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Optimisation of Extrusion Variables for the Production of Corn Snack Products Enriched with Defatted Hemp Cake

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Abstract

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The effects of defatted hemp cake added to corn grits (at ratios of 0, 5, and 10% DM), the moisture content of the mixtures (15, 20, and 25%) and the temperature in the extruder ejection zone (150, 165, and 180°C) on the physical properties of extruded products have been investigated. Statistical optimisation of investigated extrusion conditions using the response surface methodology was performed. The hemp cake was completely defatted by means of a supercritical CO_2 extraction. The extrusion was performed on a laboratory single screw extruder and regulated by the following parameters: temperature in the dosing and compression zone (120 and 150°C), screw compression ratio (4 : 1), round die (4 mm). The change of extrusion process conditions significantly affected the physical properties of produced snacks. The expansion ratio ranged between 1.38 and 3.11, bulk density between 0.14 and 0.49 g/cm^3, hardness between 18.15 and 70.62 N, fracturability from 3.65 to 10.38 mm, and the total colour change between 3.25 and 24.73.

Keywords: extruded snacks; hemp; response surface methodology

Expanded snack products are very popular among both children and adults. Since they are calorie-dense products and have a low nutritional value, recently there have been attempts to improve their composition by adding different raw materials rich in polyphenols, vitamins, fibres, healthy fats etc. (Jozinović *et al.* 2016).

Hemp seed is valued for its excellent nutrient composition; it contains 25–35% lipid, 20–25% protein, and 20–30% carbohydrate; 10–15% insoluble fibre and an array of minerals; it is rich in linoleic and linolenic acids, and in essential amino acids. It has been reported that hemp seed oil has a beneficial effect on blood pressure and cholesterol, antimicrobial and antioxidant properties (Hong *et al.* 2015). As far as the production of cold pressed hemp oil is concerned, a certain amount of oil remains in the cake and it is not possible to recover it using mechanical pressing. Therefore, such pressed hemp cake is a by-product of oil production. Nowadays, food wastes are considered a cheap source of valuable components since modern technologies enable recovery of target compounds and their recycling inside the food chain as functional additives in different products. Waste discharge does not account for the potentiality of its re-utilization inside the food chain. For this reason, the term 'food by-products' is increasingly used among scientists in order to notify that 'food wastes' are ultimate substrates for the recapture of functional compounds and the development of new products with a market value.

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In our previous work (Aladić *et al.* 2014) it was shown that it is possible to recover the oil from pressed hemp cake by means of supercritical CO_2, thus maximising the oil yield. The extraction residue appears as defatted press cake rich in protein, fibres and carbohydrates, and as such it is a valuable raw material for further application in the production of functional and enriched products.

Extrusion, as a continuous process with high versatility and productivity, represents a good avenue to the incorporation of different types of by-products into ready-to-eat snacks. In addition, incorporation of these by-products can improve the nutritional value of extruded products which are otherwise carbohydrate-rich, high glycaemic products (Paraman *et al.* 2015).

The objectives of this work are as follows: (1) to obtain completely defatted hemp cake (DHC) by the supercritical CO_2 extraction technique which is expected to be used in the development of new functional and enriched products based on the extrusion process (first time reported), (2) to investigate the effects of DHC addition and process parameters during extrusion on the physical properties of snack products, and (3) to perform statistical optimization of extrusion conditions using the response surface methodology (RSM).

This research is very significant because it gives an insight into the possibilities of exploiting food industry by-products, such as pressed hemp cake, in the production of highly valued corn snack products.

MATERIAL AND METHODS

Materials. The corn grits (harvest 2015) were donated by the mill Đakovo, Žito Company Ltd. (Croatia). The hemp (*Cannabis sativa* L.) seeds of genotype Fedora 17 (oil content 31.69%) were obtained from the family farm Organica Vita (Croatia) in 2015. The purity of CO_2 used for the extraction was 99.97% (w/w) (Messer, Croatia). *n*-Hexane was provided from Merck KGaA (Germany). All the other chemicals and reagents were of an analytical reagent grade.

The moisture content of the material was determined according to ISO 6540:1980, protein ISO 5983-2:2005, fat ISO 6492:1999, ash ISO 5984:2002, crude fibre ISO 6865:2000.

Production of defatted hemp cake. The DHC was produced using two processes – screw pressing, followed by supercritical CO_2 extraction. The pressing of hemp seeds was performed in a screw expeller (Model SPU 20; Senta, Serbia) with the capacity of 20–25 kg/hour. The nozzle size used was 6 mm, the frequency 20 Hz and the head press temperature 60°C. After the pressing, the residual oil in the pressed cake was measured by a traditional laboratory Soxhlet extraction (ISO 6492:1999).

The residual oil that remained in the cake after pressing was extracted with supercritical CO_2 in a supercritical fluid extraction (SFE) system explained in detail elsewhere (Jokić *et al.* 2014). The pressed hemp cake was placed into an extractor vessel and the residual oil was collected in a separator in previously weighed glass tubes at 1.5 MPa and 25°C. The extraction process was performed in the following extraction conditions: pressure 30 MPa, temperature 40°C, solvent flow rate 2 kg/h and extraction time 2 h (after that time the hemp cake was totally defatted). For each extraction experiment, 250 g of pressed hemp cake was used until the total amount of collected DHC reached about 1 kg (a quantity adequate for further extrusion experiments). Before the extrusion process, the DHC had been milled at a laboratory mill (MF10; IKA, Germany) with a 2 mm sieve.

