Original Article

Process Optimization of Finger Millet Incorporated Extrudates

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Abstract

The effects of incorporation of finger millet (*Eleusine coracana L.*) flour and extrusion conditions on physico-chemical characteristics of corn grit- rice grit-chickpea flour blend expanded extrudates were studied. Response surface methodology was used to study the effects of level of incorporation of millet flour in feed composition (5 to 25 %), feed moisture (12-16%), screw speed (1000-1400 rpm) and extrusion temperature (80–120°C). Single screw extruder was used for the experiments. The level of millet flour incorporation had significant effect on water solubility index ($p<0.05$), water absorption index ($p<0.05$) and starch digestibility ($p<0.001$). Feed moisture had significant effect on water solubility index ($p<0.001$) and water absorption index ($p<0.05$). Extrusion temperature had significant effect on water solubility index ($p<0.001$), water absorption index ($p<0.05$) and starch digestibility ($p<0.05$). Screw speed had significant effect on water solubility index ($p<0.001$) and water absorption index ($p<0.05$). Numerical optimization study predicted 106.8 °C of extrusion temperature, 1253 rpm of screw speed, 12.67 % of feed moisture, and 22.8 % of millet flour as optimum conditions to produce acceptable extrudates from the feed composite containing millet flour.

1. Introduction

Extrusion cooking is a specialized form of processing which involves heating to high temperatures in short time with the application of mechanical mixing and shearing, before finally extruding to form a structure (Guy, 2001). Cereals are the main raw material for extruded snack foods. Millets (*Eleusine coracana L.*) are predominantly starchy. The revitalized nutritional strengths of millets have made them functional grains. Snacks and pasta developed by extrusion process from the millet are found to be highly acceptable and hypo-glycemic (Rao et al., 2016). Chickpea flour has also been widely used in extrudates. However, contradictory effects have been reported regarding the properties of extrudates with chickpea flour. Shirani and Ganesharanee (2009) found that the addition of chickpea flour on extrudates significantly reduced lateral expansion of extrudate. In contrast, Meng et al. (2010) found positive correlation between the amount of chickpea flour and lateral expansion in a work related to multiple blends of chickpea flour,

potato starch, protein concentrate, and other additives. Anderson (1969) found that water absorption index (WAI) of extruded maize flour increased progressively with increased extrusion temperature and increased feed moisture. Ilo et al. (1999) reported that the degree of gelatinization of extruded maize grits decreased with increasing feed moisture, and increased with increasing feed rate and product temperature. Artz et al. (1990) reported that there was decrease of WAI in extrusion of corn fiber and corn starch blend. For rice based extrudates by (Ding et al., 2005) and for maize, finger millet extrudates by (Onyango et al, 2004) found that water solubility index (WSI) value decreased with increasing feed moisture content. Increase in cassava flour level was found to decrease WAI and WSI (KC et al., 2015). The increase in WSI with increasing screw speed was consistent with the results reported for corn meal and corn and wheat extrudates (Jin et al., 1995; Mezreb et al., 2003). The effect of extrusion conditions on WAI had been discussed by (Colonna and Mercier, 1983; (Gomez and Aguilera, 1983);

Pelembe et al., 2002; Ding et al., 2006; and many other researchers. Guha et al. (1998) studied structural change in starch chain due to extrusion temperature. Similarly, Fletcher et al. (1985), Wen et al. (1990), Camire and King (1991), Gutkoski and El-Dash (1999) and other many researchers studied the effect on WSI. The effect of millet flour on major commercial extrusion ingredients such as corn grits, rice grits and chickpea flour has not been fully assessed yet. This research is focused on evaluating effects of millet incorporation in properties of extrudates based on rice grits, corn grits and chickpea flour and optimizing the process parameters.

