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Multiple Parameter Optimization of Hydration Characteristics and Proximate Compositions of Rice-Soybean Extruded Foods

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Abstract

Multi-parameter extrusion cooking conditions for the extrusion of composite flours of rice and soybean were modeled using response surface methodology (RSM). A five-level-three-factors central composite rotatable design (CCRD) was employed to optimize three process variables including barrel temperature (BRT)(X_1), feed moisture (FMC)(X_2) and feed soybean composition (FSC)(X_3) for the achievement of satisfactory hydration characteristics and proximate composition. The fitted polynomial models indicated significant coefficients, satisfactory coefficients of determination (R^2 and $R^2_{adjusted}$) and non-significant lack-of-fit test. Optimum X_1, X_2 and X_3 were 120˚C, 20%, and 23% for rice-soybean based extrudates. At these optimum combinations, optimum responses were 97.10% for dispersibility, 6.11 water absorption index and 8.42 water solubility index, while proximate composition of 1.02% moisture content, 3.62% lipid, 26.26% protein, 0.48% ash content, 2.14% fibre, 70.13% and 412.14 kcal/100g of energy were obtained. Under the optimized conditions, the responses are well matched with the predicted values and therefore the models could be used to predict the extrusion system in its natural state.

Subject Areas

Analytical Chemistry, Food Science & Technology

Keywords

Optimization, Extrusion, Rice, Soybean, Hydration Properties, Proximate

Composition

1. Introduction

Rice (*Oryza sativa* L.) is one of the important crops in the world in terms of the amounts produced by developing world (480×10^6 tons of rough rice in 2012) and the number of people (3.5 billion) that depend on it as their staple food and sustenance [1]. The global per capita consumption of milled rice was 54.5 kg per person per year in 2014, this includes 77 kg in Asia, 31 kg in South America, 21 kg in Africa, 14 kg in Oceania, and <10 kg in Europe and North America [1]. In Nigeria, per capita consumption was 32 kg per person per year with urban consumption exceeding 47 kg/person/year [2] which is significantly higher than the regional average. More than 3.5 billion people depended on rice for more than 20% of their daily calorie requirement in 2009 and more than 50% of energy supply for more than 520 million people in Asia and sub-Saharan Africa (SSA) provides, most of whom are poor to very poor [1] [2].

But on the overall nutritional assessment, rice has low protein contents (7% - 9%) with slightly high lysine (4% - 5%) content when compared with some common cereals such as sorghum, millet, maize and wheat, which corresponding to an amino acid score of 67% in milled rice [1]. Rice based products can easily therefore be improved by complementing with food materials richer in lysine [3]. Food legumes have been proven to be comparatively richer in lysine and therefore combination of cereal and legume proteins provides an ideal source of dietary protein for humans [3] [4] especially in less developed countries where animal protein is beyond the reach of average populations.

The utilization of locally grown crops such as cowpea, soybean, groundnuts, sesame and Bambara groundnut for the production of high protein, energy and affordable recipes has been proposed as a suitable channel for addressing protein-energy malnutrition (PEM) in industrially less developed nations. Especially, when these products are processed using technology that results in shelf stable, convenient and consumer acceptable products, it will contribute significantly to the overall food and nutrition security of the populace [3] [4] [5]. Legumes are second only to cereals in their importance to human nutrition [6]. Legumes when processed into flour can be used in the preparation of composite flour bread, doughnuts, tortillas, chips, spreads and extruded snacks and porridges or used in liquid form to produce milk analogues and infant formulations [7] [8]. Recent studies indicated that flavones extracted from soybean and other legumes had been suggested to both reduce the risks of cancer and also lower cholesterol level in human [9] [10]. Soybean phytoestrogens have also been suggested as possible alternative to hormone replacer in postmenopausal therapy [8]. According to Iyer *et al.* [11], the necessity for laborious preparation before consumption, long cooking time and the high level of anti-nutritional factors which causes gastro-intestinal disorder after consumption are major contributing factors limiting the utilization of legumes as human foods worldwide. But food processing methods such as soaking, germination, cooking, blanching, roasting, frying, canning and more recently extrusion cooking has been used to minimize these factors in legumes and enhance its utilization [8] [11].

Extrusion cooking (EC) is a high temperature short time (HTST) food processing technique, in which mechanical energy is combined with heat energy to cook starch, plasticizing and reorganizing food materials to create at the die new shaped and textured products, and also has the ability to inactivate enzymes, destroy anti-nutritional factors and reduce microbial activity [12] [13] [14] [15]. EC has been used in the cereal industry for several years to produce diverse foods such as breakfast cereals, snack foods, baby foods, pasta products, extruded bread, modified starches, beverages powders, meat and cheese analogues, textured vegetable protein, and blended foods such as corn starch and ground meats [16] [17]. During EC, due to the varying extrusion conditions, such as extruder parameters, raw materials composition, moisture level and pH, chemical and structural changes occurs in the raw materials, these changes includes starch gelatinization [18], protein denaturation [4], pigment and vitamin degradation, and loss of volatile compounds. These changes resulted in new food product with new functional, nutritional and sensory qualities [19]. Several researchers [4] [5] [14] [16] [17] [18] [20] [21] [22] has developed food products using this technology and reported need for a systematic evaluation of appropriate extrusion variable settings to achieve the optimum quality of finished product.

But the method for the optimization of food production system by varying a single parameter at a time while holding the other parameters at a specified level have been found to be labor intensive, time consuming and economically expensive especially where the system is multiple variables dependent. Multivariate approach using response surface methodology (RSM) and designed experiment provides a more efficient, cost and energy effective alternative by examining simultaneously and systematically more than one variable at a time and their interaction [22]. This approach allows researcher to establish mathematical relationships between input variables and product properties [23] that could be used to predict the process parameters in their natural state.

In view of the industrial potentials of broken rice flour and common legumes in the production of high protein and energy foods that may be used for sustainable protein-energy-malnutrition (PEM) mitigation in developing countries, and the cost effectiveness of EC technology in the development of novel food products, the present study was designed and conducted to optimize extrusion conditions for the production of extruded foods using broken rice fractions blended with soybean. RSM and central composite rotatable design (CCRD) were used for identifying critical process variables and optimizing them for optimum hydration and proximate composition.