The chemical composition of corn grits, hemp seeds and DHC is shown in Table 1.

Experimental design and data analysis. The extrusion experiments were planned to be conducted with a Box-Behnken design (Bas & Boyaci 2007). Three different levels were used for each of the following factors: the content of DHC added to corn grits (X_1), the moisture content in samples before extrusion (X_2) and the extrusion temperature in the extruder ejection zone (X_3). The coded and uncoded levels of the independent variables and the experimental design are given in Table 2.

The following responses were evaluated: expansion ratio (Y_1), bulk density (Y_2), hardness (Y_3), fracturability (Y_4), and total colour change ΔE (Y_5). A second-order polynomial equation (Equation 1) was used to express the responses investigated (Y) as a function of the coded independent variables:

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i X_i + \sum_{i=1}^{k} \beta_{ii} X_i^2 + \sum_{\substack{i=1 \\ i<j}}^{k-1} \sum_{j=2}^{k} \beta_{ij} X_i X_j \quad (1)$$

The statistical analysis was performed using RSM software Design-Expert® v9 (Stat Ease, USA).

Blend preparation. The blend preparation (based on 2 kg DM) was performed according to the ex-

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perimental design obtained by RSM. The moisture content of samples was regulated by spraying with an estimated amount of distilled water, with continuous mixing in a laboratory mixer (KMM020; Kenwood, the Netherlands). The prepared mixtures were then put into plastic bags (one bag per sample) and stored overnight in the refrigerator at 4°C in order to equilibrate the moisture. Before extrusion, the samples were brought to the room temperature.

Extrusion. The prepared samples were extruded in a laboratory single-screw extruder (Model 19/20DN; Brabender GmbH, Germany). The extrusion parameters were as follows: screw 4 : 1; die 4 mm; temperature profile in the first (dosing) and second (compression) zone 120 and 150°C; screw speed 100 rpm; dosing speed 20 rpm. The temperature of the ejection zone was changed in the experiment according to the design given in Table 2. After extrusion, the samples were air-dried overnight. For the analysis of the expansion ratio, bulk density and texture, dried extrudates were packed into plastic bags and stored in the dark prior to the analysis. The remaining portion of the dried extrudates were milled at a laboratory mill (MF10; IKA, Germany) with a 2 mm sieve and stored in sealed plastic bags at 4°C until further analysis.

Expansion ratio. The expansion ratio (ER) was determined according to Brnčić *et al.* (2008), where the expansion ratio was calculated as

$$\text{ER} = \text{extrudate diameter (mm)/die diameter (mm)} \quad (2)$$

For each sample five measurements were taken and the results were expressed as a mean value.

Bulk density. The bulk density (BD) was calculated according to the method of Alvarez-Martinez *et al.* (1988):

$$\text{BD (g/cm}^3) = 4m / \pi d^2 L \quad (3)$$

where: m – mass (g); L – length of extrudate (cm); d – diameter (cm)

For each sample five measurements were taken and the results were expressed as a mean value.

Texture analysis. The texture properties of extrudates were determined according to Jozinović *et al.* (2016) with a texture analyser (TA.XT2Plus) and by the Texture Exponent 32 software (both from Stable Micro Systems, UK), using a method 'Measurement of the hardness and fracturability of pretzel sticks', which represents the cut test by using a Warner-Bratzler shear blade with guillotine probe, with the following settings: pre-test speed 1.0 mm/s; test speed 1.0 mm/s; post-test speed 10.0 mm/s; distance 30 mm. For the purpose of texture analysis samples were standardized to a length of 3 cm (Paula & Conti-Silva 2014). The obtained results regarding hardness and fracturability were expressed as the mean of 10 replications.

Colour. The colour was measured according to Jozinović *et al.* (2016) using a Chroma Meter CR-400 (Konica Minolta, Japan) with granular materials attached and the measuring head with 2° observer and illuminant C. The instrument was calibrated using a white standard calibration plate and the colour was expressed in CIE-Lab parameters as L^* (whiteness/darkness), a^* (redness/greenness), and b^* (yellowness/blueness). The total colour change (ΔE) was calculated as:

$$\Delta E = \sqrt{(L - L_0)^2 + (b - b_0)^2 + (a - a_0)^2} \quad (4)$$

where: 0 – initial colour values of raw corn grits

For each sample five measurements were taken and the results were expressed as a mean value.

Thermophysical properties of samples with poor expansion and textural properties. With the aim to investigate the possible application of these products as extruded modified flours in the bakery industry, the analyses of paste viscosity, water absorption index and water solubility index were performed on extrudates with poor expansion and textural properties. Moreover, the extrudates did not have any suitable textural properties to be defined as snack products.

Paste viscosity. The pasting properties of non-extruded and extruded samples (14% DM, 115 g total weight) were measured using a Micro Visco-Amylograph (Model 803202; Brabender GmbH, Germany). The flour suspensions were heated at 7.5°C/min from 30°C to 92°C, held at 92°C for 5 min, cooled at 7.5°C/min to 50°C, and held at 50°C for 1 minute. Each sample was measured twice and the results were expressed as a mean value.

