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SECTIONAL EXPANSION INDEXAND MICROSTRUCTURAL CHANGES OF BEAN–BASED EXTRUDATES AS A FUNCTION OF EXTRUSION VARIABLES

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ИНДЕКС ПОПЕРЕЧНОГО РАСШИРЕНИЯ И МИКРОСТРУКТУРНЫЕ ИЗМЕНЕНИЯ ЭКСТРУДАТОВ НА ОСНОВЕ БОБОВ В ЗАВИСИМОСТИ ОТ ПАРАМЕТРОВ ЭКСТРУЗИИ

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Annotation. Bean seeds, einkorn wheat, and buckwheat were ground. Semolina obtained from them was mixed in the ratio of 50:40:10 (w/w/w). Samples were mixed with distilled water to be obtained various moisture contents and extruded in a laboratory single screw extruder (Brabender 20 DN, Germany) with die diameter 3 mm. The effect of extrusion variables: moisture content (16, 19, 22, 25, 28%), barrel temperature (120, 140, 160, 180, 200°C), screw speed (120, 140, 160, 180, 200 rpm), and screwcompression ratio (1:1, 2:1, 3:1, 4:1, 5:1) on the sectional expansion index (SEI) of the extrudates was studied using response surface methodology. The average SEI values ranged from 151 to 194%. Statistical analysis showed that all four variables had an effect on SEI. The regression equation generated from central composite rotatable design could be used to accurately predict the studied response. The expansion of the extrudates was reflected in their microstructure.

Keywords: bean-basedextrudates, sectional expansion index, microstructure, central composite rotatable design

Аннотация. Семена фасоли, однозернянки и гречихи измельчали и затем полученную из них манную крупу смешивали в соотношении 50:40:10 (вес/вес/вес). К образцам добавляли дистиллированную воду, чтобы получить различные значения влагосодержания, и экструдировали в лабораторном одношнековом экструдере (Brabender 20 DN, Германия) с диаметром сопла матрицы 3 мм. Влияние экструзионных переменных: влажность (16, 19, 22, 25, 28%), температура цилиндра (120, 140, 160, 180, 200 °C), скорость шнека (120, 140, 160, 180, 200 об/мин) и степень компрессии шнека (1:1, 2:1, 3:1, 4:1, 5:1) — на индекс поперечного расширения (SEI) экструдатов изучали с использованием методологии поверхности отклика. Средние значения SEI колебались от 151 до 194%. Статистический анализ показал, что все четыре переменные влияли на SEI. Уравнение регрессии, полученное из центрального композитного ротатабельного плана, можно использовать для точного прогнозирования изучаемой реакции. Расширение экструдатов отразилось на их микроструктуре.

Ключевые слова: экструдаты на основе фасоли, индекс поперечного расширения, микроструктура, ротатабельный план

Introduction. Extrusion is a high temperature and short time process. It has been used to develop various types of food products. The raw material characteristics (type, moisture content, physical state, chemical composition — quantity and type of starch, proteins, fats, sugars) and operational conditions of the extruder (temperature, pressure, screw speed, die diameter, shear force) are the two main factors that influence the characteristics of extruded products (Fellows, 2000).For the production of extruded foods with good quality, it is necessary to use raw materials with highstarch contents to allow gelling and expansion to provide the desired physical and chemical characteristics (Oliveira et al., 2015). That's why, this processing method is being commercially used for corn meal, rice, wheat flour or potato flour but it is hardly used for legumes seeds owing to the perception that legumes do not expand well in extrusion. It is necessary to mix the legumes with other starchy foods, e. g., cereals, to obtain extruded products of appropriate quality (Giménez et al., 2013; Bouvier and Campanella, 2014).

Foods are usually expanded by extrusion, hot-air puffing, deep fat frying, baking, and more recently by microwave heating. Extrudate expansion is a complex phenomenon, the consequence of several events such as biopolymer structural transformations and phase transitions, nucleation, extrudate swell, bubble growth, and bubble collapse, with bubble dynamics dominantly contributing to the expansion phenomenon (Moraru and Kokini, 2003). Processing conditions also affect the degree of expansion, since they dictate the type and extent of physical and chemical modifications that take place during extrusion which, in turn, affect the expansion (Giri and Bandyopadhyay, 2000; Cha et al., 2001).

The expansion index is used to evaluation for the expansion of extrudates. Itis an important parameter and can be controlled both by changing the type and origin of the components, and by varying the process conditions in the extruder.

Scanning electron microscopy (SEM) is a suitable modern method for establishing the structural changes that occur during extrusion processing and provides additional information on the physicochemical characteristics of the extrudates.

Thus, the aim of the present work was to study the effect of extrusion process variables on the sectional expansion index as well as microstructural changes of bean-based extrudates.