2. Materials and Methods

2.1. Materials Sampling and Formulation

Broken rice (FARO 52) fractions were obtained from the Rice Processing Unit and Soybean (TGX-1448-2E) was purchased from the Soybean Research Program of the National Cereals Research Institute (NCRI), Badeggi, Niger State, Nigeria. All the materials were manually cleaned and packaged in sealed polyethylene bags at room temperature (30˚C ± 2˚C) until required. The broken rice fractions were washed in clean water and dried to about 12% moisture content at room temperature (32˚C ± 2˚C). The dried grains were then grinded in an attrition mill (locally fabricated) before sieving with 150 µm laboratory sieve (Brabender OHG Duisburg type). While, twenty (20 kg) kilograms of soybean was separately steeped in tap water at room temperature in a 20 liters plastic bowls. After 24 hrs, the soybean were dehulled using traditional wooden pestle and mortar and after several washing, the dehulled grain were dried to about 14% moisture content in an oven before manually winnowed to have clean dehulled seeds. The dried seeds were then milled into flour and stored in sealed polyethylene bags until required.

Twenty (20) different composite flour blends were formulated based on preliminary trials. The blends were conditioned to appropriate moisture content [24] by spraying with calculated amount of water and mixing continuously at medium speed in a blender. The samples were put in closed plastic buckets and stored overnight. The feed materials were then allowed to stand for 3 hrs to equilibrate at room temperature prior to extrusion exercise.

2.2. Extrusion Cooking Exercise

The different rice-soybean formulations were subjected to extrusion cooking using a twin-screw extruder (SLG 65, Jinan SaibainoTechn. Dev. Co. Ltd, China). Feeds were manually introduced at a speed of 30 rpm which insure that the flight of the screw was filled and avoiding accumulation of fed in the hopper. Desired barrel temperature was maintained by in-build thermostat and a temperature control unit. Experimental samples were collected as steady state was achieved [25] [26]. Since the operation was replicated three times, averages of 20 runs per day were possible (three days) which resulted in 60 individual extrusion runs. Extruded samples were collected and subjected to hydration characteristics and proximate composition analysis.

2.3. Determination of Hydration Characteristics

Water absorption* (*WAI*) *and solubility* (*WSI*) *indices: Water absorption and solubility indices were determined according to the methods described by Anderson *et al.* [27] and Jin *et al.* [28] as modified by Onwulata *et al.* [29]. Extruded foods were grounded and sifted through a 210 µm sieve and 1.0 g taken and placed in a centrifuge tube and 10 ml distilled water added. After standing for 15 minutes and shaking every 5 minutes interval, the samples were centrifuged for 15 minutes at 100 rpm. The supernatant was decanted and the weight gain in the gel was recorded. WAI was calculated as the weight gain by the dry gel weight (Equation (1)). The supernatant were dried overnight at 90˚C and WSI determined as weight of dried supernatant divided by the weight of dry sample (Equation (2))

$$\text{WAI}(g) = \frac{\text{Weight of sediment}}{\text{Weight of dry solid}} \tag{1}$$

$$\text{WSI}(g) = \frac{\text{Weight of disolved solid in supernatant}}{\text{Weight of dry solid}}. \tag{2}$$

2.4. Proximate Composition Determination

Proximate composition (crude protein, ash, fibre, and fat contents) were determined using the methods 920.05, 923.03, 963.09 and 920.85 respectively of Association of Analytical Chemists [30], while moisture content was determined by the standard oven method where ten grams (10 g) of sample (W_i) were dried in an oven at 105˚C for 72 hours, after which the samples were reweighed to determine the final weight (W_f). The percentage moisture content was then calculated using Equation (3). Carbohydrate was calculated by difference (Equation (4)).

$$\text{Moisture content } (\%) = \left[\left(W_i - W_f\right)/W_i\right] \times 100 \tag{3}$$

$$\text{Carbohydrate } (\%) = 100 - (\%\text{Moisture} + \%\text{Protein} + \%\text{Lipid} + \%\text{Ash}). \tag{4}$$

Calorie value was calculated using the Atwater conversion factor system, where 17 Kj/g (4.0 Kcal/g) for protein, 37.0 Kj/g (9.0 kcal/g) for fat and 17.0 Kj/g (4.0 kcal/g) for carbohydrate were used as conversion factors.

2.5. Experimental Design and Statistical Analysis

RSM in a three (3) factors CCRD [31] consisting of 20 experiments (8 factorial points, 6 axial points and 6 central point) (**Table 2**) and five levels combinations coded −1.68, −1, 0, +1, and +1.68 were used to study the effects of extruder barrel temperature (X_1, 100˚C - 140˚C), feed moisture content (X_2, 15 - 25 g/100g sample) and feed soybean composition (X_3, 8 - 24 g/100g sample) (**Table 1**) on the hydration and proximate composition of the extruded foods. Experimental data were subjected to regression analysis using MINITAB 14.13 to model the response factors as a mathematical function of a few continuous factors. Each response (Y) was represented by a mathematical equation that correlates the response surfaces.

$$\begin{aligned} Y = {} & \beta_o + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{11} X^2{}_1 + \beta_{22} X^2{}_2 + \beta_{33} X^2{}_3 \\ & + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 + \beta_{23} X_2 X_3 + \varepsilon. \end{aligned} \tag{5}$$

The model developed for each response was examined for significance, for lack of fit, while 3D response surface plot was designed after removal of the non-significant terms with the same software. Adequacy of the model was also investigated by the examination of residuals [32]. Statistical analysis was carried out using MINITAB 14.13. Hydration and proximate data were analyzed using

ANOVA and Fishers tests to evaluate if each term has a significant effect ($p \leq 0.05$), while the optimum level of each variables were obtained by graphical and numerical optimization analysis.

3. Results and Discussion

3.1. Response Surface Methodology and Central Composite Design

The effects of extruder barrel temperature, feed moisture content and feed blend composition on the quality characteristics of rice-soybean extruded food were studied using data generated from central composite rotatable experimental design (CCRD) (Table 1). The three independent variables were coded X_1, X_2 and X_3 respectively. Preliminary experiment indicated that minimum feed moisture content, soybean composition and process temperature for proper operation were 11.59 g/100g, 2.55 soybean/100 g rice flour and 86.36˚C barrel temperature. Based on these results, a central composite rotatable design was formed with five levels of variation of temperature (86.36˚C, 100˚C, 120˚C, 140˚C, and 153.64˚C), feed composition (2.55%, 8%, 16%, 24%, and 29.45%) and moisture content (11.59%, 15%, 20%, 25%, and 28.41%). The experimental matrix is shown in Table 1. The five experimental units formed a 20 full factorial design whereas 17 experimental runs were used with run 15 repeated three times (Table 2). Regression analysis of MINITAB 14.13 fitted the results to second order polynomial model (Equation (5)). The experimental and predicted results of the application of RSM and CCRD to the production of rice-soybean extruded foods in terms of functional and proximate compositions are presented in Table 3 and Table 4 respectively.

