






Optimization of Extrusion Process Using Response Surface Methodology for Producing Squid and Millet Based Extrudate

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Abstract

The squid is underutilized seafood than finfish and shellfishes in domestic consumption and millet is also an underutilized food grain. This study aimed to produce squid and millet- based extrudate under the optimized process parameters using Response Surface Methodology (RSM). The effects of process parameters such as heater 1 temperature (55°C, 60°C and 65°C), heater 2 temperature (120°C, 125°C and 130°C), screw speed (250 rpm, 275 rpm and 300 rpm), and inclusion of squid powder (3%, 4%, 5%, 6% and 7%) were tested for responses like bulk density, expansion ratio and fifty product weight in RSM. All process parameters tested had significant ($P < 0.05$) impact on responses. The optimized parameters were found to be 64.10°C of heater 1 temperature, 129.98°C of heater 2 temperature, screw speed of 317.87 rpm, and 6.56% of inclusion of squid powder.

Introduction

Snack has many definitions, Sajilata and Singhal (2005) defined a snack as a small food meal taken in between a regular meal which includes different types of products that may be in forms served as fries, baked confectionaries, pops, vegetable chips, puffs, roasted nuts, and extruded snacks. The snack food industry grows rapidly with a Compound Annual Growth Rate (CAGR) at 7.5% annually and 89% of snacks available in the market are non-extruded snack products. Snacks are appreciated by people of all ages, mainly children and teenagers; there is an appeal for the nutritional improvement of this product reported by Ferreira

(2006). The extruded snack products previously were made up of cereal which consists predominantly of high carbohydrate and low protein. In the later part incorporation of meat with cereals was done to produce extrudates which led to the incorporation of fish meat powder in extrusion. Yurjew et al. (1989) stated that the addition of meat to starch ingredients causes thermal instability due to interactions of protein and starch. There were studies carried out by in extrusion technology by incorporating fish meat and fish meat based products to produce extruded snack. Wiancki (2007) reported that the use of proper fish such as lean fish over fatty fish and fresh fish over frozen fish had better extrusion properties. Choudhury and Gautam

(2003) reported the use of protein sources in extrusion causes a low expansion ratio and increase in hardness of extrudate which leads to limited success in producing fish-based extrudate products commercially. Since the principle of extrusion technology is High-Temperature Short Time (HTST) there will be minimal loss of nutrients. The extrusion process parameters have a significant impact on the properties of an extrudate. Extrusion process parameters should be optimized to obtain an extrudate.

The objective of this study was to determine the optimized extrusion process parameters to produce squid and millet-based extrudate snack products.

Materials and Methods

This study was conducted at Metro Fish Processing Facility of College of Fisheries Engineering, TNJFU, Nagapattinam, Tamil Nadu, India.

Materials

The Indian squid (*Uroteuthis duvauceli*) was purchased from a local fish market and cleaned, cold blanched at 5% of brine solution, steamed in steam cooker for 15 to 20 minutes, and dried in cabinet drier in 50°C to 60°C for producing squid powder. Corn flour, finger millet flour, pearl millet flour, and salt were purchased from the local market.

Proximate Composition

The moisture content of the extruded snack was determined by the standard AOAC method (2006) by placing the sample in a hot air oven at 105°C for 5 hours. The nitrogen content of extruded snacks was determined by the Micro Kjeldahl method and multiplying by 6.25 with nitrogen value produces the protein content of extrudate (AOAC, 1995). The crude fat content was determined by the Soxhlet method (AOAC, 1995) using petroleum ether (60°-80°C) as solvent. The ash content of the extrudate was determined by placing the sample in a muffle furnace for 24 hours at temperatures ranging from 500°C to 600°C; the crucible was then cooled in a desiccator, and content in the crucible was weighed (AOAC, 1995).

Extrusion Cooking

Extrusion was done using a twin-screw extruder (Basics Pvt. Ltd, Kolkata, India). The extruder had two heaters for cooking. The temperature of heater 1 and heater 2 and the speed of twin-screw, feeder, and rotatory cutting blade can be controlled in the extruder. The Finger millet flour at 10% and pearl millet flour at 10% are mixed, blended with corn flour (77%, 76%, 75%, 74%, and 73%) and squid powder (3%, 4%, 5%, 6%, and 7%) with 6% water, sieved for uniform distribution and left for moisture equilibration for an hour.