2. Materials and Methods

2.1 Ingredients selection and preparation

Corn grit, rice grit and chick-pea flour (Cg-Rg-CpF) blend was used for extrusion while finger millet (kodo) flour (MF) at different proportion (Table 1) was incorporated by replacing corn grit. Millet that was stored six months after harvest was purchased from Pancha-kanya, Sunsari, Nepal and sun dried. Moisture content of millet was determined frequently unless it was reduced to 6%. Millet was milled and its flour was packaged in high density polyethylene (HDPE) bag of 30 μm and sealed. Other ingredients were supplied from CG foods (Nepal) Pvt. Ltd. Nawalparasi and packaged in HDPE.

2.2 Experimental design

Response surface methodology (RSM) was used for the experimental design using a five-level, four-factor Central Composite Rotatable Design (Myers et al., 2016). RSM can be used for the modeling and analysis of the problem in which response of the interest is influenced by several variables. The independent variables selected for the experiments were: Feed proportion, moisture content, screw speed, and extrusion temperature. Response variables were water absorption index, Water solubility index and maltose content. For numerical multi-response optimization, results of response variables; bulk density (0.23-0.46 g/cm3) and lateral expansion (91.11-144%) were used from the similar research of millet incorporated extrusion (KC et al., 2013).

2.3 Extrusion of composite blends

Ingredient formulations and experimental combinations for extrusion products are given in Table 1. In the blend preparation millet flour at levels of 5%, 10%, 15%, 20% and 25% were used. The moisture was adjusted by sprinkling distilled water in all the dry ingredients. All the ingredients were weighed and then mixed with the help of small scale mixture (Flightier series 003, Teflon, India) for 20 min. This mixture was then passed through a 2 mm sieve to reduce the lumps formed due to addition of moisture. After mixing samples were stored in polyethylene bags (HDPE, 30μm) and were kept in high precision (+ 0.1 °C) incubator (Macro Scientific works, New Delhi) at 45 °C for 12 h duration for the stabilization of moisture. Moisture content was measured by I-R moisture meter (MB35 Halogen OHAUS) of CG foods (Nepal), Nawalparasi. The single screw extruder was kept running for 30 min to stabilize the set temperatures and feed of 1 kg with different proportions of corn grit and millet flour (Table 1) were then poured into feed hopper . The feed rate of extruder was adjusted to 4 kg/h. The die diameter of 3 mm was selected as recommended by the manufacturer. The product collected at the die end and moisture content of the products was determined frequently unless it was reduced to 6%. Then, it was packed air-tight in polythene (HDPE, 30μm) for analysis.

2.4 Evaluation of extrudates characteristics

Water absorption Index (WAI) and Water Solubility Index (WSI) were determined according to the method developed for cereals (Anderson, 1969). The ground extrudate was suspended in water at room temperature for 30 min, gently stirred during this period, and then centrifuged at 3000 rpm for 15 min. The supernatant was decanted into an evaporating dish of known weight. The WAI was the weight of gel obtained after removal of the supernatant per unit weight of original dry solids. The WSI was the weight of dry solids in the supernatant expressed as a percentage of the original weight of sample. Reducing sugar (maltose content) was estimated by DNS method (Garriga et al., 2017) for starch digestibility of the extrudate. One mL standard maltose solution was pipetted out in different test tubes and make up the volume of all test tubes to 3 mL with distilled water. One mL DNS reagent was added to all the test tubes and the tubes were taken to a bath thermostatized at 100 °C for 5 min. Final volume was made 10 mL and homogenized. After cooling to room temperature, UV absorbance was read at 540 nm against the blank.

$$WAI = \frac{Weight\ gain\ by\ gel}{Dry\ weight\ of\ extrudate}$$

$$WSI\ (\%) = \frac{Weight\ of\ dry\ solid\ in\ Supernant}{Dry\ weight\ of\ extudate} \times 100$$

Table 1: Experimental combinations in Coded and Uncoded levels for millet flour with rice grit, corn grit and chick-pea flour based extrudates