3.2. Effect of Process Variables on Hydration Characteristics

3.2.1. Effect of Extrusion Variables on Dispersion Index (DI)

The dispersibility attributes of extrudated foods is its ability to be wet without the formation of lumps, with simultaneous disintegration of agglomerates in aqueous solution. The most dispersed sample (98.16%) was obtained at 140˚C BRT, 25 g/100g FMC and 24 g/100g soybean composition and the lowest (96.99%) at 100˚C BRT, 15 g/100g FMC and 24 g/100g FSC. DI seems to decrease with decreasing extrusion temperature and feed moisture composition, while soybean addition did not show any effect on the dispersibility. Similar results were reported by Almeida-Dominguez *et al.* [33] and Kone and Launay [34]. Molecular

Table 1. Process variables and their coded and natural levels in central composite design for k = 3.

Independent variables	Unit	Symbol	Variable levels				
			$-\alpha$	Low	Medium	High	$+\alpha$
			−1.682	−1	0	+1	+1.682
Barrel temperature	˚C	X_1	86.36	100.00	120.00	140.00	153.64
Feed moisture content	% (g/100g)	X_2	11.59	15.00	20.00	25.00	28.41
Feed soybean composition	% (g/100g)	X_3	2.55	8.00	16.00	24.00	29.45

Table 2. Central composite rotatable design matrix and variable combinations for experimental runs.

Run	Coded and natural forms of variables					
	BRT (˚C) (X_1)		FMC (g/100g) (X_2)		FSC (g/100g) (X_3)	
	Coded	Un-coded	Coded	Un-coded	Coded	Un-coded
1	−1	100.00	−1	15.00	−1	8.00
2	1	140.00	−1	15.00	−1	8.00
3	−1	100.00	1	25.00	−1	8.00
4	1	140.00	1	25.00	−1	8.00
5	−1	100.00	−1	15.00	1	24.00
6	1	140.00	−1	15.00	1	24.00
7	−1	100.00	1	25.00	1	24.00
8	1	140.00	1	25.00	1	24.00
9	−1.682	86.36	0	20.00	0	16.00
10	1.682	153.64	0	20.00	0	16.00
11	0	120.00	−1.682	11.59	0	16.00
12	0	120.00	1.682	28.41	0	16.00
13	0	120.00	0	20.00	−1.682	2.55
14	0	120.00	0	20.00	1.682	29.45
15	0	120.00	0	20.00	0	16.00
16	0	120.00	0	20.00	0	16.00
17	0	120.00	0	20.00	0	16.00

BRT = Barrel temperature, FMC = Feed moisture content, FSC = Feed soybean composition. Duplicate runs were carried out at all design point and average recorded. The experimental runs were randomized. Sample 17 is a mean of four experimental runs.

dispersion of starch-based extruded foods was affected by moisture content and temperature during processing. Jackson *et al.* [35] also observed relatively low dispersion at temperature slightly higher than starch gelatinization temperature. This justifies the increased dispersion of extrudates at higher temperature.

3.2.2. Effect of Extrusion Variables on WAI and WSI

The observed and predicted values of the WAI and WSI are presented in Table 3. The observed mean values for WAI ranged from 6.13 to 7.28 g/g for samples 5 and 7 representing 100˚C extrusion temperature, 15% FMC and 24% feed soybean composition (FBC) and 100˚C BRT, 25% FMC and 24% FBC respectively. From these results it is clear that there is an inverse relationship between dispersion and WAI, while addition of soybean did not significantly alters the WAI value (Table 3), barrel temperature and feed moisture content on the other hands seems to be the main processing variable affecting WAI. Similar result was reported by Ding *et al.* [15]. Water absorption index (WAI) is the measure of the volume occupied by food materials after swelling in excess water, which maintains the integrity of foods in aqueous dispersion, while water solubility index (WSI) is the measure of the degree of starch degradation during food processing which is reflected in the amount of polysaccharides from starch component. WSI therefore is a measure of molecular degradation and molecule solubility in water during preparation.

The WSI value varied between 8.31 and 8.93 in samples 13 and 7 respectively (Table 3). The highest value was recorded in sample extruded under 100˚C BRT, 25% FMC and 24% soybean composition conditions, and the least value observed in sample extruded at 120˚C BRT and 20% FMC and 2.6% soybean blend. Increasing barrel temperature and decreasing feed moisture and blend compositions during extrusion exercise seems to favor WSI characteristics of extrudates. This observation may probably be as a result of fact that during extrusion cooking, water is absorbed by feed material and at high temperature, favors the degradation of starchy component, hence increase the amount of small molecules in the final product. Conway [36], Artz *et al.* [37] and Badrie and Mellows [38] reported similar observation, where they all agree that at high temperature and

Table 3. Observed and predicted values of the hydration characteristics of the extruded foods.

Experimental runs	Hydration characteristics					
	Dispersibility (%)		Water absorption		Water solubility	
	Observed	Predicted	Observed	Predicted	Observed	Predicted
1	97.41	97.39	6.45	6.51	8.39	8.38
2	97.57	97.56	7.03	7.05	8.67	8.67
3	97.49	97.50	6.52	6.54	8.37	8.36
4	98.14	98.15	6.18	6.20	8.49	8.61
5	98.16	98.13	6.13	6.12	8.71	8.69
6	97.59	97.57	6.64	6.62	8.37	8.38
7	97.07	97.08	7.28	7.27	8.93	8.95
8	96.99	97.00	6.88	6.88	8.53	8.59
9	97.41	97.42	6.73	6.73	8.44	8.45
10	97.48	97.49	6.88	6.86	8.42	8.40
11	97.51	97.65	6.40	6.39	8.74	8.74
12	97.28	97.26	6.66	6.64	8.93	8.90
13	97.96	97.96	6.47	6.44	8.31	8.37
14	97.61	97.62	6.68	6.69	8.63	8.62
15	97.82	97.87	6.28	6.29	8.53	8.59
16	97.81	97.86	6.31	6.30	8.55	8.60
17	97.83	97.87	6.28	6.29	8.54	8.59
Mean	97.60	97.61	6.58	6.58	8.56	8.58
CV (%)	0.338	0.341	4.801	4.762	2.166	2.081

low moisture, long chain molecules of food materials are broken down during extrusion to simple soluble molecules which increase WSI.

3.3. Effect of Process Variables on the Proximate Composition

3.3.1. Effect of Process Variables on Moisture Content of Extrudate

The observed and predicted proximate composition results for rice-soybean based extrudates are presented in Table 4. The moisture content of the extrudates varied between 0.08% and 1.31%. These results demonstrate that product moisture content decrease with increasing extrusion temperature.

3.3.2. Effect of Process Variables on Protein Content of Extrudate

The highest protein content of 28.30% was observed in run 13, representing 120˚C BRT, 20% FMC and 2.6% FBC, while the lowest value of 21.93% was recorded in run 9 (86.4˚C BRT, 20% FMC and 8% FBC).