Determination of Expansion Ratio

The expansion ratio of extrudate samples was calculated by measuring the diameter of extrudate using a digital vernier caliper (Mitutoyo, Japan). The expansion ratio of the extrudate sample was determined as the diameter of the extrudate to the diameter of the die or orifice (Korkerd, et al, 2016).

$$\text{Expansion ratio} = \frac{\text{Diameter of extrudate}}{\text{Diameter of die or orifice}}$$

Determination of Bulk Density

The diameters of extrudate samples were measured with a digital vernier caliper (Mitutoyo, Japan), from which the average radius of the extruded snacks was calculated. The shape of the extrudate is assumed to be cylindrical. The total volume is calculated by adding up the length of 20 extrudate samples. The bulk density was determined by the average weight of extrudate to the volume of extrudate samples (Case, et al. 1992).

$$\text{Bulk density (g/cm}^3\text{)} = \frac{\text{Weight}}{\text{Volume of extrudate samples}}$$

Determination of 50 Product Weight

Fifty product weight of the sample was done by measuring the weight of randomly selected 50 extruded products using a digital weighing balance (Ishtaa, India). Case et al. (1992) stated that the extrudate low fifty product weight tends to have more acceptability.

Experimental Design and Statistical Analysis

The Response Surface Methodology was used for determining the extrusion process parameters heater 1 temperature (55°C, 60°C, and 65°C), heater 2 temperature (120°C, 125°C, and 130°C), screw speed (250 rpm, 275 rpm, and 300rpm) and inclusion of squid powder (3%, 4%, 5%, 6%, and 7%) towards bulk density, expansion ratio, and fifty product weight as responses or variables in the Box-Behnken model (Box and Behnken, 1960). R² and lack of fit test determined for testing the reliability. The surface plots were done based on the effect of two variables over a response for optimization. These studies were carried on Design Expert 11.1.2.0 (STAT – EASE, Inc., USA). Twenty-four runs were conducted based on RSM.

Results and Discussion

Proximate Composition of Raw Materials

The proximate compositions of raw materials were given in Table 1. The crude protein was found to be 84.19±0.27% in squid powder, 7.92±0.22% in finger

millet flour, and 10.34±0.22% in pearl millet flour. Crude fat was found to be 1.27±0.33% in squid powder, 2.53±0.11% in finger millet flour, and 4.83±0.09% in pearl millet flour. The optimized squid and millet-based extrudate was found to have 19.66±0.26% crude protein, 1.98±0.11% lipid, 2.64±0.09% ash, and 5.73±0.11% moisture content. From the proximate composition of raw materials and extrudate, it can be interpreted that crude protein was contributed by the inclusion of squid powder and extrusion cooking removes moisture from extrudate which was blended mixture with additional water added at 6% level before extrusion. Goes et al. (2015) observed 9% addition of different fish powders increased protein content 11.85±0.21% in Tuna added extrudate, 9.80±0.49 % in Tilapia added extrudate and 9.21±0.17% in Salmon added extrudate. Osman et al. (2017) reported that the shrimp-corn extrudate had a crude protein of 18.8%.

Effect of Process Variables

The effect of process parameters over the expansion ratio is given in Table 2. The expansion ratio

varied from 3.8611 to 4.603. The highest expansion ratio was found at 60°C of heater 1 temperature, 130°C of heater 2 temperature, 300 rpm of screw speed, and 5% of squid powder inclusion. Lue et al. (1990) stated that optimized expansion ratio is an important parameter for obtaining better sensory quality. Faller et al. (1998) reported that there is a negative relationship between the expansion ratio and hardness of extrudates. Rehrh et al. (2009) also observed a similar negative relationship between the expansion ratio and protein content of the extrudate.

The effect of process parameters over the bulk density is given in Table 2. The bulk density varied from 1.3543 g/cm³ to 3.4466 g/cm³. The highest bulk density was found at 60°C of heater 1 temperature, 125°C of heater 2 temperature, 275 rpm of screw speed and 5% of squid powder inclusion. Ding et al. (2020) stated that bulk density plays a major role in packaging and transportation, the higher the bulk density the lower the transportation and packaging cost; higher bulk density leads to less volume of space occupied.