S. No.	Coded variables				Feed Proportion (%)	Uncoded variables		
	A	B	C	D		Feed Moisture (%)	Temperature (°C)	Screw speed (rpm)
1	-1	-1	-1	-1	58:32:10	13	90	1100
2	1	-1	-1	-1	58:22:20	13	90	1100
3	-1	1	-1	-1	58:32:10	15	90	1100
4	1	1	-1	-1	58:22:20	15	90	1100
5	-1	-1	1	-1	58:32:10	13	110	1100
6	1	-1	1	-1	58:22:20	13	110	1100
7	-1	1	1	-1	58:32:10	15	110	1100
8	1	1	1	-1	58:22:20	15	110	1100
9	-1	-1	-1	1	58:32:10	13	90	1300
10	1	-1	-1	1	58:22:20	13	90	1300
11	-1	1	-1	1	58:32:10	15	90	1300
12	1	1	-1	1	58:22:20	15	90	1300
13	-1	-1	1	1	58:32:10	13	110	1300
14	1	-1	1	1	58:22:20	13	110	1300
15	-1	1	1	1	58:32:10	15	110	1300
16	1	1	1	1	58:22:20	15	110	1300
17	-2	0	0	0	58:37:05	14	100	1200
18	2	0	0	0	58:17:25	14	100	1200
19	0	-2	0	0	58:27:15	12	100	1200
20	0	2	0	0	58:27:15	16	100	1200
21	0	0	-2	0	58:27:15	14	80	1200
22	0	0	2	0	58:27:15	14	120	1200
23	0	0	0	-2	58:27:15	14	100	1000
24	0	0	0	2	58:27:15	14	100	1400
25	0	0	0	0	58:27:15	14	100	1200
26	0	0	0	0	58:27:15	14	100	1200
27	0	0	0	0	58:27:15	14	100	1200
28	0	0	0	0	58:27:15	14	100	1200
29	0	0	0	0	58:27:15	14	100	1200
30	0	0	0	0	58:27:15	14	100	1200

Note: Feed composition [(50% rice grit + 8% chick-pea flour): corn grit: millet flour), Where X= (50% rice grit + 8% chick-pea flour) was kept constant.

2.5 Data analysis

The responses (WAI, WSI and starch digestibility) for different experimental combinations were related to the coded variables by a second degree polynomial equation:

$$Y=\beta_0+\beta_1 A+\beta_2 B+\beta_3 C+\beta_4 D+\beta_{11} A^2 +\beta_{22} B^2 +\beta_{33} C^2 +\beta_{44} D^2 +\beta_{12} AB+\beta_{13} AC+\beta_{14} AD+\beta_{23} BC+\beta_{24} BD+\beta_{34} CD+\varepsilon$$

Where A, B, C and D are the coded values of independent variables namely feed composition (% millet flour), feed moisture content (%), extrusion temperature (°C) and screw speed (rpm), respectively. The coefficients of the polynomial were represented by β_0 (intercept); β_1, β_2, β_3, β_4 (coefficient of linear effects), β_{12}, β_{13}, β_{14}, β_{23}, β_{24}, β_{34} (coefficient of interaction effects); β_{11}, β_{22}, β_{33}, β_{44} (coefficient of quadratic effects); and ε (random error). Design Expert software (STAT-EASE Inc., USA, version 6.0) was used to analyze data and optimization.

2.6 Optimization study

A numerical multi-response optimization technique was applied to determine the optimum combination of millet flour in feed composition, feed moisture, screw

speed and extrusion temperature for the development of extrudate containing corn grit, rice grit, chick-pea flour and millet flour. The assumptions were to develop a product which would have maximum expansion, minimum bulk density, maximum WAI, WSI content in range and maltose content in range. Responses were maintained as mentioned whereas independent variables were kept within range.