Water absorption index (WAI) and water solubility index (WSI). WAI and WSI were determined according to Anderson *et al.* (1969) and calculated by the following equations:

$$\text{WAI (g/g)} = \text{weight of gel/dry weight of sample} \quad (5)$$

$$\text{WSI (\%)} = (\text{weight of dry solids in supernatant/dry weight of sample}) \times 100 \quad (6)$$

Each sample was measured twice and the results were expressed as a mean value.

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Table 1. Chemical composition of corn grits, hemp seeds, and defatted hemp cake

Parameter (%)	Corn grits	Hemp seeds	Defatted hemp cake
Protein	7.44 ± 0.00	23.06 ± 0.08	34.5 ± 0.26
Fat	1.04 ± 0.09	33.56 ± 0.03	0.51 ± 0.03
Crude fibre	1.18 ± 0.20	31.69 ± 0.98	60.38 ± 1.42
Ash	0.35 ± 0.01	6.05 ± 0.14	9.78 ± 0.02
Moisture	13.61 ± 0.01	6.61 ± 0.01	7.57 ± 0.04

Statistical analysis. The experimental data for the determined thermophysical properties (paste viscosity, WAI and WSI) were subjected to analysis of variance (ANOVA) and Fisher's least significant difference (LSD) with the significance defined at $P < 0.05$. All statistical analyses were carried out using the STATISTICA v10.0 software program (StatSoft Inc., USA).

RESULTS AND DISCUSSION

Characteristics of corn grits and defatted hemp cake. The initial oil content in hemp seeds turned out to be 31.69% (Table 1), which corresponds to earlier reported values (Anwar *et al.* 2006). The moisture content was 6.61%. The residual oil after screw press extraction in the hemp cake was measured to be 6.72% and such pressed hemp cake was used in supercritical CO_2 extraction experiments with the aim to obtain DHC in further extrusion experiments.

The chemical composition of corn grits and DHC included moisture (13.61 and 7.57%), protein (7.44 and 34.50%), fat (1.04 and 0.51%), ash (0.35 and 9.78%) and crude fibre (1.18 and 60.38%), respectively (Table 1).

It was determined that the remaining oil in DHC after supercritical CO_2 extraction was very low (0.51%), which means that complete recovery of the oil from pressed hemp cake using supercritical CO_2 is possible and such DHC can be further used in the development of new functional and enriched products based on the extrusion process. Furthermore, such DHC is rich in protein and fibre.

Optimisation of extrusion process. In this study, a Box-Behnken design (Table 2) was used to optimise the extrusion process variables in order to achieve the highest expansion ratio, lower bulk density, lower hardness, higher fracturability, and minor total colour change. The operating conditions varied at 3 levels and the design required 17 experiments with five replicates for the central point. The content of DHC (0, 5, and 10%), investigated moisture content (15, 20, and 25%) and extrusion temperatures (150, 165, and 180°C) were established according to preliminary trials and our previous investigations focused on the

Table 2. Box-Behnken design for experimental runs with different combinations of extrusion variables

Run	DHC (%)	Moisture content (%)	Temperature (°C)	Expansion ratio	Bulk density (g/cm^3)	Hardness (N)	Fracturability (mm)	ΔE
1	+1 (10)	0 (20)	+1 (180)	2.23 ± 0.07	0.24 ± 0.01	44.10 ± 2.07	6.34 ± 0.21	24.73
2	+1 (10)	+1 (25)	0 (165)	1.38 ± 0.06	0.49 ± 0.01	70.62 ± 3.49	3.65 ± 0.41	20.95
3	−1 (0)	0 (20)	+1 (180)	2.38 ± 0.02	0.19 ± 0.01	37.01 ± 2.82	8.11 ± 0.77	7.71
4	0 (5)	0 (20)	0 (165)	2.58± 0.03	0.20 ± 0.01	36.78 ± 6.14	7.85 ± 0.71	14.88
5	0 (5)	−1 (15)	+1 (180)	2.80 ± 0.05	0.15 ± 0.01	22.22 ± 1.47	9.43 ± 0.29	19.52
6	0 (5)	+1 (25)	−1 (150)	1.39 ± 0.01	0.40 ± 0.02	66.61 ± 2.40	4.86 ± 0.56	12.82
7	0 (5)	−1 (15)	−1 (150)	2.77 ± 0.09	0.16 ± 0.01	24.95 ± 2.05	9.29 ± 0.46	14.27
8	0 (5)	0 (20)	0 (165)	2.29 ± 0.06	0.24 ± 0.01	38.92 ± 4.06	7.37 ± 0.31	15.83
9	0 (5)	0 (20)	0 (165)	2.31 ± 0.04	0.23 ± 0.01	38.08 ± 4.81	7.34 ± 0.34	15.60
10	0 (5)	+1 (25)	+1 (180)	1.74 ± 0.01	0.35 ± 0.02	58.76 ± 4.30	5.38 ± 0.61	17.51
11	+1 (10)	0 (20)	−1 (150)	2.20 ± 0.09	0.25 ± 0.02	51.20 ± 3.97	6.13 ± 0.59	17.96
12	0 (5)	0 (20)	0 (165)	2.31 ± 0.10	0.23 ± 0.01	38.50 ± 3.67	7.34 ± 0.92	15.66
13	0 (5)	0 (20)	0 (165)	2.31 ± 0.05	0.22 ± 0.01	37.51 ± 5.15	7.29 ± 0.48	15.17
14	+1 (10)	−1 (15)	0 (165)	2.51 ± 0.02	0.16 ± 0.01	26.24 ± 1.93	9.00 ± 0.58	21.99
15	−1 (0)	−1 (15)	0 (165)	3.11 ± 0.05	0.14 ± 0.01	18.15 ± 1.61	10.38 ± 0.79	6.49
16	−1 (0)	0 (20)	−1 (150)	2.24 ± 0.09	0.24 ± 0.01	40.35 ± 2.15	7.17 ± 0.59	6.28
17	−1 (0)	+1 (25)	0 (165)	1.73 ± 0.04	0.39 ± 0.03	59.55 ± 3.84	5.16 ± 0.37	3.25