The effect of process parameters over fifty product weight is given in Table 2. The fifty product weight varied

Table 1. Proximate composition of raw materials

Sl.No.	Raw material	Moisture (%)	Protein (%)	Lipid (%)	Ash (%)
1.	Squid powder	6.77±0.11	84.19±0.27	1.27±0.33	3.34±0.28
2.	Finger millet flour	5.24±0.29	7.92±0.22	2.53±0.11	1.33±0.11
3.	Pearl millet flour	6.71±0.25	10.34±0.22	4.83±0.09	1.47±0.18
4.	Extrudate	5.73±0.11	19.66±0.26	1.98±0.11	2.64±0.09

Table 2. Effect of process parameters on responses

Run	Factor 1 A:Heater 1 °C	Factor 2 B:Heater 2 °C	Factor 3 C:Screw speed Rpm	Factor 4 D:Squid powder %	Response 1 50 Product weight G	Response 2 Expansion Ratio	Response 3 Bulk Density g/cm ³
1	60	125	275	3	11*	4.108	1.7153
2	60	125	275	5	12	4.125	1.9319
3	60	125	275	6	12	4.189	1.9289
4	55	120	275	5	11*	4.2701	1.6322
5	65	120	275	5	13	4.451	2.49
6	60	125	275	7	12	4.157	1.9304
7	65	130	275	5	13	4.4384	1.5328
8	55	130	275	5	13	4.3923	2.5192
9	60	130	300	5	16**	4.603**	1.8911
10	60	125	275	4	12	4.165	1.932
11	60	130	250	5	11*	4.221	2.5637
12	55	125	250	5	12	4.3004	2.0268
13	60	125	275	5	13	4.133	1.3543*
14	65	125	300	5	13	4.563	1.8113
15	60	120	300	5	14	4.4366	2.5887
16	55	125	300	5	15	3.8611*	3.4466*
17	65	125	250	5	11*	4.2519	1.9591
18	60	120	250	5	11*	4.1861	1.8524
19	65	125	300	5	14	4.465	1.9005
20	60	120	300	5	15	4.549	2.4751
21	60	125	250	3	11	4.1924	2.0124
22	65	125	275	3	12	4.1145	1.8546
23	60	125	300	7	14	4.4265	1.9234
24	55	120	275	5	12	4.1971	1.6572

* represents the lowest value, and ** represents the highest value.

from 11 g to 16 g. The highest fifty product weight was found at 60°C of heater 1 temperature, 130°C of heater 2 temperature, 300 rpm of screw speed, and 5% of squid powder inclusion.

From the ANOVA (Table 3, Table 4 and Table 5), it was observed that the extrusion process parameters were found significant ($P \leq 0.05$) in overall responses. The lack of fit test was determined to be insignificant ($P \geq 0.05$) in responses. R^2 value was found to be 0.8182 in expansion ratio, 0.8085 in bulk density, and 0.8649 in fifty product weight. From the significance test, it can be observed the extrusion parameters and percentage of squid powder inclusion had an impact on the expansion ratio, bulk density, and fifty product weight.

Optimization of Extrusion Process Parameters Using RSM

The relationship between process parameters (Independent variables) and responses (Dependent variables) are shown in Figure 1, Figure 2, and Figure 3. The optimum process parameters are indicated in Figure 4. For better responses value, the optimum process parameters have been arrived at from RSM. The responses from the predicted values can be verified if the response values from the optimum process parameter are closer to the predicted value. The optimum process parameters were determined as 64.10°C heater 1 temperature, 129.98°C heater 2

Table 3. Analysis of variance and Regression coefficient for expansion ratio on quadratic model.

Source	Sum of square	df	Mean Square	F- value	P- value	
Model	0.6108	13	0.0470	3.46	0.0281	Significant
A-Heater 1	0.2251	1	0.2251	16.58	0.0022	
B-Heater 2	0.0019	1	0.0019	0.1364	0.7196	
C-Screw speed	0.0160	1	0.0160	1.18	0.3030	
D-Squid powder	0.0171	1	0.0171	1.26	0.2884	
AB	0.0070	1	0.0070	0.5137	0.4899	
AC	0.1270	1	0.1270	9.36	0.0121	
AD	0.0071	1	0.0071	0.5241	0.4857	
BC	0.0001	1	0.0001	0.0039	0.9517	
BD	0.0000	0				
CD	0.0063	1	0.0063	0.2574	0.6229	
A ²	0.0041	1	0.0041	0.3002	0.5958	
B ²	0.1661	1	0.1661	12.24	0.0057	
C ²	0.0064	1	0.0064	0.4682	0.5094	
D ²	0.0001	1	0.0001	0.0097	0.9237	
Lack of Fit	0.1219	6	0.0203	5.88	0.0540	Not significant
R ²	0.8182					
Adjusted R ²	0.5818					