3. Results and Discussion

3.1 Effects of process variables on WAI

The WAI ranged from 5.40 to 6.99 g/g. Table 2 shows the coefficients of the model and statistical attributes of WAI. All the linear terms except moisture content had negative effect on WAI. The decrease in millet flour proportion will increase the WAI of the product. This might be attributing to relative decrease in fiber content with addition of millet flour and competition of absorption of water between millet flour and available starch. Similar findings were reported by (Artz et al., 1990) in extrusion of corn fiber and corn starch blend. At highest temperature the WAI value was found to be increased with increase in feed moisture (Fig 1). This might be due to higher moisture content, acting as a plasticizer during extrusion cooking, reduces the degradation of starch granules and this result in an increased capacity for water absorption. This decrease in WAI values with an increase in temperature might be due to an increase in starch decomposition degradation (Colonna and Mercier, 1983). It was stated that WAI decreases with increasing temperature if dextrinization or starch melting prevails over the gelatinization phenomenon (Ding et al., 2006; Pelembe et al., 2002). Increase in screw speed will decrease WAI. The reduction of WAI by increasing screw speed might be due to starch degradation. At a low shear rate (low screw speed) and/or low temperature, there might be less damaged polymer chains and a greater availability of hydrophilic groups which can bind more water resulting in higher values of WAI (Gomez and Aguilera, 1983). WAI was affect by the extrusion temperature and screw speed. The higher water absorption index was observed at high extrusion temperature and low screw speed conditions from (Fig 2). This might be due to high residence time at low screw speed permitting enhanced extent of cooking. Only gelatinized starch granules absorb water at room temperature and swell; however, starch fragmentation increases when the gelatinization degree increases thus decreasing water absorption.
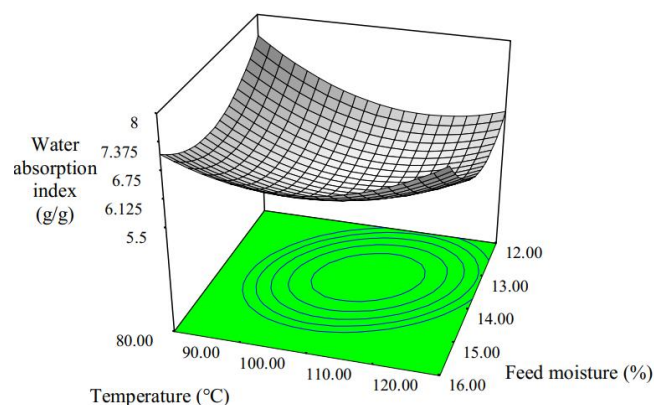

Fig. 1: Response surface plot for water absorption index as a function of feed moisture and extrusion temperature at center value of feed composition and screw speed.
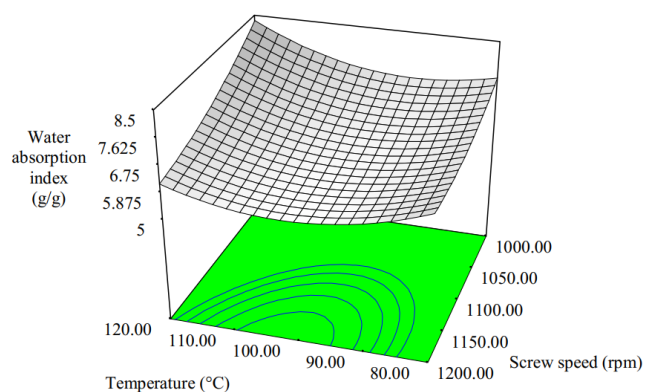

Fig. 2: Response surface plot for water absorption index as a function of screw speed and extrusion temperature at center value of feed moisture and feed composition