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production of expanded snack products enriched with different raw materials (Obradović *et al.* 2015), and the investigations of other researchers who studied the application of by-products of the oil production industry were used (Yağci & Göğüş 2008; Nascimento *et al.* 2012).

The data in Table 2 indicate that all the investigated variables varied significantly according to the applied process parameters of extrusion. The expansion ratio ranged between 1.38 and 3.11, bulk density between 0.14 and 0.49 g/cm^3, hardness between 18.15 and 70.62 N, fracturability between 3.65 and 10.38 mm, and total colour change between 3.25 and 24.73.

The effect of linear, quadratic or interaction coefficients on the response was tested for significance by ANOVA. The regression coefficients of intercept, linear, quadratic, and interaction terms of the model were calculated using the least squares method. The degree of significance of each factor is represented in Table 3 by its P. If $P < 0.05$, the factor has a significant influence on the process (for a confidence level of 0.95).

Table 3 shows that the linear term of DHC as well as the linear term of moisture exhibits a statistically significant effect on all the investigated responses. The obtained results are similar to the results obtained by other authors. Nascimento *et al.* (2012) have reported that the expansion ratio decreases as a result of an increasing protein content because it has a limited or non-puffing capacity compared to starch. Similar results for expanded corn extrudates with added whey protein were also published by Brnčić *et al.* (2008). In our study, the addition of DHC resulted in an increase of bulk density. Other authors have also connected the increase of bulk density with the addition of brewer's spent grain (Stojceska *et al.* 2009), lentil flour (Lazou & Krokida 2010) and partially defatted hazelnut flour in a feed mixture (Yağci & Göğüş 2008). The results obtained in this study also show that the addition of DHC resulted in a decrease of fracturability, whereas hardness of extrudates increased. According to Akdogan *et al.* (1997) the changes in the texture are caused by the loss of moisture, formation or disintegration of emulsions and gels, and by hydrolysis of polymeric carbohydrates, coagulation of protein or hydrolysis of protein. It is evident from our results that the addition of DHC significantly affects the total colour change and brings to an increase in the darkness of extrudates. Other authors (Ilo *et al.* 1999; Wang & Ryu 2013) have demonstrated that the colour changes of extrudates are primarily caused by the formation of Maillard reaction products.

As observed by Ryu and Ng (2001), the expansion ratio decreased with an increase in the feed moisture content. The bulk density of extrudates was higher when samples were extruded using a higher moisture content. Other researches where it was concluded that an increase of moisture content increased the bulk density of extrudates (Garber *et al.* 1997; Hagenimana *et al.* 2006) also show the same trend. The results obtained in our study show that moisture content significantly affects the texture of extrudates. Products extruded at a higher moisture content had greater hardness and lower fracturability. Brnčić *et al.* (2006) also showed that the moisture content had the greatest impact on the texture of extrudates.

The temperature of extrusion in our study had a statistically significant influence only on the hardness and the total colour change of samples. The hardness of extrudates increased with the increase of temperature.

The interactions between the DHC addition and temperature in the extruder ejection zone had a statistically significant influence on the total colour change ($P < 0.01$). Other interactions between the investigated variables did not show a statistically

Table 3. Corresponding P for selected responses for each obtained coefficient

Response	X_1	X_2	X_3	X_1^2	X_2^2	X_3^2	X_1X_2	X_1X_3	X_2X_3
Expansion ratio	0.0154	< 0.0001	0.1685	0.5575	0.0815	0.4849	0.3565	0.6771	0.2469
Bulk density	0.0296	< 0.0001	0.1122	0.1579	0.0023	0.3269	0.1306	0.4203	0.4203
Hardness	< 0.0001	< 0.0001	0.0003	0.0014	0.0015	0.0043	0.2217	0.1344	0.0546
Fracturability	0.0002	< 0.0001	0.0616	0.0725	0.9671	0.4827	0.8278	0.2454	0.5304
ΔE	<0.0001	<0.0001	0.0005	0.0018	0.0020	0.0052	0.2568	0.1632	0.0716

X_1 – DHC; X_2 – moisture; X_3 – temperature; $P < 0.01$ – highly significant; $0.01 \leq P < 0.05$ – significant; $P \geq 0.05$ – not significant

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significant influence on the obtained parameters (Table 3).