Materials and methods. Bean seeds (*Phaseoluscoccineus* L.), variety "Bivolare", were grown in an experiment station in the Rhodope Mountains, Bulgaria. Einkorn wheat (*Triticummonococcum*) and buckwheat (*Fagopyrum*) were grown in an experimental station, village of Lomets, municipality of Troyan, Bulgaria. Einkorn wheat, buckwheat, and bean seeds were ground using a hammer mill (SWH 20, Glen Creston LTD, UK) and passed through standard sieves. The resulting semolina was a particle size of 0.4–0.5 mm. The bean semolina, einkorn wheat semolina, and buckwheat semolina were mixed in the ratio of 50:40:10 (w/w/w). Samples of the prepared composite were mixed with distilled water to be obtained various moisture contents (16, 19, 22, 25, and 28%) according to the experimental design (Table 1).The wet materials were placed and kept in sealed plastic bags for 12 h in a refrigerator at 5°C and tempered for 2 h at room temperature prior to extrusion.

A laboratory single screw extruder (Brabender 20 DN, Germany) was used for extrusion. The extruder barrel (476.5 mm inlength and 20 mm in diameter) contained threesections and independently controlled die assemblyelectric heaters. Feed zone temperature andmetering zone temperature were kept constant at100 and 140 °C, respectively. The extruder die temperature was 120, 140, 160, 180, and 200 °C. The feed screw speed was fixed at 50 rpm and the screw speed was changing according to the experimental design (120, 140,160, 180, and 200 rpm). The compression ratio of the screw was1:1, 2:1, 3:1, 4:1, and 5:1 according to the experimental design. The die diameter was 3 mm.

Sectional expansion index (SEI,%) was measured as the ratio of the diameter of the extrudate to that of the die (3 mm). The diameter of extrudate was determined as the mean of 10 random measurements using a Vernier caliper.

$$SEI = \frac{D_e}{D_0}.100 \quad (1)$$

where D_e is average diameter of the extrudates (mm), D_o — diameter of the die (mm).

For scanning electron microscopy (SEM) examination, the semolina and extrudates were coated with gold as dry specimens with JEOLJFC-1200 coater. Microphotographs were obtained with JEOL-JSM-5510 SEM at magnification of ×600.

A central composite rotatable design in 27 runs of which 16 were for the factorial points, 8 were for axial points, and 3 were for centre pointswas used to investigate the effect of the extrusion variables: moisture content (X_1), barrel temperature (X_2), screw speed (X_3), and screw compression ratio (X_4) on the sectional expansion index (response, y) (Myers, 2016). The levels of the independent variables were established according to literature information and preliminary trials. The outline of the experimental design is given in Table 1.

A second order polynomial model for the dependent variable (SEI) was established to fit the experimental data:

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

where b_0 = intercepts, b_i are linear, b_{ii} are quadratic, and b_{ij} are interaction regression coefficient terms.

SYSTAT statistical software (SPSS Inc., Chicago, USA, version 7.1) and Excel were used to analyze the data results.

Results and discussion. Variation of response (expansion of bean-based extrudates) with independent variables (moisture content, barrel temperature, screw speed, and screw compression ratio) is shown in Table 1. Acomplete second order model was tested for its adequacy to decide the variation of response with the independent variables. To aid visualization of variation in response with respect to the processing variables was drawn three dimensional response surface (Figure 2).

The sectional expansion index values of the bean-based extrudates are shown in Table 1. The values vary from 151 to 194%. The greatest expansion of the extrudates is obtained at the lowest barrel temperature (from the experimental plan), and the smallest — at the lowest moisture content of the mixture fed for extrusion.

The results of the statistical analysis of variance (ANOVA) for the sectional expansion index are shown in Table 2. In this case, 5 effects had p-values less than 0.05, indicating that they are significantly different from zero at the 95.0% confidence level. The R-squared statistic is 0.86. The R-squared is defined as theratio of the explained variation to the total variation and is a measure of the degree of fit. As the R-squared value for the model is more than 80% it can be considered forfurther analysis (Saunders et al., 2012).The regression equation describing the effect of extrusion variables on SEI of the bean-based extrudates is given in Table 2. The coefficients in the regression equation can be used to examine the significance of each term relative to each other when used with coded values. Statistical analysis showed that all four variables had an effect on SEI ($p<0.05$).