The addition soybean flour to the rice to produce extruded starch-based food increases protein content, probably due to the increase in the number of sites for crosslinking, but this shortens the starch matrix, resulting in tough, non-expanded crusts, similar result was reported by Martinez-Serna and Villota (1992). Generally, EC results in significant decrease in protein content of extruded materials (Sobota *et al.*, 2010). The reduction in protein content may be attributed to reduction in nitrogen content as a result of the effect of the formation of isopeptide bonds between ε-amine groups of lysine and amine groups of asparagines or glutamine, accompanied by the release of ammonia (Stanley and Baker, 2002).

3.3.3. Effect of Process Variables on Lipid Content of Extrudate

The lipid content remain within a close range of 3.20% and 4.36% for runs 3 and 15 representing 100˚C BRT, 25% FMC and 8% FBC and 120˚C barrel temperature, 20% feed moisture and 16% soybean. The high lipid content was observed when soybean was added to the rice flour may be because soybean is an oil crop, though the value is not as high as 40% - 42% often quoted for soybean seeds. Lipid levels over 5% - 6% impair extruder performance (Camire, 2001).

The presence of soybean protein in the feed formulation creates a favorable-condition for the formation of starch-lipid and lipid-protein complexes, thereby resulting in varying levels of fat contents. Sabota *et al.* (2010) reported that in a sample containing 20% of wheat bran and extruded at a die diameter of 4.2 mm, the degree of fat complex formation was 70.77%. As the content of wheat bran in the formulation increased, there was a concurrent reduction in the complex of fat reaching 29.63% at 80% bran content. They also report that similar effect of lipid bonding may be exerted due to change in feed moisture content. The fat content of extrudate increased considerably with increasing moisture content of the feed materials. Fat increased from 0.62% at feed moisture content of 14% to 0.92% at 29% moisture content (Sabota *et al.*, 2010) similar to what is seen in this study.

3.3.4. Effect of Process Variables on Fibre Content of Extrudate

The fiber content of the rice-soybean extrudates ranged from 0.77% - 3.22% for runs 10 and 8 representing 153.6˚C barrel temperature, 20% feed moisture and 16% soybean and 140˚C barrel temperature, 25% feed moisture and 24% soybean. Increasing soybean content and reducing extrusion temperature favorsfiber recovery. For the crude fiber content of rice-soybean extrudates, the highest value (0.62%) was observed in sample 2 extruded under extrusion condition of 140˚C barrel temperature, 15% feed moisture and 8% soybean and the least value of 0.35 was observed in sample 14 (120˚C barrel temperature, 20% feed moisture and 29.5% soybean).

Varo *et al.* (1983) reported insignificant changes in fibre content of twin- screw extruded wheat flour and whole-wheat meal at 161˚C - 180˚C process temperature and 15% feed moisture. Non-significant change was also found in dietary fibre content when wheat was extruded under milder conditions, but the fibre present became slightly more soluble (Mendez-Garcia *et al.*, 2011). On the other hand, an increase in dietary fibre content of wheat flours with increasing product temperature (150˚C - 200˚C) was reported. This is similar to results of this study.

3.3.5. Effect of Process Variables on Carbohydrate Content of Extrudate

The carbohydrates and caloric values for rice-soybean extrudates as influence by the three independent variables varied from 66.30% - 73.00%, and 403.97 - 416.08 kcal/100g respectively. The highest and least values of carbohydrate were recorded in samples 13 and 10 representing extrusion conditions 120˚C barrel temperature, 20% feed moisture and 2.6% soybean and 153.6˚C barrel temperature, and 20% feed moisture and 16% soybean.

Carbohydrates contents of foods range from simple sugars to more complex molecules, like starch and fibre. Several researchers have reported sugar losses during EC (Noguchi *et al.*, 1982; Camire *et al.*, 1990). This may probably be as a result of the conversion of sucrose into reducing sugars, and loss of these reducing sugars during Maillard reactions with proteins.

3.3.6. Effect of Process Variables on Caloric Value of Extrudate

While the highest and lowest values for caloric values were seen in samples 8 and 15 produced under independent variable combination of 140˚C barrel temperature, 25% feed moisture and 24% soybean for sample 8 and 120˚C barrel temperature, 20% feed moisture and 16% soybean for sample 15. The caloric value of 403.97 - 416.08 kcal/100g for rice-soybean composite flour extrudates is higher than 345 kcal/100g often reported for milled rice. The addition of these legumes improves the protein, lipid and other compositions of the final product, hence its contribution to increased calories per 100 g.

3.4. Estimation and Validation of the Model Parameters for Hydration Characteristics

By applying multiple regression analysis on the experimental data (Table 4 and Table 5), the model fitted for the response variables could be expressed by the

Table 4. Observed and predicted mean effects of barrel temperature, feed moisture content and feed composition on the proximate composition of rice-soybean based extrudates.

Run	Moisture Content (%)		Lipid content (%)		Crude Protein (%)		Fibre (%)		Ash content (%)		Carbohydrate (%)		Energy (Kcal/100g)	
	OBS	PDT	OBS	PDT	OBS	PDT	OBS	PDT	OBS	PDT	OBS	PDT	OBS	PDT
1	1.310	1.297	3.220	3.338	25.800	25.906	0.960	1.372	0.480	0.435	69.540	68.794	410.340	409.392
2	0.700	0.693	3.410	3.536	25.220	25.318	0.980	0.663	0.620	0.593	69.770	69.920	410.650	412.529
3	0.810	0.817	3.200	3.321	24.680	24.596	1.180	0.897	0.560	0.513	70.380	70.578	409.040	410.684
4	0.470	0.451	3.880	3.889	25.110	25.128	1.000	1.030	0.500	0.446	69.510	69.422	413.400	413.606
5	0.660	0.626	4.000	4.102	23.000	22.941	1.080	0.837	0.390	0.364	71.130	71.388	414.120	415.521
6	0.410	0.385	3.500	3.530	23.020	23.081	1.120	1.163	0.500	0.487	71.860	71.782	411.020	410.962
7	0.330	0.319	3.820	3.829	23.620	23.465	1.380	1.427	0.500	0.472	70.680	70.646	411.580	411.253
8	0.310	0.315	3.610	3.627	24.810	24.663	3.220	2.578	0.400	0.370	67.880	68.757	403.970	406.479
9	1.090	1.040	3.650	3.478	21.930	21.982	0.930	0.774	0.510	0.490	72.890	73.156	412.490	412.118
10	0.580	0.529	3.580	3.474	22.120	22.494	0.770	1.145	0.530	0.544	73.000	72.515	412.700	410.741
11	0.750	0.742	3.890	3.685	24.140	24.005	1.020	0.909	0.500	0.477	70.450	70.705	413.370	412.724
12	0.340	0.281	3.810	3.752	24.140	24.286	1.360	1.699	0.400	0.444	70.220	69.661	411.730	410.041
13	0.830	0.791	3.880	3.657	28.300	28.168	1.080	0.987	0.440	0.450	66.300	66.622	413.320	412.397
14	0.140	0.112	4.120	4.080	25.100	25.283	1.560	1.852	0.350	0.326	68.870	68.244	412.960	411.557
15	0.080	0.083	4.360	4.367	24.240	24.211	1.020	0.964	0.410	0.394	69.970	69.981	416.080	416.432

OBS = Observed, PRD = Predicted, X_1 = Barrel temperature, X_2 = Feed moisture content, X_3 = Feed composition. Duplicate runs were carried out all design point and average recorded. The experimental runs were randomized.