Table 4. Analysis of variance and Regression coefficient for bulk density on quadratic model

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	3.68	13	0.2832	3.25	0.0345	Significant
A-Heater 1	1.14	1	1.14	13.04	0.0048	
B-Heater 2	0.3895	1	0.3895	4.47	0.0607	
C-Screw speed	0.6810	1	0.6810	7.81	0.0190	
D-Squid powder	0.0556	1	0.0556	0.6375	0.4432	
AB	1.08	1	1.08	12.39	0.0055	
AC	0.6750	1	0.6750	7.74	0.0194	
AD	0.0002	1	0.0002	0.0021	0.9644	
BC	0.4747	1	0.4747	5.44	0.0418	
BD	0.0000	0				
CD	0.0525	1	0.0525	0.6025	0.4556	
A ²	0.1696	1	0.1696	1.94	0.1934	
B ²	0.0189	1	0.0189	0.2173	0.6511	
C ²	0.5545	1	0.5545	6.36	0.0303	
D ²	0.0120	1	0.0120	0.1374	0.7186	
Lack of Fit	0.6946	6	0.1158	2.61	0.01863	Not significant
R ²	0.8085					
Adjusted R ²	0.5594					

Table 5. Analysis of variance and Regression coefficient for 50 product weight on quadratic model

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	41.19	13	3.17	4.93	0.0081	Significant
A-Heater 1	0.5046	1	0.5046	0.7846	0.3965	
B-Heater 2	1.25	1	1.25	1.95	0.1931	
C-Screw speed	4.65	1	4.65	7.23	0.0228	
D-Squid powder	0.2848	1	0.2848	0.4428	0.5208	
AB	0.9941	1	0.9941	1.55	0.2421	
AC	0.2947	1	0.2947	0.4582	0.5138	
AD	0.4619	1	0.4619	0.7181	0.4166	
BC	0.4084	1	0.4084	0.6350	0.4440	
BD	0.0000	0				
CD	0.2137	1	0.2137	0.3322	0.5771	
A ²	0.0954	1	0.0954	0.1483	0.7083	
B ²	0.3818	1	0.3818	0.5936	0.4589	
C ²	1.09	1	1.09	1.70	0.2220	
D ²	0.9231	1	0.9231	1.44	0.2585	
Lack of Fit	4.43	6	0.7386	1.48	0.3677	Not significant
R ²	0.8649					
Adjusted R ²	0.6894					

temperature, 317.87 rpm screw speed, and 6.56% inclusion of squid powder. The predicted values of response for optimized extrudate should be 16.5879 g of fifty product weight, 1.326 g/cm³ of bulk density, and 5.19665 of expansion ratio. The obtained response values of the optimized extrudate were 16.524 g of fifty product weight, 1.319 g/cm³ of bulk density, and 5.108 of expansion ratio. In this study, the response values from the optimum process parameters were found closer to the predicted values reflect the degree of perfection of the optimization process.

Nutritional and Sensory Assessment of Optimized Extruded Snack

The amino acid composition, and fatty acid composition of optimized squid and millet snack were analyzed. The essential amino acid such as histidine, threonine, lysine, tryptophan, methionine, valine, isoleucine, leucine and phenylalanine were found to be 0.345, 0.480, 0.418, 0.578, 0.603, 0.957, 0.477, 0.947 and 0.581 mg/100 mg respectively in squid extrudate. Omana et al. (2010) determined amino acid profile of 10% ribbon fish and rice based extruded snack 0.1250, 0.1016, 0.1230, 0.0816, 0.0839, 0.1686, 0.1123, 0.2488 and 0.1493 g/100g of histidine, threonine, lysine, tryptophan, methionine, valine, isoleucine, leucine and phenylalanine respectively. The essential fatty acid such linolenic acid, eicosapentaenoic acid, docosahexaenoic acid and arachidonic acid were found to be 0.98%, 0.42%, 0.98% and 0.18% respectively of total fatty acid present in optimized extrudate. Ganesan et al. (2017) found that EPA content of 0.062% in sardine fish extrudate and 0.33% in lizard fish extrudate and DHA content of 0.053% in sardine fish extrudate and DHA was not detected in Lizard fish extrudate.

