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Optimization of Extrusion on Blend Flour Composed of Corn, Millet and Soybean

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Abstract: Formulations made of blends of 44% corn flour, 36% millet flour and 20% soybean flour were performed using DPSv 11.50 software. The blends were extruded in Twin screw extruder and orthogonal array $L_9 (3^4)$ was used to evaluate the optimized extrusion conditions. The explanatory variables used were temperature (for the feeding, mixing, cooking and die zones), Rolling speed, feeding speed and moisture content of the samples. The response variables were bulk density, WAI, WSI, pasting properties, thermal analysis, swelling power and the color of the extrudates. The better factors and levels showed that the temperature 80, 110, 140 and 170°C, rolling speed of 110 rpm, feeding speed of 37 g/min and moisture content varies from 25-30% are the best for the extrusion of that formulation.

Key words: Optimization, extrusion, corn, millet and soybean

INTRODUCTION

Cereals are staples food worldwide. To produce nutritious products, cereals are usually fortified with lysine or pulse proteins. Legumes are an important source of food protein and other nutrients (Thakur and Saxena, 2000). Soybeans are widely recognized by medical and health professionals for their health benefits. Soybeans protein has been found to reduce the risk of coronary heart disease when consumed as part of a diet low in saturated fat and cholesterol (Tripathi and Misra, 2005). So blend flour composed of corn, millet and soybean can give a product which has a high-energy value and proteins with high biological value.

Extrusion cooking of cereals is a very important process in food industry, since it regards a wide range of products such as snack-foods, baby-foods, breakfast cereals, noodle, pasta and cereals based blends. Extruders minimize the operating costs and higher productivity than other cooking process, combining energy efficiency and versatility (Ficarella *et al.*, 2004). Guy (2001) found that the ability of extruders to blend diverse ingredients in novel foods can also be exploited in the developing functional foods market such as soybean which is relatively unpalatable alone can be incorporated into new food items.

The purpose of using DPSv11.5 software was to find the formulation of corn, millet and soybean flours that would result in optimized nutritional qualities in protein, fat, fiber and Iron with minimized cost.

The purpose of this study was to optimize the extrusion cooking parameter in order to obtain a product with good

physico-chemical properties. An $L_9 (3^4)$ orthogonal array was selected for experimental layout and nine experiments was carried out.

MATERIALS AND METHODS

Yellow maize, millet and soybeans grains were purchased from the local market, sorted, washed and dried overnight in oven (DHG-9140A, Shanghai Sanfa Scientific Instruments co., LTD., China) at 40°C and milled by using the WK-800 high-speed miller (Shangdong mechanism co., LTD., China) and the flour obtained was passed through a 80 mesh sieve to get a homogenized flour.

Chemical composition of raw materials: Samples of corn, millet and soybean flours were analyzed for protein, fat, fiber, ash, moisture and carbohydrates according to AOAC (1984). Minerals content were analyzed using Atomic Absorption Spectrophotometer and vitamins content by using agilent 1100.

Formulation: The objective was to make a formulation which has a low cost and contain at least the minimum quantity of four nutrients protein 17%, fat 3%, dietary fiber 5% and Iron 3.5 µg.

Variables:

X_1 = Amount of corn in 1 kg of mixture

X_2 = Amount of millet in 1 kg of mixture

X_3 = Amount of soybean in 1 kg of mixture

Where $X_1 > 0$, $X_2 > 0$ and $X_3 > 0$

Constraints

Nutrients constraints:

$$12x_1 + 15x_2 + 45x_3 > 17 \text{ (nutrient A)}$$

$$3.5x_1 + 2.5x_2 + 19x_3 > 3 \text{ (Nutrient B)}$$

$$19.3x_1 + 7x_2 + 20x_3 > 5 \text{ (Nutrient C)}$$

$$2.7x_1 + 3.9x_2 + 4.42x_3 > 3.5 \text{ (nutrient D)}$$

Balancing constraint:

$$X_1 + X_2 + X_3 = 1$$

Objective: Presumably to minimize cost, i.e

$$\text{Minimize } 7x_1 + 12x_2 + 13.2x_3$$

DPS (Data Processing System) v11.50 software was used to find out the formulation to be done, where regression analysis was used at $p < 0.05$.