Table 1

Central composite rotatable design in coded form and natural units of independentvariables and experimental data for sectional expansion index of bean-based extrudates

№	Coded levels				Actual levels				SEI (%)	
	X_1	X_2	X_3	X_4	X_1	X_2	X_3	X_4	Experimental	Predicted
1	–1	–1	–1	–1	19	140	140	2:1	156	167
2	1	–1	–1	–1	25	140	140	2:1	165	159
3	–1	1	–1	–1	19	180	140	2:1	181	171
4	1	1	–1	–1	25	180	140	2:1	155	159
5	–1	–1	1	–1	19	140	180	2:1	191	186
6	1	–1	1	–1	25	140	180	2:1	189	193
7	–1	1	1	–1	19	180	180	2:1	174	179
8	1	1	1	–1	25	180	180	2:1	183	182
9	–1	–1	–1	1	19	140	140	4:1	179	170
10	1	–1	–1	1	25	140	140	4:1	168	169
11	–1	1	–1	1	19	180	140	4:1	156	158
12	1	1	–1	1	25	180	140	4:1	159	154
13	–1	–1	1	1	19	140	180	4:1	168	170
14	1	–1	1	1	25	140	180	4:1	184	184
15	–1	1	1	1	19	180	180	4:1	153	148
16	1	1	1	1	25	180	180	4:1	163	158
17	–2	0	0	0	16	160	160	3:1	151	154
18	2	0	0	0	28	160	160	3:1	154	156
19	0	–2	0	0	22	120	160	3:1	194	193
20	0	2	0	0	22	200	160	3:1	165	171
21	0	0	–2	0	22	160	120	3:1	152	156
22	0	0	2	0	22	160	200	3:1	179	179
23	0	0	0	–2	22	160	160	1:1	186	183
24	0	0	0	2	22	160	160	5:1	155	162
25	0	0	0	0	22	160	160	3:1	174	174
26	0	0	0	0	22	160	160	3:1	175	174
27	0	0	0	0	22	160	160	3:1	174	174

X_1 — moisture content (W,%), X_2 — barrel temperature (T, °C), X_3 — screw speed (N, rpm), X_4 — screw compression ratio (K)

Each of the estimated effects and the interactions are shown in the standardized diagram (the Pareto chart — Figure 1). It consists of horizontal blocks with lengths proportional to the absolute values of the estimated effects, divided by their standard errors. The vertical line on the diagram represents the value of the Student criterion at 95% confidence level. From the diagram, it can be seen that the linear effect that is due to the screw speed (factor C) is dominant among the studied factors influencing the expansion. As the screw speed increases, the degree of expansion increases, while the barrel temperature (factor B) and the screw compression ratio (factor D) have negative effect on SEI, i. e. expansion increases with decreasing the temperature and screw compression ratio.

Table 2

Regression coefficients and analysis of variance (ANOVA) for the sectional expansion index of bean-based extrudates

Variables	Coefficients	Degree of freedom	Mean square	*F*-value	*P*-value
Constant	–250.185				
X_1	15.482	1	8.167	0.15	0.7008
X_2	0.233	1	748.167	14.19	0.0027*
X_3	2.040	1	816.667	15.49	0.0020*
X_4	51.417	1	661.5	12.55	0.0041*
X_1X_1	–0.546	1	515.704	9.78	0.0087*
X_1X_2	–0.017	1	16.0	0.30	0.5918
X_1X_3	0.060	1	210.25	3.99	0.0690
X_1X_4	0.583	1	49.0	0.93	0.3541
X_2X_2	0.005	1	71.704	1.36	0.2662
X_2X_3	–0.007	1	110.25	2.09	0.1738
X_2X_4	–0.188	1	225.0	4.27	0.0612
X_3X_3	–0.004	1	59.259	1.12	0.3100
X_3X_4	–0.231	1	342.25	6.49	0.0256*
X_4X_4	–0.417	1	3.704	0.07	0.7955

*Significant at 95% CI.