Table 5. Estimated regression equation coefficients for response variables (proximate composition) in rice-soybean blends.

Coefficient	DI	WAI	WSI	Moisture	Lipid	Protein	Fiber	Ash	CHO	Energy
Constant	88.999**	10.632**	5.858**	16.6771*	−12.2626*	15.1346*	11.5455*	1.0045	84.3212*	288.547*
					Linear coefficient					
X_1	0.0837**	−0.0613**	0.0519**	−0.1773*	0.1986*	0.3536*	−0.0607	−0.0140	−0.4826*	1.244*
X_2	0.1870**	0.0362**	−0.1352**	−0.3585*	0.2851*	−0.4657*	−0.4996*	0.0250	0.7613*	3.318*
X_3	0.2819**	−0.1714**	0.1054**	−0.1799*	0.2800*	−0.9057*	−0.3755*	−0.0007	0.9640*	2.894*
					Quadratic coefficient					
X_1^2	−0.0004**	0.0005**	−0.00015**	0.0006*	−0.0008*	−0.0017*	0.0000	0.0001*	0.0025*	−0.004*
X_2^2	−0.0059**	0.0033**	0.00329**	0.0061*	−0.0092*	−0.0009	0.0048	0.0009*	0.0029	−0.071*
X_3^2	−0.0004*	0.0016**	−0.00054**	0.0020*	−0.0028*	0.0139*	0.0025	−0.00003	−0.0141*	−0.025*
					Interaction coefficient					
X_{12}	0.0012**	−0.0022**	−0.00011	0.0006*	0.0009*	0.0028*	0.0021*	−0.0006*	−0.0057*	−0.001
X_{13}	−0.0011**	−0.0001	−0.00094**	0.0006*	−0.0012*	0.0010*	0.0016*	−0.00005	−0.0011	−0.012*
X_{23}	−0.0073**	0.0070**	0.00168**	0.0011*	−0.0016	0.0115*	0.0067*	0.00019	−0.0158*	−0.035*
R^2	99.62	99.61	99.01	99.20	88.50	99.00	74.20	81.80	93.80	75.80
R^2_{adj}	99.41	95.01	98.60	98.90	83.30	98.60	62.60	73.60	91.00	64.90
Lack-of-fit	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS

[a] $Y = \beta_o + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{11} X_1^2 + \beta_{22} X_2^2 + \beta_{33} X_3^2 + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 + \beta_{23} X_2 X_3$; X_1 = Barrel temperature, X_2 = Feed Moisture content, X_3 = feed blend composition, * and ** = significant at 5% and 1% level of probability respectively.

following quadratic polynomial equations in the form of coded values (Equations ((6)-(8))) for hydration characteristics.

Dispersion index

$$\begin{aligned}&88.9988 + 0.0837X_1 + 0.1870X_2 + 0.2819X_3 - 0.0004X_1^2 - 0.0059X_2^2 \\ &-0.0004X_3^2 + 0.0012X_1X_2 - 0.0011X_1X_3 - 0.0073X_2X_3\left(R^2 = 99.01, R_{\text{adj}}^2 = 98.60\right)\end{aligned} \quad (6)$$

Water absorption index

$$\begin{aligned}&10.632 - 0.0613X_1 + 0.0362X_2 - 0.1714X_3 + 0.0005X_1^2 + 0.0033X_2^2 \\ &+0.0016X_3^2 - 0.0022X_1X_2 - 0.0011X_1X_3 + 0.0007X_2X_3\left(R^2 = 99.62, R_{\text{adj}}^2 = 99.41\right)\end{aligned} \quad (7)$$

Water solubility index

$$\begin{aligned}&5.8581 + 0.0519X_1 - 0.1352X_2 + 0.1054X_3 - 0.00015X_1^2 + 0.00329X_2^2 \\ &-0.0054X_3^2 - 0.00011X_1X_2 - 0.00094X_1X_3 + 0.00168X_2X_3\left(R^2 = 99.61, R_{\text{adj}}^2 = 95.01\right).\end{aligned} \quad (8)$$

Equation (6) indicates that at linear level, process variables (X_1, X_2, and X_3) had positive effects on the dispersion. At quadratic level, all the variables had negative effect on the dispersion of the extrudate in water, while interaction between barrel temperature and feed soybean composition had negative effect. **Table 5** is the ANOVA of the coefficient of regression. From this result, there is a significant ($p < 0.05$) interaction between the process variables. Significant interaction indicates that the level of one of the interaction variables can be increased while that of other decreased for constant value of the response (Adeyanju *et al.*, 2016a, 2016b). For WAI, addition of soybean at linear level to the formulation resulted in reduction of response (Equation (7)). While interaction of barrel temperature and feed moisture content were positive, indicating antagonistic effect on response. There was negative contribution of barrel temperature to WSI, indicating that as the temperature increase, WSI also decreases (Equation (8)).

The analysis of variance (ANOVA) for the regression models representing the relationship between independent variables and hydration characteristics in terms of DI, WAI and WSI is presented in **Table 6**. The coefficient of determination (R^2) 99.01%, 99.62% and 99.61% for the DI, WAI and WSI respectively indicated that only 0.99%, 0.38% and 0.39% of the variability observed could not be explained by the fitted models. For a statistical model to better explain variability in response, it is important that the adjusted determination coefficient (R_{adj}^2.) be close to R^2. As shown in Equations (6)-(8), R^2 was close to R_{adj}^2 and this confirmed that the models were highly significant and that the model could be used to navigate the design space. Koocheki *et al.* [39] reported that for a good fitted polynomial model, R^2 should not be less than 80%, while Chauhan and Gupta [40] were of the opinion that R^2 greater than 75% is acceptable for multivariate system model. It is also important to take into consideration the fact that adding additional variable to the model will always increase R^2, regardless of whether the additional variable is statistically significant or not. Thus, a large R^2 does not always imply adequacy of the model. Adjusted R^2 is therefore the best measure as higher R^2_{adj} indicated that non-significant terms have not been included in the model as observed in this study.