The sensory analysis of optimized squid and millet extrudate was analyzed by panel consisting of 30 semi trained people for analyzing the sensory properties such

as appearance, colour, taste, texture, and overall acceptance. The sensory assessment was done in scale of 1 to 5 where 5 was the maximum scale of preference/like. The appearance of optimized squid and millet extruded snack was 4.38±0.05, colour was assessed as 4.56±0.04, taste was assessed as 4.46±0.05, texture was found to be 4.52±0.04, and overall acceptance was found as 4.35±0.04. Jeyakumari and Rathnakumar (2006) stated that addition of high protein source affects the hardness and crispiness of the extrudate due to interaction of protein and starch. It was also found in the present study that the increase in inclusion of squid powder resulted in less crispiness.

Conclusion

In this study squid and millet-based extrudate was produced by optimizing extrusion process parameters and percentage inclusion of squid powder. The optimization of the extrusion process parameter added value to the extrudate. The extrusion process parameters such as heater1 temperature, heater 2 temperature, screw speed, and percentage of squid powder inclusion had a significant ($P \leq 0.05$) impact on bulk density, expansion ratio, and fifty product weight. The optimum process parameter was determined as 64.10°C of heater 1 temperature, 129.98°C of heater 2 temperature, screw speed of 317.87 rpm, and 6.56% of inclusion of squid powder.

Ethical Statement

Not applicable.

Funding Information

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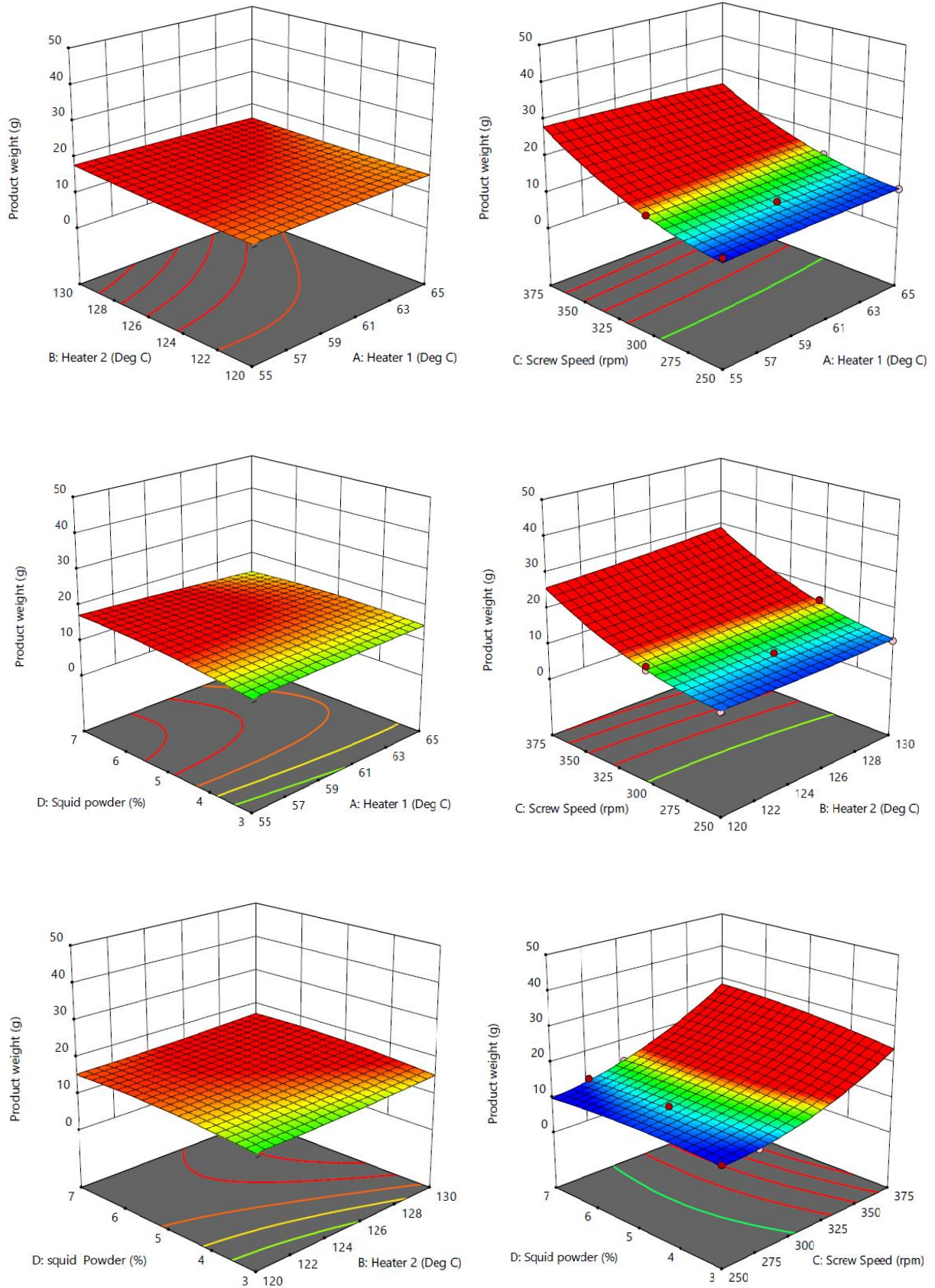