3.2 Effects of process variables on WSI

The WSI values ranged from 4.72% to 9.705%. Table 2 shows the coefficients of the model and statistical attributes of WSI. All the linear terms except screw speed had negative effect on WSI. At higher level of millet flour in the feed blend, the WSI is found to be decreased with increasing moisture content (Fig 3). This decrease with the increase in moisture content may be attributed to reduction in lateral expansion due to plasticization of melt as observed by (Ding et al., 2005). WSI was increased as millet content decreases. The feed blend was the mixture of corn grit, rice grit, chick-pea flour and millet flour. Proportion of rice grit and chick-pea flour was kept constant and hence there was increase in corn grit which contain high amount of fiber which may bind water more strongly than starch during extrusion. This water binding capacity inhibits water loss at the die, that is, at the exit of the extruder, reducing expansion (Camire and King, 1991); thus the starch present may cause degradation increasing WSI. WSI was found to be decreased with increasing temperature and decreasing screw speed (Fig 4). WSI depends on mainly on amylopectin content (quantity of

soluble matter) which increases due to the degradation of starch. The high mechanical shear stress degraded macromolecules, the molecular weight of starch granules decreased and consequently, the WSI increased because degraded starch granules are more soluble in water (Fletcher et al., 1985). The increase in WSI with increasing screw speed was consistent with the results reported for corn meal and corn and wheat extrudates (Jin et al., 1995; Mezreb et al., 2003). Mezreb et al., (2003) reported that the increase of screw speed induced a sharp increase of specific mechanical energy, the high mechanical shear degraded macromolecules, and so the molecular weight of starch granules decreased and hence increased WSI. Wen et al., (1990) indicated that screw speed had a direct effect on size distribution of polysaccharide. A higher screw speed (increasing the shear) resulted in more fragmentation than a lower screw speed. WSI decreased significantly with increasing extrusion temperature. Gutkoski and El-Dash (1999) observed similar behavior in extruded oat products.
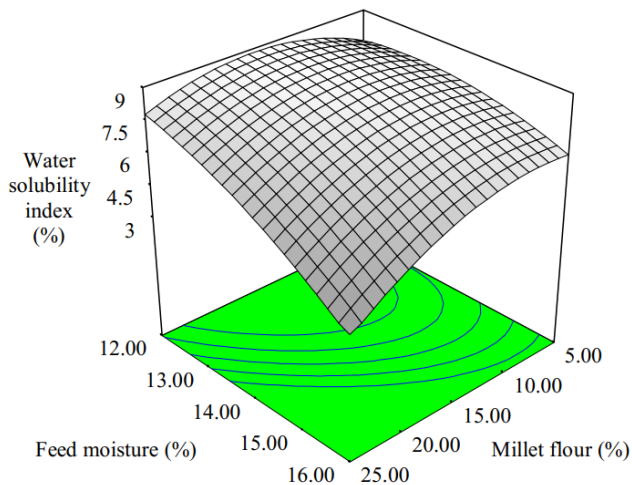

Fig. 3: Response surface plots for the variation of water solubility index as function of feed composition and feed moisture at central level of extrusion temperature and screw speed.
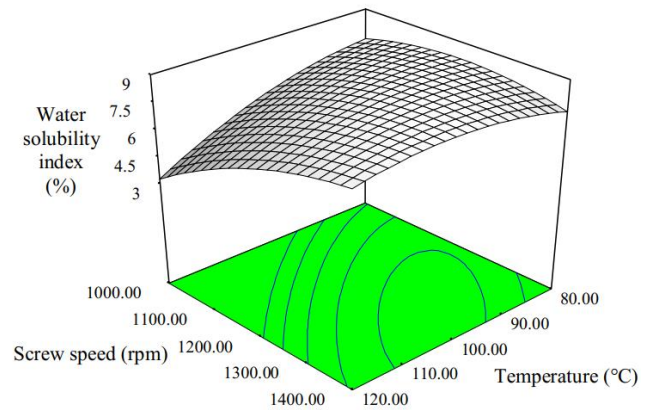

Fig. 4: Response surface plots for the variation of water solubility index as function of screw speed and extrusion temperature at central level of feed composition and feed moisture