Extrusion process: The formulation was extruded using PTW 24/25D laboratory co-rotating fully-intermeshing Twin screw extruder (thermo Haake Polylab System Rheomex, Germany) with 4 zones (feeding zone, mixing zone, cooking zone and die zone) and a 5 mm die was used. An orthogonal array $L_9 (3^4)$ experimental design was used to carry out the extrusion (Jurkovic *et al.*, 2006; Wu *et al.*, 2008; Bolboaca and Jantschi, 2007) and the variables used were temperature (attributed to feeding, mixing, cooking and die zones), rolling speed, feeding speed and moisture content of the samples. According to orthogonal test, nine products resulted from the 9 runs. These extrudates were dried in an air dryer oven (DHG-9140A, Shanghai Sanfa Scientific Instruments co., LTD., China) at 105°C for 15 min, milled. The Flour obtained was passed through 80 mesh sieve to get homogenized extruded flour and stored into plastic bags at room temperature for further analysis.

Chemical composition of the extrudates: Protein, fat, fiber, ash, moisture and carbohydrates of the ground extrudates were analyzed according to the official methods of AOAC (1984). Mineral contents were analyzed using Atomic Absorption Spectrophotometer and Vitamins B1, B2, B6, B12, Folic acid; Niacin, Vitamin E and A were determined using Agilent 1100 Liquid chromatography. Energy conversion factors were used in calculating the calorific value of the nutrients:

Color: The color of the grounded extruded products was determined by using the color meter (DATA PROCESSOR DP-400 for chroma meter, KONICA MINOLTA SENSING, INC) where values in lightness (L^*), redness (a^*) and yellowness (b^*) were recorded. Three measurements were taken for each sample and their means reported.

Bulk density: The bulk density of the extrudates was determined using a 25 ml graduated cylinder by packing gently and tapping on the bench. The volume of the flour was recorded and the weight of the flour was weighed using an electronic analytical balance (FA1104, Shanghai Balance Instrument Factory, China). The bulk density was calculated as the weight of flour divided by the volume and it was recorded as grams per cubic centimeter (g/cm^3). Three measurements were performed for each sample.

Water Absorption Index (WAI) and Water Solubility Index (WSI): Water Absorption Index (WAI) and Water Solubility Index (WSI) were measured with a slight modification of the method used by Anderson *et al.* (1969). 1 g of the ground extrudates was suspended in 20 ml of distilled water in a tared 45 ml centrifuge tube. The slurry was shaken with a glass rod and put in water bath at 30°C for 30 min then centrifuge (Himac CR21GII, High-speed refrigerated centrifuge, Hitachi koki co., LTD, Japan) at 3000 rpm for 15 min. The supernatant was decanted into an evaporator dish of known weight. The WAI was calculated from the weight of the remaining gel and expressed as grams of gel per grams of solid. The WSI expressed as gram of solids per gram of original solids, was calculated from the weight of dry solids recovered by evaporating the supernatant overnight at 105°C.

$$\text{WAI} = \text{Weight of sediment} / \text{weight of dry solids}$$

$$\text{WSI} = (\text{wt of dissolved solids in supernatant} / \text{wt of dry sample solids in the original sample}) * 100.$$

Pasting properties: The pasting properties of extrudates starches were determined using Rapid Visco Analyser (RVA tec master, Newport scientific Pvt., LTD., Australia) according to the method reported by Zaidul *et al.* (2007) and Kim *et al.* (2005) and Fred *et al.* (2003) with some modifications. 4 g of extrudates flour was added to 25 ml of distilled water. The heating and cooling cycles were programmed in the following manner. The sample was held at 50°C for 1 min, heated to 95°C in 3.42 min, held at 95°C for 2.7 min, cooled to 50°C in 3.88 min and finally held at 50°C for 2 min. The total time for analysis was 13 min.

Swelling power: The swelling power (by weight) of starch was measured using a modified method from the one reported by Tester and Marrison (1994). 0.5 g of extrudate was dispersed in 15 ml of distilled water. The suspension was heated at 90°C in a water bath for 30 min with vigorous shaking very 5 min. The starch gel was then centrifuged at 3000 rpm for 15 min. The weight of sediment was used to calculate the swelling power. Swelling power was calculated as follow:

$$\text{Swelling power} = \frac{\text{Mass of swollen sample (g)}}{\text{Initial mass (g)}}$$

Thermal analysis: Thermal analysis of extruded products proteins was done according to the method reported by Leblanc *et al.* (2008) and Zaidul *et al.* (2007) with some modifications. The thermal behavior of the proteins from extrudate was examined with a Perkin Elmer Model PYRIS 1- DSC Differential Scanning Calorimeter. 10 mg of extrudates was weighed in silver pan and an empty pan was used as blank. Measurements were carried out under Nitrogen ambience (100/ml) with a heating rate of 10°C/min by a scanning temperature range from 20-100°C. The gelatinization on set temperature (T₀), peak temperature (T_p) and enthalpy (delta H) were recorded.