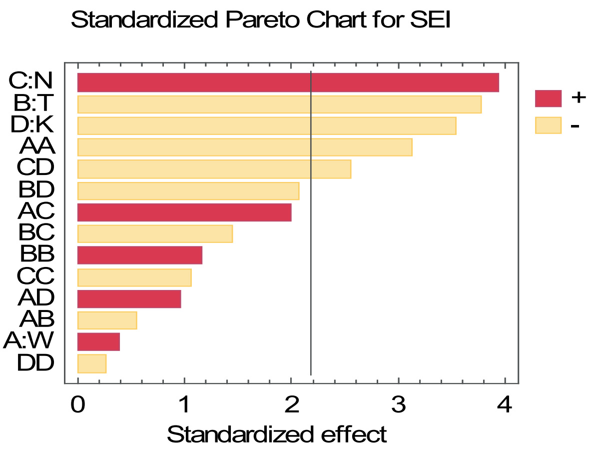


Figure 1. Estimated effects of regression model coefficients on SEI

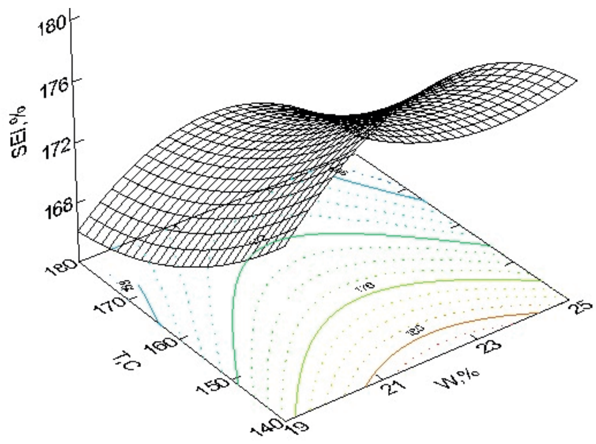


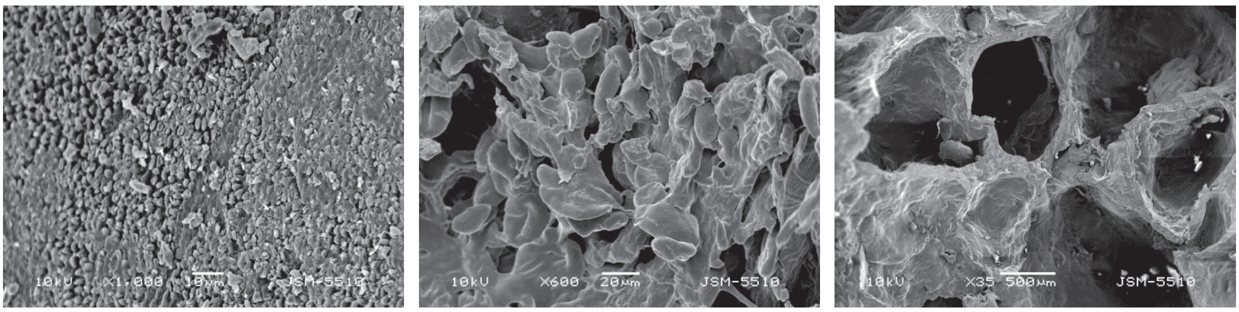
Figure 2. SEI (%) depending on T (°C) и W (%) at n = 160 rpm and K = 3:1

There is a positive correlation between the degree of expansion and the screw speed, which was also been reported by other researchers (Ibanoglu et al., 2006; Meng et al., 2010). It is possible to expect a lower viscosity of the melt in the extruder as the screw speed increases, resulting in extrudates with greater expansion. It increases by 18% when increasing the screw speed from 120 to 200 rpm at moisture content 22%, barrel temperature 160^{o}C, and screw compression ratio 3:1.

Extrusion temperature also significantly affects the expansion of extrudates (Chakraborty et al., 2011; Hagenimana et al., 2006; Meng et al., 2010). As the temperature increases, the degree of expansion decreases (Fig. 1), which is most likely due to an increase in the dextrinization of the starch and changes in the structure of the ingredients of the mixture due to extrusion. It decreases by 18% when the barrel temperature increases from 120 to 200^{o}C at moisture content 22%, screw speed 160^{o}C, and screw compression ratio 3:1. These results correspond to those established by Leonel et al. (2009), Ruiz-Ruiz et al. (2008).

The quadratic effect of the moisture content of the mixture also influence on the degree of expansion of the obtained extrudates, with a negative correlation observed between the two variables. As the moisture content of the material increases, the sectional expansion index increases up to a certain point, after which the increased moisture content leads to a decrease in expansion (Fig. 2).Higher moisture content in the extruded mixture creates a lubricating effect in the extruder cylinder and reduces friction (Pérez et al., 2006). From there, the specific mechanical energy input decreases, resulting in reduced motor torque, lower product temperature, and die pressure (Ryu, 2004). These conditions generate less steam, resulting in less expansion.

The obtained results wereconfirmed by scanning electronmicrographs (SEM). SEM of the semolina and bean-based extrudates are shown in Figure 3.



A *B* *C*

Figure 3. SEM of semolina (A) and bean-based extrudates (B, C)

It is possible to be seen several differences between SEM of the semolina (Fig. 4 a) and bean-based extrudates (Fig. 4 b, c). The dense structure of the raw material is changed significantly. The combined effect of extrusion parameters and chemical content of the starting material may result in the appearance of large void spaces within the extrudates (Fig. 4 c) resulting in increased the degree of expansion of the extrudates. Figure 4B shows gelatinized and partiallygelatinized starch granules entrapped byprotein bodies. In figure 4C it is seen that most starch granules are'disappeared»; they have melted with the proteins and have shaped “blocks” of different sizes.

Conclusion

The average SEI values of the bean-based extrudates varied from 151 to 194%. Statistical analysis showed that all four variables had an effect on SEI. The obtained results were confirmed by scanning electron micrographs (SEM).

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