3.3 Effects of process variables on product starch digestibility (SD)

Extrusion cooking is one processing method that has been used to modify the digestible characteristics of starchy materials. The amount of starch digestion was determined by comparing the absorbance of each sample to a standard Reducing sugar (maltose). Maltose content of an extruded product directly reflects the degree of gelatinization during extrusion. Maltose content of extrudate varied from 256.5 to 314.5 µg. Table 2 shows the coefficients of the model and statistical attributes of Maltose content. Among the linear terms, feed composition and extrusion temperature had significant positive effect on Maltose content. One factor plot for feed composition is shown in (Fig. 5). Maltose content of extrudates was found to increase with increased feed composition. Increase in millet flour increases the availability of starch. Maltose content is directly related with amount of starch present in raw material. Positive effect of feed composition indicates that the highest millet flour causes high gelatinized extrudate. This result is supported by trends in expansion ration with respect to feed composition. Increase in millet flour caused increase in expansion ratio. Expansion ratio is also a degree of gelatinization dependent factor. Study showed starch digestion (maltose content) increased with increase in temperature at increased proportion of millet flour in feed blend as shown in (Fig. 6). Higher temperature might have favored hydrolytic breakage of starch chains along with formation of shorter chain during gelatinization (Guha et al., 1998).

Table 2: Regression coefficients of second order polynomial and their significance for dependent variables

Factors	Water absorption index (WAI)			Water solubility index (WSI)			Starch digestibility (SD) (Maltose content)		
	Coefficient	F value	P value	Coefficient	F value	P value	Coefficient	F value	P value
Intercepts	5.564383	15.27606	< 0.0001**	7.95	15.01998	< 0.0001**	265.917	8.309	0.0001 **
A	-0.086	6.343322	0.0236*	-0.39164	14.96646	0.0015*	8.142	49.028	< 0.0001**
B	0.12123	12.59992	0.0029*	-0.77728	58.95243	< 0.0001**	-2.267	3.800	0.0702
C	-0.0754	4.877019	0.0432*	-0.44202	19.06465	0.0006**	3.608	9.630	0.0073 *
D	-0.1061	9.64497	0.0072*	0.565746	31.23126	< 0.0001**	-0.392	0.113	0.7409
A^2	0.17533	30.11959	< 0.0001**	-0.29205	9.511373	0.0076*	6.402	34.646	< 0.0001**
B^2	0.20978	43.12033	< 0.0001**	-0.16955	3.205658	0.0936	2.715	6.229	0.0247 *
C^2	0.19141	35.89864	< 0.0001**	-0.18705	3.901563	0.0669	0.090	0.007	0.9354
D^2	0.31473	97.05671	< 0.0001**	-0.1958	4.275129	0.0564	-0.660	0.369	0.5528
AB	-0.093	4.948088	0.0419*	-0.22854	3.397697	0.0851	-3.600	6.390	0.0232 *
AC	-0.01	0.05752	0.8137	0.311093	6.295563	0.0241*	-1.663	1.363	0.2613
AD	0.0086	0.042317	0.8398	0.263848	4.528574	0.0503	1.838	1.665	0.2165
BC	0.10473	6.268653	0.0243*	0.889729	51.49567	< 0.0001**	-2.338	2.694	0.1215
BD	0.0779	3.468076	0.0823	-0.03694	0.088785	0.7698	-0.712	0.250	0.6241
CD	-0.1666	15.85542	0.0012*	0.28164	5.159934	0.0383*	0.975	0.469	0.5040
R^2	0.93			0.933			0.88		
Adj. R^2	0.87			0.871			0.77		
Adeq precision	12.43			15.13			13.13		
Lack of fit	2.26	0.19		1.7	0.28		0.75	0.66	