At the same time, relatively low values of coefficient of variation (CV) (**Table 6**) (0.338, 0.341 and 4.801) indicates a better precision and reliability of the experimental values. Lack-of-fit test also indicated non-significant *p*-value ($p <$ 0.05) for the hydration characteristics (**Table 5**). Non-significant lack-of-fit is desired as a significant test indicates that there may be contributions in the regression models that are not accounted for by the fitted models. Therefore, the models are quite adequate for the prediction of the system in the range of experimental variables.

3.5. Estimation and Validation of the Model Parameters for Proximate Composition

Seven predictive models (Equations (9)-(15)) were obtained as a result of fitting the second order polynomial of Equation (1) to experimental data for the effects of different combinations of extrusion process variables on the proximate composition. These predictive models were tested for adequacy and fitness by analyses of variance (ANOVA, **Table 5**). According to Myers and Montgomery [41] and Milán-Carrillo *et al.* [42], a satisfactory predictive model should have an adjusted $R^2 \geq 0.80$, a significance level of $p < 0.05$, coefficients of variance (CV) values ≤ 10%, and lack of fit test non-significant; all these parameters could be used to decide whether the model satisfactorily predict the relationship between the independent and dependent variable. From this study, the adjusted R^2 for the predictive models are greater than 80% except for fibre (62.60%), ash (73.60) and caloric value (64.90). These results are indication that only 1.1% moisture, 16.7% lipid, 1.4% protein, 37.4 fibre, 26.40% ash, 9.0% carbohydrate and 35.1% calorie variations are not accounted for by the model and therefore can satisfactorily predict optimum response in terms of proximate composition. The models were also having significant p-value ($p < 0.05$) and the lack-of-fit test were non-significant ($p < 0.05$) (**Table 5**) indicative of the appropriateness of the models. The significance of each coefficients measured using p-value and F-value is listed in **Table 5**. The p-value of the models and coefficients are less than 0.0001, which indicates that the model is significant and can be used to optimize the process variables R^2.

Moisture

$$\begin{aligned}&16.677-0.1773X_1-0.3585X_2+-0.1799X_3+0.0006X_1^2+0.0061X_2^2\\&+0.002X_3^2+0.0006X_1X_2+0.0006X_1X_3+0.0011X_2X_3\left(R^2=99.20, R^2_{\text{adj}}=98.90\right)\end{aligned} \tag{9}$$

Lipid

$$\begin{aligned}&-12.2626+0.1896X_1+0.2851X_2+0.2800X_3-0.0008X_1^2-0.0092X_2^2\\&-0.0028X_3^2+0.0009X_1X_2-0.0012X_1X_3-0.0016X_2X_3(R^2=88.50, \mathrm{R}^2_{\text{adj.}}=83.30)\end{aligned} \tag{10}$$

Protein

$$\begin{aligned}&15.1346+0.03536X_1-0.4657X_2-0.9057X_3-0.0017X_1^2-0.0009X_2^2\\&+0.0139X_3^2+0.0028X_1X_2+0.001X_1X_3+0.0115X_2X_3\left(R^2=99.00, R^2_{\text{adj}}=98.60\right)\end{aligned} \tag{11}$$

Fibre

$$11.5455 - 0.0607X_1 - 0.4996X_2 - 0.3755X_3 + 0.0048X_2^2 + 0.0025X_3^2 + 0.0021X_1X_2 + 0.0016X_1X_3 + 0.0067X_2X_3 \left(R^2 = 74.20, R_{\text{adj}}^2 = 62.60\right) \quad (12)$$

Ash content

$$0.0045 - 0.0140X_1 + 0.02498X_2 - 0.0007X_3 + 0.00011X_1^2 + 0.00094X_2^2 - 0.00003X_3^2 - 0.00056X_1X_2 - 0.00005X_1X_3 + 0.00019X_2X_3 \left(R^2 = 81.80, R_{\text{adj}}^2 = 73.60\right) \quad (13)$$

Carbohydrate content

$$84.3212 - 0.4826X_1 + 0.7613X_2 + 9640X_3 + 0.0025X_1^2 + 0.0029X_2^2 - 0.0141X_3^2 - 0.0057X_1X_2 - 0.0011X_1X_3 - 0.0158X_2X_3 \left(R^2 = 93.80, R_{\text{adj}}^2 = 91.00\right) \quad (14)$$

Caloric value

$$288.547 + 1.244X_1 + 3.318X_2 + 2.894X_3 - 0.004X_1^2 - 0.071X_2^2 - 0.025X_3^2 - 0.001X_1X_2 - 0.012X_1X_3 - 0.035X_2X_3 \left(R^2 = 75.80, R_{\text{adj}}^2 = 64.90\right). \quad (15)$$

3.6. Optimization of the Extrusion Process

The 3D response surface plot of the effect of process variables on the hydration characteristics are presented in Figures 1-3 for DI, WAI and WSI respectively of rice-soybean extrudate. The 3D surface plots are graphical representation of regression equation and are very useful in judging the relationship between independent and dependent variables and also locating point of optimum conditions.

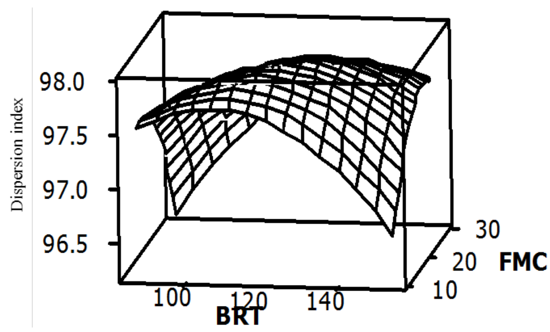


Figure 1. Response surface plot for the effect of barrel temperature versus feed moisture content on the dispersion index (%) of rice-soybean extrudates.

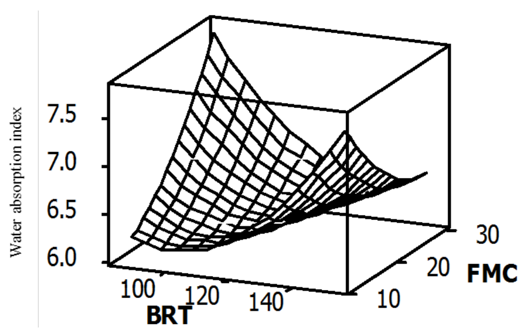


Figure 2. Response surface plot for the effect of barrel temperature versus feed soybean content on the water absorption index of rice-soybean extrudates.