Statistical analysis: The determination of color attributes (L*, a* and b*) was carried out in triplicate and the values were averaged. Data was assessed by the Analysis of Variance (ANOVA) (Snedecor and Cochran, 1987). Duncan Multiple Range Test was used to separate means. Significance was accepted at p<0.05.

RESULTS AND DISCUSSION

Formulation: The results obtained after using DPS V11.50 are shown in Table 3 and 4. 1 kg of the formulation should contain: 440 g of corn, 360 g of millet, 200 g of soybean and it will cost 10RMB.

Equation:

$$Y = 11.86069983 - 4.854160070 \times x_1 + 15.632241114 \times x_3 + 6.956906615 \times x_3^2 + 7.050481863 \times x_1 \times x_3 + 7.167660759 \times x_2^2 \times x_3$$

Chemical composition: The proximate nutrient composition of the nine extruded products is presented in Table 7. The results indicated that the minimum quantity of nutrients based on which the formulation was done in Table 2 were achieved for dietary fiber and fat (for the 2nd, 6th, 8th and 9th products). Therefore, the nutrients obtained meet the requirements for FAO/WHO standards (1991) and FAO/WHO (1971).

Bulk density: The bulk density of extrudates is important in relation to their ability to float or sink when poured into water and their packaging requirement. The bulk density

Table 1: Chemical composition of raw materials

Raw materials	Corn	Millet	Soybean
Protein	12	15	45
Fat	3.5	2.5	19
Ash	2.8	0.81	4.77
Moisture	9.7	4.39	5.2
Fiber	19.3	7	20
Carbohydrates	48.9	70.3	6.03
Zn (µg/ml)	0.965	1.14	1.21
Fe (µg/ml)	2.7	3.9	4.42
Na (µg/ml)	2.80	1.15	5.50
K (µg/ml)	136	110	553
Mg (µg/ml)	120	35.5	140
Ca (µg/ml)	135	10.7	178
P (µg/ml)	0.728	0.022	0.72
Vit A (µg/ml)	2.1	9.575	11.3
Vit E (µg/ml)	444.6	58.484	-
Vit B1(µg/ml)	1.6	0.770	6.6
Vit B2 (µg/ml)	8.7	9.258	5.6
Niacin (µg/ml)	5.7	0.976	7.3
Folic acid (µg/ml)	10	0.568	3
Vit B6 (µg/ml)	7.8	140.757	35.9
Vit B12 (µg/ml)	11.6	6.131	3.2

Table 2: Nutrients values and cost of the raw materials

Raw materials	Protein (A)	Fat (B)	Dietary fiber (C)	Fe (D)	Cost/ 500 g
Corn (g/100 g)	12%	3.5%	19.3%	2.7 µg	7 RMB
Millet	15%	2.5%	7%	3.9 µg	12 RMB
Soybean	45%	19%	20%	4.42 µg	13.2 RMB

Table 3: Formulation made

Raw materials	Corn	Millet	Soybean
g	440	360	200
%	44	36	20
Cost	10 RMB/1 kg		

Table 4: Each factor combination at lowest index

Y	x1	x2	x3
10	0.4400	0.3600	0.2000

of extruded products varies from 0.8974 up to 1.0782 (see Table 8). There is an increase in bulk density as the temperature increases; this is due to the liquefaction of sugar via melting during extrusion process (Meuser and Wiedmann, 1989). As it is shown in Table 9, the optimized factor levels for density were in the combination A₃D₂B₂C₂ - which means [T: 80, 110, 140 and 170°C, Feeding speed of 3 (37 g/min), Rolling speed 130 rpm and Moisture content varies from 20-25%]. These extrusion conditions were not done but they are slight similar to the last three experiments we did (7th, 8th and 9th experiments/runs). These conditions can also be suggested for the bulk density.