Figure 1. Effect of variables upon 50 product weight response on surface plots.

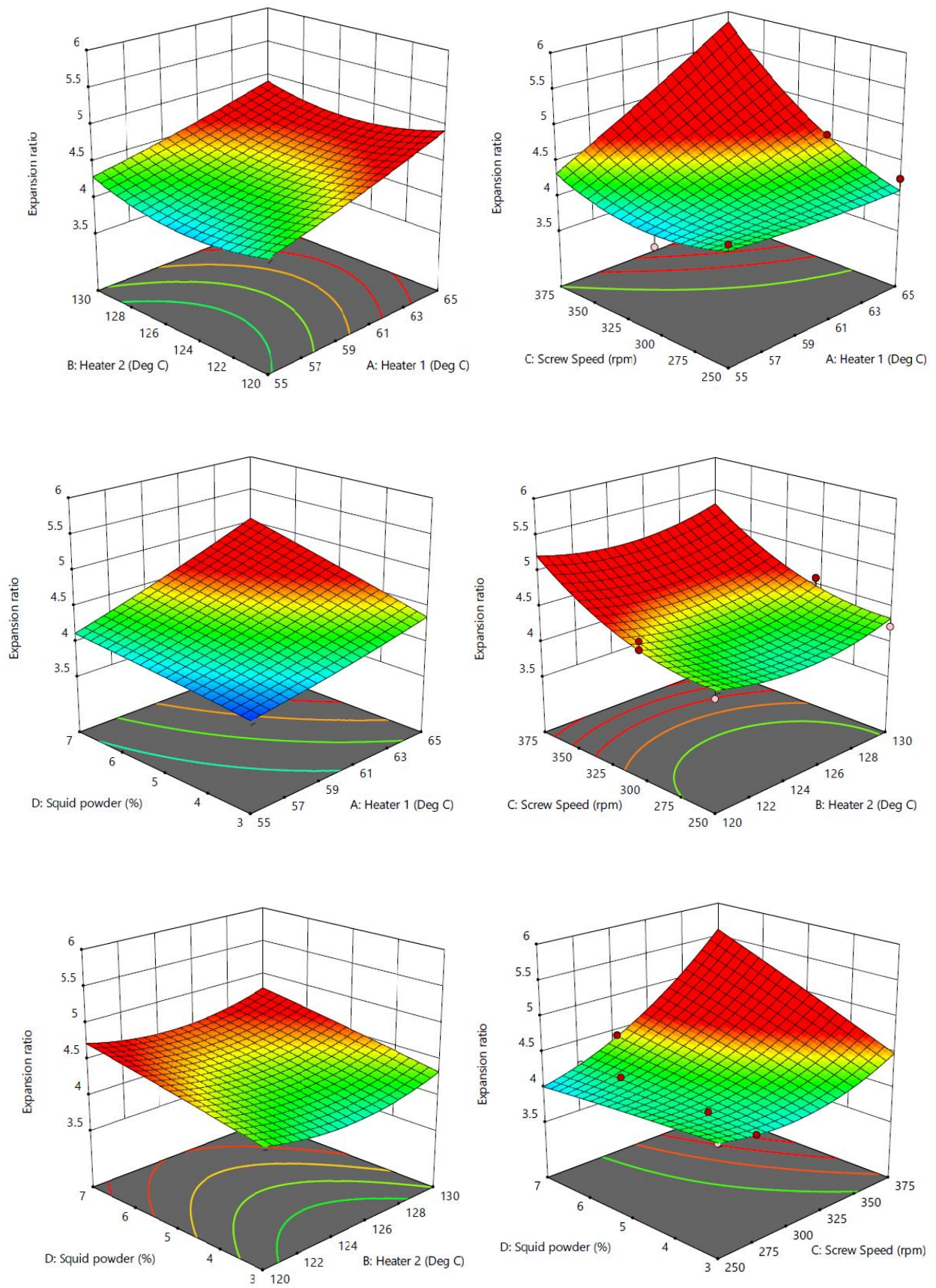


Figure 2. Effect of variables upon expansion ratio response on surface plots.