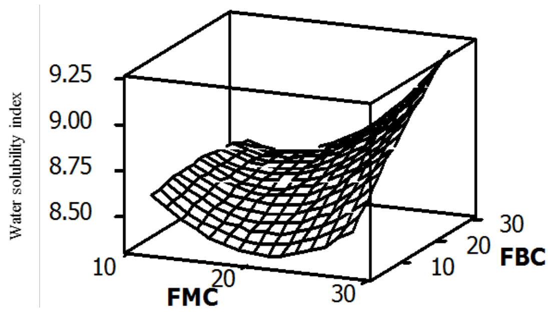


Figure 3. Response surface plot for the effect of barrel temperature versus feed soybean content on the water solubility index of rice-soybean extrudates.

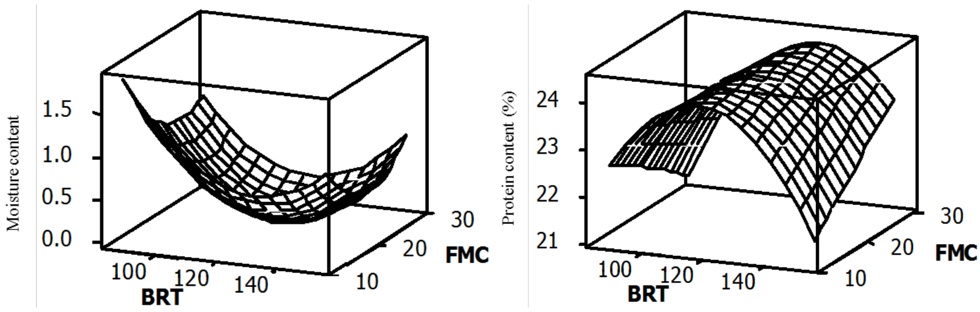
In these plots, two variables are depicted in three-dimensional surface plot; the third variable is fixed at constant level. In this study, the response surfaces are typical for rice based products during extrusion and are indicative that the ranges of variables were chosen properly [43] and optimum DI and WAI are located between 120˚C BRT and 20% FMC (**Figure 1** and **Figure 2**).

As shown in **Figure 1** and **Figure 2**, at high barrel temperature and low moisture content, DI and WAI are significantly ($p \leq 0.05$) reduced and at barrel temperature around 120˚C optimum DI and WAI are attained. Feed moisture content and feed soybean composition significantly ($p \leq 0.05$) affects DI value. Increase in fiber content as a result of the addition of soybean flour may be responsible for the reduction in DI value. Charunuch *et al.* [44] reported that fibre content of food reduce or delay swelling of granules thereby resulting in poor reconstitution in water and therefore lower dispersion. The flat surface plot for WSI (**Figure 3**) is an indication that the optimum WSI can be attained even when one of the variables is varied. At moisture content of about 10%, the WSI decrease gradually as the FMC reaches 20% and steadily increases again as the moisture increases to about 30% (**Figure 3**). While WSI index increased as the feed soybean content increase and reaches a maximum at about 28% feed soybean content (**Figure 3**). But the 3D for the WSI is rather symmetrical and flat near the optimum, indicating that the optimized values may not vary widely from the single variable conditions.

Beside the starch degradation at high temperature, low moisture and high shear force which results in the release of low molecular compounds, gelatinization and other related reactions have also been reported to occur during extrusion which may results in the formation of low molecular weight compounds, hence influencing water solubility behavior of the extrudates (**Figures 1-3**). Camire and Krumhar [45], Marzec and Lewecki [46] and Mesquita *et al.* [47] reported that protein denaturation, starch gelatinization and swelling of crude fiber content of feed during extrusion cooking could also be responsible for the variations of the WAI and WSI behavior in extruded materials as seen in this study. Uncovering of hydrophilic groups in extruded starch-protein materials by unfolding and loosing of biopolymers chains increases greater availability of simple soluble materials and easier penetration of structures by water molecules, thereby affecting WAI and WSI and indispersion.

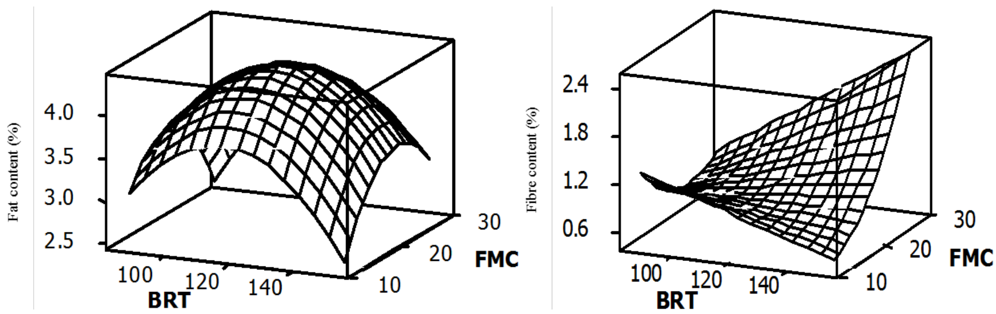
3D response surface plots for the proximate composition are presented in Figures 4-7. The graphs are representations of regression equation as it relates to the relationship between the dependent and independent variables. The three-dimensional representation of the response surfaces and generated by the model shows when two variables are depicted in three-dimensional surface plots, and the third variable is fixed at zero level.

As the feed moisture content increased, higher extrudate moisture was obtained due to the higher amount of water present in dough. While increasing processing temperature decrease product moisture content, the effect was only significant for high added moisture level. Similar results were reported by Badrie



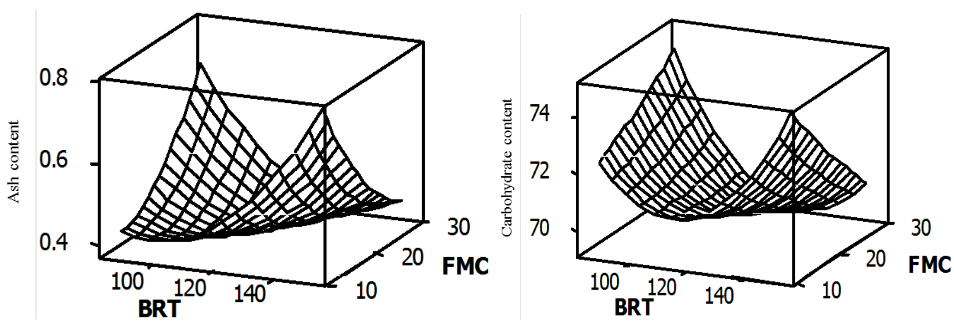
(a) (b)

Figure 4. Response surface plot for the effect of barrel temperature versus feed moisture content on the moisture content (%) (a) and protein (%) (b) of rice-soybean extrudates.