Table 5: Variables and their levels under extrusion process

Symbol	Variables	Levels		
		1	2	3
A	Temperature (T)	40, 70, 100, 130°C	60, 90, 120, 150°C	80, 110, 140, 170°C
B	Rolling Speed (RS)	110	130	150
C	Moisture Content (M.C)	15-20%	20-25%	25-30%
D	Feeding Speed (FS)	2 (20 g/min)	3 (37 g/min)	4 (55.4/min)

Table 6: Experimental layout using an L₉ (3⁴) orthogonal array

Experiment number	Variables and levels			
	A	B	C	D
1	1	1	1	1
2	1	2	2	2
3	1	3	3	3
4	2	1	2	3
5	2	2	3	1
6	2	3	1	2
7	3	1	3	2
8	3	2	1	3
9	3	3	2	1

Water Absorption Index (WAI) and Water Solubility Index (WSI): Water absorption index of the extrudate varied from 3.63 and 6.04 g/g and the products with high WAI were those with the highest moisture content before extrusion (25-30%). As shown on Table 10, the best factors' combination for WAI is C₃A₃B₁D₂ which means MC: 25-30%, T: 80-170°C, RS: 130 rpm and FS: 3 (37 g/min). These extrusion conditions were fulfilled in the 7th treatment.

In general, the WSI increased as the temperature increased and it varies from 9.1-16.95%. This may be due the inactivation of antiphenological factors contained in the soybean or may be due to the damage of starch content at high temperature. The best factors and combination for WSI was the treatment C₁A₁D₁B₂ (Table 11); this means Moisture content varies from 15-20%, temperature of 40, 70, 100 and 130°C, Feeding speed 2 (20 g/min) and Rolling speed of 130 rpm. These extrusion conditions were not done but they are almost similar to the 1st experiment (A₁B₁C₁D₁), the only difference is the rolling speed which is 110 rpm. So these conditions are also recommended for WSI.

Color: Color is an important characteristic of extruded foods. Color changes can give information about the

extent of browning reactions such as caramelization, maillard reaction, degree of cooking and pigment degradation during the extrusion process (Ilo and Berghofer, 1999). The lightness (L*) is an indication of the brightness. The lightness value of the products ranges from 79.70-84.73. The Table 12 shows that there are no significant different between the 2nd, 3rd, 4th, 6th and 8th products and there are significant difference between them and others products for (p<0.005). The color parameter a*, indicative of the redness of sample varied from 1.62-4.84 and there is a significant difference between all the products for p<0.05 and in general, the products cooked with high temperature have the highest values. Dark color is also developed during caramelization of sugar from the maillard reaction that why redness (a*) value is high as the temperature increases. The yellowness value (b*) of extruded products varies from 21.87-26.07. There was a significant different between the yellowness value of almost all the product for p<0.005. The change in yellowness during extrusion cooking was most induced by the effects of nonenzymatic browning and pigment destruction reactions. All these differences could have been due to the shear forces generated during extrusion which accelerated the chemical reactions between amino acids and reducing sugars (maillards reaction) that take place during extrusion (Guy, 2001) and to the different temperature cooking, rolling speed and feeding speed conditions during extrusion. The combination A₃B₁C₃D₂ (Moisture content varies from 25-30%, Temperature 80, 110, 140 and 170°C, Rolling speed of 130 rpm and Feeding speed 37 g/min) yielded the lowest lightness value (L* = 79.70), the highest redness value (a* = 4.84) and the 2nd highest value for brightness (b* = 25.04).

