tropical Journal of Pharmaceutical Research*, 14(10):1857-1863.

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