The ANOVA results are presented in Table 4. The regression models for all the investigated responses were highly significant according to *P* with satisfactory coefficients of determination (R^2) in the range of 0.9680–0.9973. These results show that the predicted models for the investigated responses were adequate, as indicated by the error analysis that showed a non-significant lack of fit. These results show that the predicted models for the investigated responses were adequate, indicating that the second-order polynomial model could therefore be effectively used to represent the relationship between the selected parameters.

The best way to visualise the effect of independent variables on dependent ones is to draw surface response plots of a proposed model like those given in Figure 1, which shows the combined effects of process variables on expansion ratio, bulk density, hardness, fracturability, and total colour change of extruded products. The effect of moisture content, content of DHC and temperature in the extruder ejection zone on the expansion ratio shows that an increase in the moisture content significantly reduces the expansion ratio. Also, the addition of

Table 4. Analysis of variance (ANOVA) of the modelled responses

Source	Sum of squares	Degree of freedom	Mean square	*F*	*P*
Expansion ratio					
Model	3.39	9	0.38	23.53	0.0002
Residual	0.11	7	0.016		
Lack of fit	0.078	3	0.026	3.08	0.1529
Pure error	0.034	4	0.0008		
Total	3.51	16			
Bulk density					
Model	0.15	9	0.017	30.99	< 0.0001
Residual	0.0031	7	0.00054		
Lack of fit	0.0029	3	0.00097	4.20	0.0996
Pure error	0.00009	4	0.00023		
Total	0.16	16			
Hardness					
Model	3702.63	9	411.40	282.53	< 0.0001
Residual	10.19	7	1.46		
Lack of fit	7.37	3	2.46	3.48	0.1297
Pure error	2.82	4	0.71		
Total	3712.83	16			
Fracturability					
Model	50.66	9	5.63	49.73	< 0.0001
Residual	0.79	7	0.11		
Lack of fit	0.58	3	0.19	3.57	0.1254
Pure error	0.22	4	0.054		
Total	51.45	16			
Δ*E*					
Model	556.17	9	61.80	119.97	< 0.0001
Residual	3.61	7	0.52		
Lack of fit	2.99	3	1.00	6.52	0.0508
Pure error	0.61	4	0.15		
Total	559.78	16			

R^2 = 0.9680

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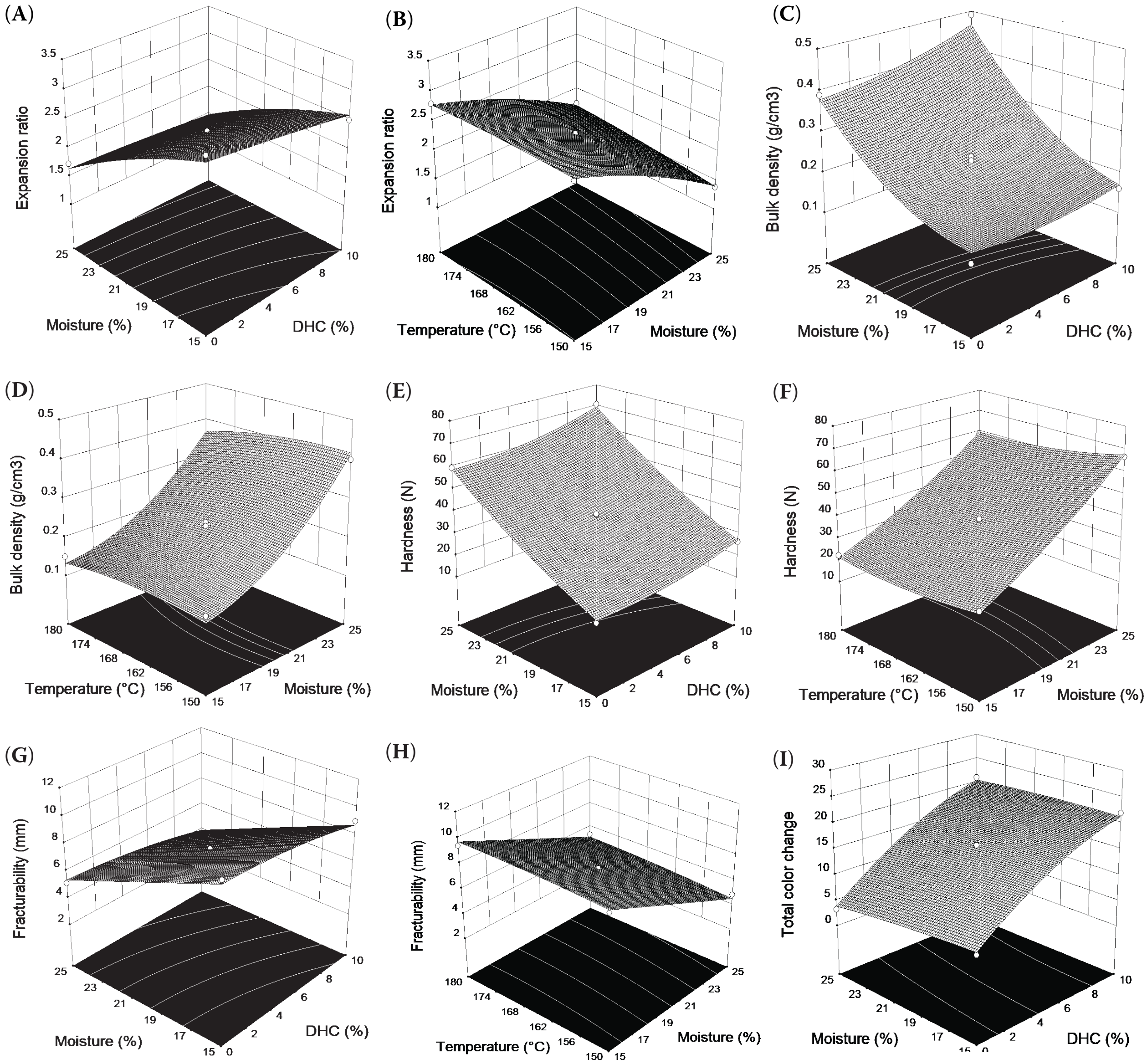