Table 7: Chemical composition of extrudates

	1	2	3	4	5	6	7	8	9
Protein %	15.22	15.35	15.13	15.33	15.46	15.92	15.89	16.43	15.88
Fat %	2.78	3.72	2.32	2.8	2.7	3.6	2.64	4.58	3.16
Moisture %	10.57	10.49	10.37	10.40	9.13	7.15	8.63	6.06	6.67
Ash %	1.72	1.80	1.84	1.76	1.97	2.04	1.86	1.84	1.94
Fiber %	13.8	14.3	12.7	12	13.6	14.9	15.4	13.6	14.5
*Carbohydrates %	55.91	54.34	57.67	58.34	57.14	56.39	55.58	57.49	57.85
Zn (µg/ml)	1.03	0.922	1.11	0.873	0.989	0.949	0.977	0.950	0.974
Fe (µg/ml)	1.20	1.64	1.70	1.80	1.94	1.47	1.69	1.81	1.60
Na (µg/ml)	2.55	2.20	2.02	1.79	1.99	1.67	1.95	1.63	2.65
K (µg/ml)	278	285	284	285	282	302	305	307	296
Ca (µg/ml)	30.1	32.6	31.1	28.2	30.8	32.7	31.6	33.5	33.9
Mg (µg/ml)	38.1	46.1	45.5	40.9	44.1	48.1	46.8	50.5	46.1
P (µg/ml)	0.033	0.032	0.031	0.034	0.032	0.031	0.032	0.034	0.030
Vit A (µg/ml)	1.63	2.26	1.33	1.27	4.70	1.30	2.54	2.59	1.22
Vit E (µg/ml)	100.78	83.29	37.79	33.96	70.15	55.65	65.99	70.92	44.19
Vit B1 (µg/ml)	0.68	0.26	0.31	7.35	4.25	0.70	1.17	1.95	1.32
Vit B2 (µg/ml)	11.09	11.15	18.16	11.32	0.16	10.55	11.22	10.04	10.63
Niacin (µg/ml)	4.86	0.35	5.40	3.32	3.30	3.95	3.97	4.86	4.28
Folic acid (µg/ml)	2.63	1.49	2.31	1.18	1.10	1.27	1.56	3.26	1.39
Vit B6 (µg/ml)	226.82	172.68	247.64	168.16	150.61	192.60	208.46	210.31	196.10
Vit B12 (µg/ml)	6.35	6.71	11.46	6.59	6.44	5.85	8.80	5.68	6.27

*Carbohydrates by difference

Table 8: results for orthogonal test

Experiment number	Density	WAI	WSI	Swelling power
1	0.9191	3.63	16.95	5.80
2	0.9810	3.95	14.1	5.77
3	0.8974	4.78	11.45	5.53
4	1.0146	4.53	13.25	6.51
5	1.0296	5.16	12.15	5.83
6	1.0598	4.03	15.3	5.42
7	1.0564	6.04	9.1	13.79
8	1.0782	4.52	13.35	5.87
9	1.0681	4.65	12.25	9.09

Table 9: Bulk density results

	A	B	C	D
K ₁	0.9325	0.9967	1.0190	1.0056
K ₂	1.0347	1.0296	1.0212	1.0324
K ₃	1.0676	1.0084	0.9945	0.9967
R-value	0.1351	0.0329	0.0267	0.0357

Where A is temperature variable, B is rolling speed, C is moisture content and D is feeding speed.

K₁ is means of combination of all 1 in the same column

K₂ is means of combination of all 2 in the same column

K₃ is means of combination of all 3 in the same column and

R-value is the highest K minus the lowest K in the same column (K_{maximum}-K_{minimum})

Table 10: WAI results

	A	B	C	D
K ₁	4.12	4.73	4.06	4.48
K ₂	4.57	4.54	4.38	4.67
K ₃	5.07	4.49	5.33	4.61
R-value	0.95	0.24	1.27	0.19

Table 11: WSI results

	A	B	C	D
K ₁	14.17	13.1	15.2	13.78
K ₂	13.57	13.2	13.2	12.83
K ₃	11.57	13	10.9	12.68
R-value	2.6	0.2	4.3	1.1

Table 12: Color attributes results

	1	2	3
L*	80.33C	84.63A	84.10A
a*	2.4E	1.67F	1.62F
b*	26.07A	23.26E	24.48BC
	4	5	6
L*	84.68A	82.27B	84.73A
a*	1.69F	3.39C	2.20E
b*	23.19E	23.87D	22.46F
	7	8	9
L*	79.70C	84.04A	80.62C
a*	4.84A	2.68D	4.60B
b*	25.04B	21.87G	23.93CD

Swelling power: Swelling power of the extrudates varied from 5.42-13.79. The Table 13 showed that the best combination in factors levels for a good swelling power is A₃B₁C₃D₂ (Moisture content: 25-30%, Temperature: 80, 110, 140 and 170°C, Rolling speed: 130 rpm and Feeding speed: 37 g/min). In general, the products cooked with the highest swelling power, these may be due to starch gelatinization and degradation during extrusion process.