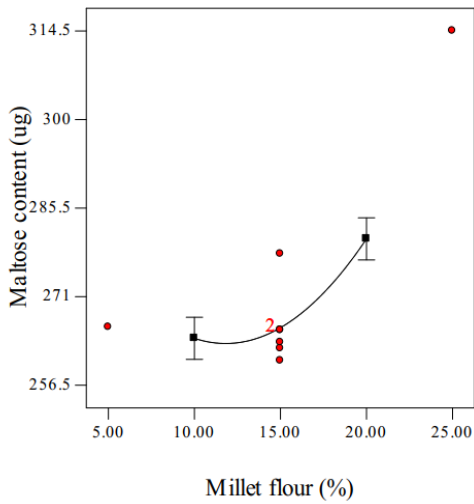**Significant at P < 0.05, **Significant at P < 0.001*

Fig. 5: One factor plot for the variation of maltose content of extrudates as function of feed composition
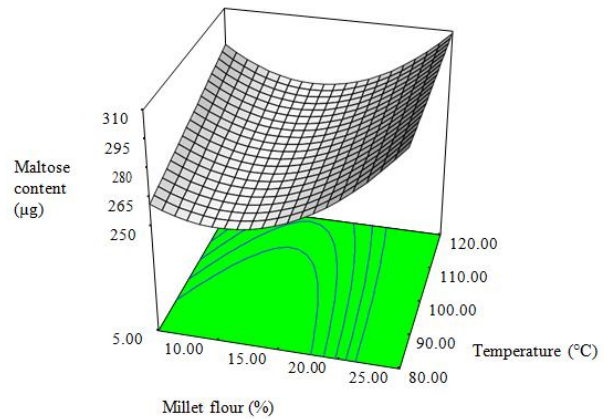

Fig. 6: Response surface plot for maltose content as a function of extrusion temperature and feed composition at center value of feed moisture and screw speed.

The suitability of the model developed for predicting the optimum response values was tested using the recommended optimum conditions of the variables and was also used to validate experimental and predicted values of the responses. The results of the different physic-chemical properties of extrudate are shown in Table 4.

Table 3: Multi response optimization constraints of extrudates with millet flour

Name	Goal	Lower Limit	Upper Limit	Lower weight	Upper weight	Importance
Millet flour (%)	Max	5	25	1	1	3
Feed moisture (%)	R	12	16	1	1	3
Temperature (°C)	R	80	120	1	1	3
Screw speed (rpm)	R	1000	1400	1	1	3
LE (%)	Max	91.11	144.0	1	1	3
BD (g/cm^3)	Min	0.23	0.46	1	1	3
WSI (%)	Max	4.72	9.71	1	1	3
WAI (g/g)	Min	5.40	7.00	1	1	3
Maltose content (µg)	Max	256.5	314.50	1	1	3

Max (Maximize), R (Range), (Min) Minimize

Table 4: Predicted and actual values of the responses at the optimized condition

Responses	Predicted values	Actual values	% deviation from predicted value
Lateral expansion (%)	141.57	152.76	7.9
Bulk density (gm/cm^3)	0.231	0.21	9.09
WSI (%)	7.56	6.9	8.7
WAI (g/g)	6.12	6.7	9.4
Maltose content (µg)	314.5	295	6.2

4. Conclusions

The results revealed that the WAI, WSI and starch digestibility (on the basis of maltose content) were almost equally affected by the variation of millet composition, temperature, screw speed and extrusion temperature. WSI was found decreased with increase in millet incorporation, feed moisture and extrusion temperature but was directly proportional to screw speed. Similarly, WAI decreased with increase in millet incorporation, extrusion temperature and screw speed but was directly proportional to feed moisture. SD increased with increase in millet incorporation and screw speed. Optimizing the process variables could play key role in enhancing the functional properties of extrudates.

Conflicts of Interest

No any conflict of interest.

Funding

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How to cite: KC, Y., Rajbanshi, R., Bhattarai, P., Dhungana, P. K., & Subba, D. (2020). Process Optimization of Finger Millet Incorporated Extrudates. Himalayan Journal of Science and Technology, 3-4, 60-67.