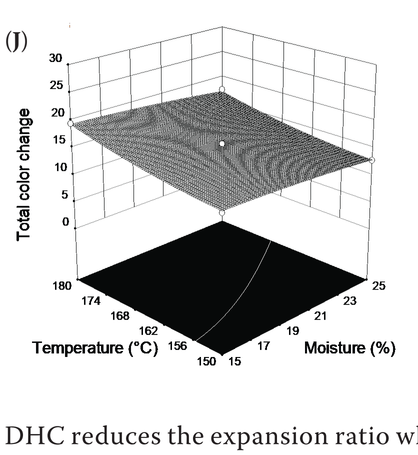

Figure 1. Response surface plots showing the combined effects of process variables on expansion ratio (**A**, **B**), bulk density (**C**, **D**), hardness (**E**, **F**), fracturability (**G**, **H**), and total colour change (**I**, **J**) of extruded products

DHC reduces the expansion ratio while temperature does not significantly affect the expansion ratio. Furthermore, the increase in moisture leads to an increase in the bulk density of the extrudate, and the temperature in the extruder ejection zone has no significant effect on the bulk density. The increase in the moisture content results in increased hardness. Reducing the temperature and increasing the content in DHC result in the increased hardness of extrudates. The increase in the moisture content and

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increase in the content of DHC reduce the fracturability of extrudates while temperature does not have a significant impact on the fracturability of the extrudates. Increase of moisture content, content of DHC and temperature in the extruder ejection zone affect an increase in the total colour change of obtained extrudates.

The final goal of RSM is process optimisation where the developed model can be used for process simulation. Optimisation is an essential tool for the efficient operation of different processes to yield a highly acceptable product (Bas & Boyaci 2007). Optimal extrusion parameters were obtained for all the investigated responses. The treatment of multiple responses and selection of the optimal conditions were based on the desirability function D. In this study, the desirability was set to show the best conditions for extrusion with the addition of DHC, which maximise the expansion ratio, minimise bulk density, minimise hardness, maximise fracturability and minimise the total colour change of a final product. By applying the desirability function method, the optimum extrusion conditions were obtained using 5% of DHC, moisture 15% and temperature 150°C. The verification and validity of the generated mathematical model were performed in these optimal conditions. The experimental and predicted values were further compared. The predicted expansion ratio was 2.78, bulk density 0.14 g/cm^3, hardness 19.34 N, fracturability 10.19 mm and the total colour change 14.47. At the calculated optimal conditions, the following experimental data were obtained: expansion ratio 2.77, bulk density 0.16 g/cm^3, hardness 24.95 N, fracturability 9.29 mm and total colour change was 14.27, namely. A higher degree of compliance between the experimental and predicted values is noticeable.

Thermophysical properties of samples with poor expansion and textural properties. The extruded samples, characterised by poor expansion and high bulk density and hardness, were milled to produce modified flours. The viscosity, WAI and WSI of modified flours were determined and compared with the non-extruded blends (Table 5). The viscosity significantly decreased after extrusion. The most significant decline of peak and cold paste viscosity was observed for the samples with no DHC, which had the highest peak and cold paste viscosity before extrusion.

The peak viscosity reduction implies high starch damage and starch gelatinization during processing (Hagenimana *et al.* 2006). DHC apparently protects

Table 5. Viscosity, water absorption index (WAI), and water solubility index (WSI) of samples with poor expansion and textural properties