Table 13: Orthogonal results for swelling power

	A	B	C	D
K ₁	5.7	8.7	5.70	6.91
K ₂	5.92	5.82	7.12	8.33
K ₃	9.58	6.68	8.38	5.97
R-value	3.88	2.88	2.68	2.36

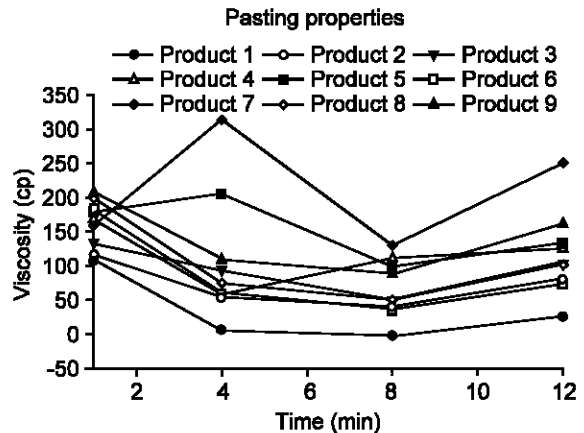


Fig. 1: Pasting properties

Pasting properties: The results in pasting properties of extrudates are shown in Table 13 and the Fig. 1. As it is shown on this figure, the viscosity increases as the temperature increases for the products cooked with a high temperature (from 80-170°C). These may be attributed to the starch gelatinization during extrusion and the Table 13 shows that the product cooked with these condition A₃B₁C₃D₂ has the highest peak, trough, breakdown, final viscosity and setback 46.17, 10.58, 35.58, 22.83 and 12.25 cp respectively.

Thermal analysis: Thermal properties of the extrudates starch are shown in Table 15. T₀ of extrudates were ranged from 43.99-96.17°C and the high temperature was found with starches derived from the 7th product (MC: 25-30%, T: 80-170°C, RS: 130 rpm and FS: 37 g/min) while the lowest was observed from the 2nd product (temperature 40, 70, 100 and 130°C, rolling speed: 110 rpm, feeding speed: 37 g/min and moisture content: 20-25%). Gelatinized extrudates starches were ranged from 51.27-99.17°C for T_p and 58.09-99.92 for T_e. In general, it has been found that the products cooked at low temperature had a low T₀, T_p and T_e while those cooked at high temperature had a high T₀, T_p and T_e and the 7th product has the highest values (MC: 25-30%, T: 80-170°C, RS: 130 rpm and FS: 37 g/min). These may be due to the increase of temperature, shear and pressure during extrusion which increased the rate of gelatinization. Guy (2001) and O'Connor (1987) reported that the complete gelatinization of starch is necessary in human nutrition for a good digestibility and this can be done by using a high temperature in cooking starches.

Table 14: Pasting characteristics of extrudates

	Peak viscosity	Trough	Breakdown	Final viscosity	Set back	Peak time	Pasting temperature
1	1.67	-0.33	2	2.25	2.58	1.53	46.45
2	7.83	3.17	4.67	6.75	3.58	1.6	46.6
3	13.83	4.17	9.67	9	4.83	1.07	46.85
4	14.58	3.08	11.5	10.5	7.42	1.4	46.9
5	24.5	6.5	20.08	11.5	5	2.53	47.4
6	16.5	2.75	13.75	6.33	3.58	1.4	47.3
7	46.17	10.58	35.58	22.83	12.25	1.67	47
8	16.17	3.83	12.33	8.67	4.83	1.93	46.95
9	23.17	5.75	17.33	14.08	8.33	2.2	46.75

Table 15: Results of thermal analysis

	T ₀	T _p	T _e	Delta H	Peak height	Area
1	45.50	52.52	59.88	0.9184	0.1849	6.796
2	43.99	51.27	58.09	0.7600	0.1754	6.300
3	45.70	53.27	60.83	1.0508	0.2505	9.194
4	91.08	95.66	96.16	0.2825	0.1416	1.661
5	47.39	54.04	59.52	0.1584	0.0321	0.968
6	86.67	87.15	87.15	2.0006 e-4	0.0019	0.003
7	96.17	99.17	99.92	0.0287	0.0225	0.207
8	86.64	87.39	87.64	9.5095 e-4	0.0086	0.008
9	90.15	90.90	90.90	8.8323e-4	0.0029	0.008

Where t₀: temperature onset, T_p: peak temperature, T_e: end temperature

Conclusion: According to the physico-chemicals results of the extruded products; It has been found that the product cooked with these conditions (Moisture content: 25-30%, Temperature: 80, 110, 140 and 170°C, Rolling speed: 130 rpm and Feeding speed: 37 g/min) are the best. So extrusion of the formulation done should be cooked in these conditions.

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