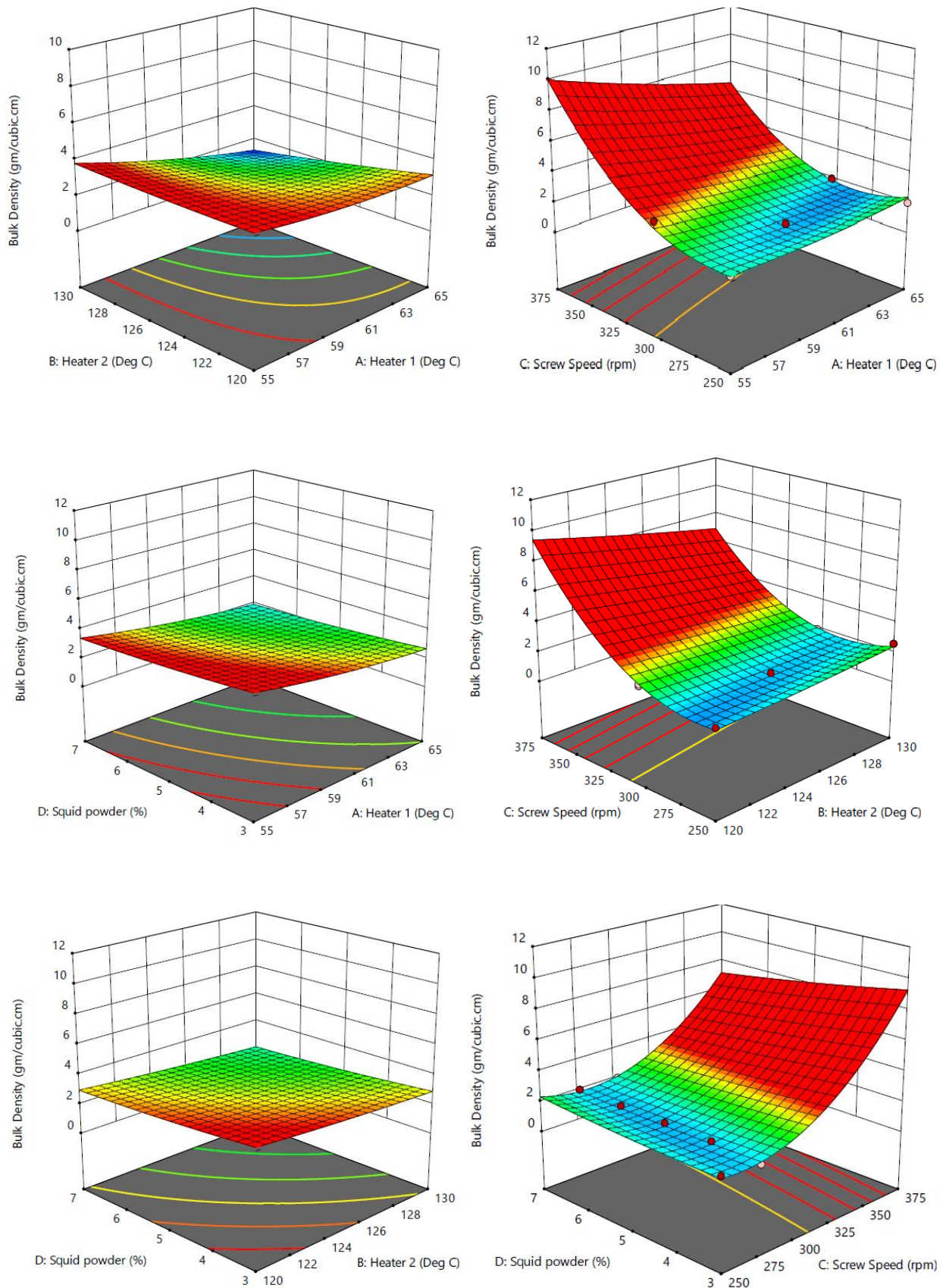


Figure 3. Effect of variables upon expansion ratio response on surface plots.

Design-Expert® Software

Factor Coding: Actual

All Responses**Actual Factors**

A: Heater 1 = 64.1003
 B: Heater 2 = 129.988
 C: Screw Speed = 317.874
 D: Squid Powder = 6.56802

Responses

Desirability = 1
 Product weight (g) = 16.5879
 Expansion ratio = 5.19665
 Bulk Density (gm/cubic.cm) = 1.32643

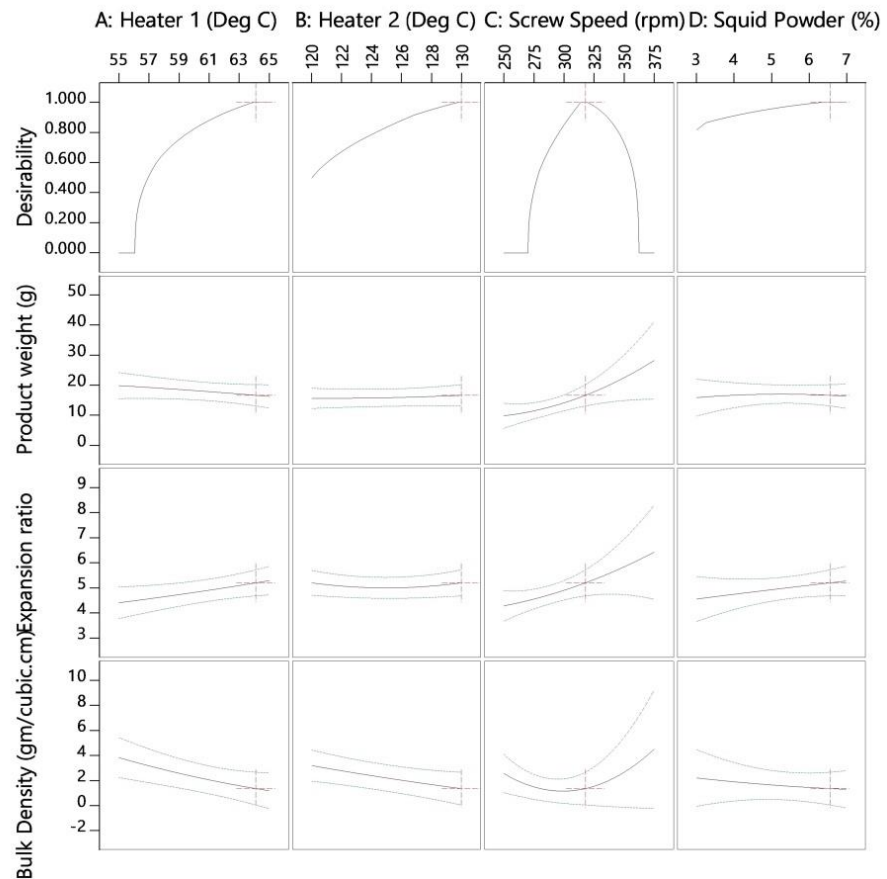


Figure 4. Optimum process parameter overlay graph.

Author Contribution

First Author: Formal Analysis, Investigation, Methodology, Visualization and Writing -original draft; Second Author: Data Curation and Writing -review and editing; Third Author: writing – review and editing; Fourth Author: Writing review and editing; Fifth Author: Conceptualization, supervision and Sixth Author: Methodology and analysis.

Conflict of Interest

The author(s) declare that they have no known competing financial or non-financial, professional, or personal conflicts that could have appeared to influence the work reported in this paper.

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