(a) (b)

Figure 5. Response surface plot for the effect of barrel temperature versus feed moisture content on the fat content (%) (a) and fibre (%) (b) of rice-soybean extrudates.



(a) (b)

Figure 6. Response surface plot for the effect of barrel temperature versus feed moisture content on the ash content (%) (a) and carbohydrate (%) (b) of rice-soybean extrudates.

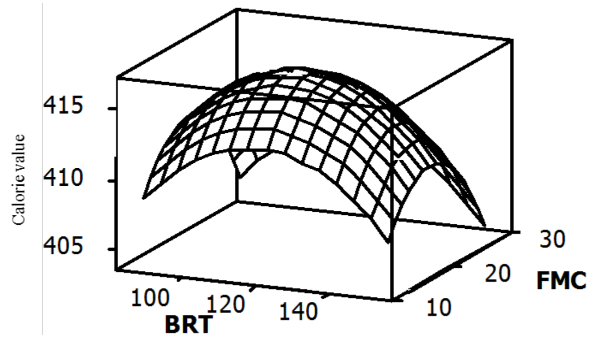


Figure 7. Response surface plot for the effect of barrel temperature versus feed moisture content on calorie value (kcal/100g) of rice-soybean extrudates.

and Mellowes [38] and attributed this increase in extrudate moisture content to the flash of water that happened as the product leaves the die. As indicated in **Figure 5(a)**, the maximum extrudate fat level occurs at around 130˚C barrel temperature and 20% feed moisture conditions. The fat content increased with increasing temperature, however, when the temperature reaches slightly above 130˚C, the effect of temperature significantly ($p \leq 0.05$) decreased to its minimum level above 140˚C. But fat content increased as the feed moisture increased until it reaches its optimum around 20%, then decreased as the moisture level increased above this percentage. It can be concluded that the lipid content of rice-soybean extrudates is almost primarily controlled by the barrel temperature and amount of legume. Based on the fitting model for the effects of process variables on the protein content of rice-soybean extrudates, the optimal process of the extruded foods protein content was optimized. The relationship between the independent variables and dependent variables (protein) is graphically represented by 3D surface plots (**Figure 4(b)**). It depicted that the highest protein level could be achieved when temperature were set at around 120˚C and feed moisture content of slightly above 20%.

3.7. Numerical Optimization of Process Variables

MINITAB's Response Optimizer was used for simultaneous numerical optimization of the multiple responses, to search for a combination of independent variables levels that simultaneously satisfy the target requirement placed on each response and factors. Anuar *et al.* [48] and Gupta *et al.* [49] suggested that numerical optimization require that goals (*i.e.* None, Maximum, Minimum, Target or Range) should be set for the variables and response where all goals are combined into one desirable function. In this study, good sets of conditions that will meet all the goals, the independent variables (i) barrel temperature (100˚C - 140˚C), (ii) feed moisture content (15 - 25 g·100g^{-1}) and (iii) feed blend composition (8 - 24 g·100g^{-1}) were all set within range after preliminary experiment, while WAI, WSI, DI and protein score were set at 5. Importance of score of a goal is within 1 to 5 and setting goal importance at 3 indicates that the variable is considered to be equally important, but set at 5, the response target objective is to meet the objective of getting response at maximum level as applied in this study (Table 6).

By applying the desirability function, the best optimum independent variables for X_1, X_2 and X_3 were 120˚C BRT, 20% FMC, 23 FSC for rice-soybean based extrudates. At these combinations, optimum hydration properties were 97.10% DI, 6.11 WAI and 8.42 WSI, while proximate composition of 1.02% moisture content, 3.62% lipid, 26.26% protein, 0.48% ash content, 2.14% fibre, 70.13% and 412.14 kcal of energy (Table 6) were the optimum value attained.

4. Conclusions

Extrusion cooking of varying formulations of composite flour of broken rice and soybean was performed for the production of extruded foods having good hydration characteristics and proximate composition using designed experiment and optimized by RSM. CCRD was used to evaluate and optimize the effects of barrel temperature, feed moisture content and feed soybean composition on the hydration and proximate composition. The results shows that the process variables significantly affect the hydration behavior and proximate composition of the rice-soybean extruded foods, while the high coefficient of determination of regression models fitted from the experimental data and the non-significant lack-of-fit indicated that the models could be used to satisfactorily predict the hydration and proximate composition during application.

The optimized hydration properties were 97% dispersion index, 6.11 WAI and 8.42 WSI, while proximate composition is of 1.02% moisture content, 3.62%

Table 6. Constraints and goals applied to derive optimum conditions of processing parameters and responses for rice-soybean based formulations.

Variables	Goal	Upper limit	Lower limit	Importance	Optimum level
Independent variables					
Moisture content (% w.b)	In range	8	24	3	20.00
Blend ration (% legume)	In range	15	25	3	23.00
Barrel Temperature (˚C)	In range	100	140	3	130.00
Response variables					
Functional characteristics					
Dispersibility (DSPLTY)	Maximize	96.99	98.16	5	97.10
Water absorption capacity (WAI)	Maximize	6.13	7.28	5	6.11
Water solubility index (WSI)	Maximize	8.31	8.93	5	8.42
Proximate composition					
Moisture content (%)	Minimize	0.08	1.31	3	1.02
Lipid content (%)	Minimize	3.20	4.36	3	3.62
Protein (%)	Maximize	21.93	28.30	5	26.26
Ash content (%)	Minimize	0.35	0.62	3	0.48
Fibre (%)	Maximize	0.77	3.22	5	2.14
Carbohydrate (%)	Minimize	66.30	73.00	3	70.13
Calorie value (kcal/100g)	Maximize	403.97	416.08	3	412.14

lipid, 26.26% protein, 0.48% ash content, 2.14% fibre, 70.13% carbohydrate and 412.14 kcal/100g calorie value (Table 6). Therefore, the models can predict the extrusion condition in its natural state. Combining response surface methodology and extrusion cooking at the optimum process conditions not only improved the quality of rice-legume based food products but also simplified the experiment process. In order to develop extruded foods having functional health benefits and ensure the steady development of the cereal industry in sub Saharan Africa (SSA), appropriate processing and system analysis and development of extrusion cooking using common raw materials will provide scientific theoretical basis.

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Abbreviations and Acronyms

RSM = Response Surface Design
CCRD = Central Composite Rotatable Design
WAI = Water Absorption Index
WSI = Water Solubility Index
DI = Dispersion Index
SSA = Sub Saharan Africa
AfricaRice = Africa Rice Centre
BRT = Barrel Temperature
FMC = Feed Moisture Content
FSC = Feed Soybean Composition
NCRI = National Cereals Research Institute, Badeggi