Run	Hemp content (%)	Moisture (%)	Temperature (°C)	Viscosity (BU)			WAI (g/g)	WSI (%)
				peak	hot	cold paste		
Non-extruded samples								
1	10	20	180	348.5 ± 7.78^{e}	349.5 ± 6.36^{e}	636.5 ± 9.19^{e}	2.63 ± 0.03^{b}	3.38 ± 0.03^{c}
2	10	25	165	354.5 ± 0.71^{e,f}	357.5 ± 4.95^{e}	653.0 ± 14.14^{f}	2.64 ± 0.02^{b}	3.36 ± 0.07^{c}
6	5	25	150	430.0 ± 0.00^{g}	431.5 ± 3.54^{g}	839.0 ± 11.31^{g}	2.51 ± 0.01^{a}	3.16 ± 0.03^{b}
10	5	25	180	432.5 ± 2.12^{g}	435.0 ± 0.00^{g}	848.5 ± 3.54^{g}	2.51 ± 0.01^{a}	3.15 ± 0.01^{b}
11	10	20	150	367.0 ± 16.97^{f}	373.5 ± 17.68^{f}	668.5 ± 7.78^{f}	2.62 ± 0.00^{b}	3.37 ± 0.06^{c}
16	0	20	150	518.5 ± 6.36^{h}	522.5 ± 2.12^{h}	938.5 ± 4.95^{h}	2.45 ± 0.06^{a}	2.85 ± 0.02^{a}
17	0	25	165	584.0 ± 0.00^{i}	584.0 ± 1.41^{i}	1103.5 ± 7.78^{i}	2.46 ± 0.01^{a}	2.84 ± 0.02^{a}
Extruded samples								
1	10	20	180	129.0 ± 4.24^{b}	51.5 ± 0.71^{a}	165.5 ± 0.71^{a}	7.19 ± 0.02^{g}	13.37 ± 0.02^{h}
2	10	25	165	157.5 ± 4.95^{c}	110.5 ± 2.12^{c}	225.0 ± 1.41^{b}	6.77 ± 0.00^{c,d}	9.82 ± 0.01^{e}
6	5	25	150	154.5 ± 2.12^{c}	147.5 ± 2.12^{d}	294.5 ± 0.71^{c}	6.82 ± 0.03^{d,e}	8.92 ± 0.11^{d}
10	5	25	180	182.0 ± 8.49^{d}	95.5 ± 6.36^{b}	233.0 ± 2.83^{b}	6.85 ± 0.07^{e}	10.00 ± 0.03^{f}
11	10	20	150	102.5 ± 6.36^{a}	64.5 ± 6.36^{a}	175.5 ± 6.36^{a}	7.07 ± 0.05^{f}	13.27 ± 0.02^{h}
16	0	20	150	129.0 ± 8.49^{b}	120.5 ± 7.78^{c}	284.5 ± 12.02^{c}	7.02 ± 0.01^{f}	12.33 ± 0.05^{g}
17	0	25	165	165.5 ± 2.12^{c}	161.0 ± 2.83^{d}	332.0 ± 5.66^{d}	6.75 ± 0.02^{c}	9.74 ± 0.07^{e}

Data are expressed as mean value of replication (n) ± SD; $^{a-f}$the same letter in the same column of analysed variable indicates no significant differences (Duncan's test, $P < 0.05$)

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starch from damage by physically protecting starch molecules and/or through the complexation of starch and oligosaccharides from corn with proteins and fibres from DHC. The buckwheat and chestnut flour in our previous research had the opposite effect – the decrease of viscosities was more evident in corn grits/flour mixtures than in pure corn grits, however, these flours are rich in starch, and had a significantly lower content of protein and crude fibre (Jozinović *et al.* 2012). Cold paste viscosity is an indicator of starch retrogradation during cooling and is equivalent to the viscosity at the end of the cooling phase. DHC addition significantly decreases cold paste viscosity values indicating better stability of starch in a suspension, again most probably due to the protective effect of protein and fibre. The extrusion resulted in a further decrease of cold paste viscosity, as reported in our previous study (Jozinović *et al.* 2012) and those other groups of authors (Hagenimana *et al.* 2006). WAI and WSI increased proportionally to the hemp content added to corn grits, most likely due to an increase of fibre content. After extrusion, a significant increase of WAI and WSI was observed in all samples, due to the combined effect of starch damage and swelling, fibre swelling and protein denaturation (Singh *et al.* 2007).

CONCLUSION

RSM was used to model and optimise the experimental conditions to produce corn snack products enriched with defatted hemp cake. The obtained results showed that the increase of the DHC content and the moisture of mixtures both decrease the expansion ratio and fracturability, and increase the bulk density and hardness of extrudates. Temperature had a significant influence only on hardness and total colour change. After the extrusion process, WAI and WSI significantly increased, while the values of peak, hot and cold paste viscosity decreased. The optimization procedure indicated that optimum extrusion conditions were obtained with the following experimental settings: temperature in the extruder ejection zone 150°C, moisture content of the mixtures 15% and DHC addition to corn grits at the ratio of 5%. Furthermore, the obtained results of physical properties of extrudates suggest that DHC, as a by-product of oil production and cheap raw material, can be successfully incorporated into corn grits with the aim to produce snack products or modified flours. Further research is needed to evaluate the influence of DHC addition on the nutritional value of extruded snacks and modified flours.

References

Akdogan H., Tomas R.L., Oliveira J.C. (1997): Rheological properties of rice starch at high moisture contents during twin-screw extrusion. Lebensmittel-Wissenschaft & Technologie, 30: 488–496.

Aladić K., Jokić S., Moslavac T., Tomas S., Vidović S., Vladić J., Šubarić D. (2014): Cold pressing and supercritical CO_2 extraction of hemp (*Cannabis sativa*) seed oil. Chemical and Biochemical Engineering Quarterly, 28: 481–490.

Alvarez-Martinez L., Kondury K.P., Harper J.M. (1988): A general model for expansion of extruded products. Journal of Food Science, 53: 609–615.

Anderson R.A., Conway H.F., Pfeifer V.F., Griffin E.L. (1969): Gelatinization of corn grits by roll and extrusion cooking. Cereal Science Today, 14: 4–12.

Anwar F., Latif S., Ashraf M. (2006): Analytical characterization of hemp (*Cannabis sativa*) seed oil from different agro-ecological zones of Pakistan. Journal of the American Oil Chemists' Society, 83: 323–329.

Bas D., Boyaci I.H. (2007): Modeling and optimization I: Usability of response surface methodology. Journal of Food Engineering, 78: 836–845.

Brnčić M., Tripalo B., Ježek D., Semenski D., Drvar N., Ukrainczyk M. (2006): Effect of twin-screw extrusion parameters on mechanical hardness of direct-expanded extrudates. Sadhana, 31: 527–536.

Brnčić M., Ježek D., Rimac Brnčić S., Bosiljkov T., Tripalo B. (2008): Influence of whey protein concentrate addition on textural properties of corn flour extrudates. Mljekarstvo, 58: 131–149.

Garber B.W., Hsieh F., Huff H.E. (1997): Influence of particle size on the twin-screw extrusion of corn meal. Cereal Chemistry, 74: 656–661.

Gutkoski L.C., El-Dash A.A. (1999): Effect of extrusion process variables on physical and chemical properties of extruded oat products. Plant Foods for Human Nutrition, 54: 315–325.

Hagenimana A., Ding X., Fang T. (2006): Evaluation of rice flour modified by extrusion cooking. Journal of Cereal Science, 43: 38–46.

Hong S., Sowndhararaja K., Joo T., Lim C., Cho H., Kim S., Kim G.Y., Jhoo J.W. (2015): Ethanol and supercritical fluid extracts of hemp seed (*Cannabis sativa* L.) increase gene expression and antioxidant enzymes in HepG2 cells. Asian Pacific Journal of Reproduction, 4: 147–152.

doi: 10.17221/83/2017-CJFS

Ilo S., Liu Y., Berghofer E. (1999): Extrusion cooking of rice flour and amaranth blends. LWT-Food Science and Technology, 32: 79–88.

Jokić S., Moslavac T., Bošnjak A., Aladić K., Rajić M., Bilić M. (2014): Optimization of walnut oil production. Croatian Journal of Food Science and Technology, 6: 27–35.

Jozinović A., Šubarić D., Ačkar Đ., Babić J., Klarić I., Kopjar M., Valek Lendić K. (2012): Influence of buckwheat and chestnut flour addition on properties of corn extrudates. Croatian Journal of Food Science and Technology, 4: 26–33.

Jozinović A., Šubarić D., Ačkar Đ., Babić J., Miličević B. (2016): Influence of spelt flour addition on properties of extruded products based on corn grits. Journal of Food Engineering, 172: 1–37.

Lazou A., Krokida M. (2010): Structural and textural characterization of corn–lentil extruded snacks. Journal of Food Engineering, 100: 392–408.

Nascimento E.M.D.G.C., Carvalho C.W.P., Takeiti C.Y., Freitas D.D.G.C., Ascheri J.L.R. (2012): Use of sesame oil cake (*Sesamum indicum* L.) on corn expanded extrudates. Food Research International, 45: 434–443.

Obradović V., Babić J., Šubarić D., Jozinović A., Ačkar Đ., Klarić I. (2015): Influence of dried Hokkaido pumpkin and ascorbic acid addition on chemical properties and color of corn extrudates. Food Chemistry: 183: 136–143.

Paraman I., Sharif M.K., Supriyadi S., Rizvi S.S.H. (2015): Agro-food industry byproducts into value-added extruded foods. Food and Bioproducts Processing, 96: 78–85.

Paula A.M., Conti-Silva A.C. (2014): Texture profile and correlation between sensory and instrumental analyses on extruded snacks. Journal of Food Engineering, 121: 9–14.

Ryu G.H., Ng P.K.W. (2001): Effects of selected process parameters on expansion and mechanical properties of wheat flour and whole cornmeal extrudates. Starch-Stärke, 53: 147–154.

Singh B., Sekhon K.S., Singh N. (2007): Effects of moisture, temperature and level of pea grits on extrusion behaviour and product characteristics of rice. Food Chemistry, 100: 198–202.

Stojceska V., Ainsworth P., Plunkett A., İbanoğlu Ş. (2009): The effect of extrusion cooking using different water feed rates on the quality of ready-to-eat snacks made from food by-products. Food Chemistry, 114: 226–232.

Wang Y.Y., Ryu G.H. (2013): Physicochemical and antioxidant properties of extruded corn grits with corn fiber by CO_2 injection extrusion process. Journal of Cereal Science, 58: 110–116.

Yağcı S., Göğüş F. (2008): Response surface methodology for evaluation of physical and functional properties of extruded snack foods developed from food-by-products. Journal of Food Engineering, 86: 122–132.

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