

## DISTANCE BASED RESPONSE SURFACE METHODOLOGY AND EXTRUSION PROCESS OPTIMIZATION FOR DEVELOPMENT OF GLUTEN FREE SNACK

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### Abstract

*Extruded snack products are the most abundantly consumed products amongst the world as they are cheaper, easily available and have great tastes. Gluten free extruded snacks are not abundantly present in the market. This study will definitely augment and give a lift to the gluten free snack producing industries. Present study deals effect of extrusion parameters (die temperature and feed moisture) on expansion, density, color (L value), and hardness, water solubility index (WSI) and water absorption index (WAI) of gluten free extruded snack consisting of rice, sorghum, unripe banana, water chestnut and moong flour. Distance based response surface methodology was used. Expansion was increased with increasing die temperature at high moisture levels. Density increased with moisture at low temperature and vice versa. L value was positively affected at lower temperature and moisture however, the product obtained was dark in color due to Maillard reaction at higher temperature. Hardness was decreased with increasing die temperature at high moisture. While WSI was not found to be significantly affected by these two parameters, WAI was observed to be increasing with increasing moisture followed by a decrease in its value. Incorporation of extruded flour improved gluten free dough and flatbread properties also found to enhance sensory acceptability.*

**Keywords:** Extrusion; gluten free snack; distance based method; RSM; PCA

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## 1. INTRODUCTION

Extruded snack products are the most abundantly consumed products amongst the world as they are cheaper, easily available and have great tastes. These products are well liked as they are characterized due to their ease of storage, handling. Hence, they are demonstrated as a principle model which helps enhance the dietary characteristics (Gatet al., 2015<sup>1</sup>).

Extrusion process is characterized by production of a variety of food stuffs with diverse shapes, textures, flavor, color and other parameters. Riaz (Riaz., 2012<sup>2</sup>) has mentioned miscellaneous products like snacks, breakfast cereals, pasta, instant foods, texturized vegetable proteins and variety of other products that can be exploited for a range of other purposes can be produced with the help of extruder.

Extrusion is a continuous high temperature short time (HTST) food processing techniques. A screw and barrel mechanism is operated to achieve the process. Several chemical

modifications and structural changes take place during extrusion process, chemical modifications and structural changes occurs in the raw materials, such as starch gelatinization, protein denaturation, degradation of pigments and vitamins as well as loss of volatile compounds (Camire, 1998<sup>3</sup>). As a result, the new food product the knowledge of changes in extruder operation variables is essential which provides necessary information for the prediction of specific reactions endured by particular fraction of food materials during extrusion process and its possible effects on the characteristics of finished product. Ding et al.(Ding et al., 2005<sup>4</sup>) stated that little change in extrusion variables such as composition of feed, its moisture content, screw geometry and speed, die configuration; feed rate, processing temperature significantly affect quality of finished product. Consequently, it has put a critical demand for food technologists to appropriately and effectively optimize production variables if extrusion technology is to be implemented for the manufacture of product.

Although it is observed that extruded cereals and snacks have gained a huge market; the gluten free extruded snacks are limited in the market shelves. And hence, efforts have been carried out in the present study relating development of extruded snack utilizing extrusion technology.

As reported by Noordin et al (Noordin et al., 2005<sup>5</sup>), response surface methodology (RSM) is an assortment made of arithmetic techniques in addition to statistical procedures that are useful in optimizing the procedures of product development. It provides an ideal tool for scrutinizing and optimizing process and product parameters in food processing industries.

The present work deals with assessment of effect of extrusion cooking parameters like feed moisture and die temperature on properties of gluten free formulated snack. The objectives of this work are to evaluate the influence of extrusion cooking parameters (feed moisture and die temperature) on gluten free formulated extruded snack and to standardize optimum conditions to yield the best quality extrudate using distance-based technique of RSM.

## 2. MATERIALS AND METHODS

### Materials

Wheat (*Triticum aestivum*) flour (Aashirvaad, ITC<sup>TM</sup>, Mumbai, Maharashtra, India), rice (*Oryza sativa*) flour (Bhagirathi<sup>TM</sup>, Mumbai, Maharashtra, India), sorghum (*Sorghum bicolor*) flour (Bhagirathi<sup>TM</sup>, Mumbai, Maharashtra, India), moong (*Vigna radiate*) flour (Swad<sup>TM</sup>, Mumbai, Maharashtra, India), water chestnut (*Trapa natans*) flour (Swad<sup>TM</sup>, Mumbai, Maharashtra, India) were purchased from local market of Mumbai. Unripe banana (*Musa paradisiaca*) flour (Mahila Gruh Udyog<sup>TM</sup>, Jalgaon, India) was purchased from Jalgaon banana market. All the flours were sieved (60 mesh) and then used for analysis. Whey proteins (Royal Ingredients, Mumbai, Maharashtra, India<sup>TM</sup>) were also purchased. Additives (guar gum, xanthan gum and

glycerol monostearate) were gifted by Royal Ingredients, Mumbai, Maharashtra, India.

### Sample preparation

Gluten free formulation consisted of rice (60%), sorghum (10%), unripe banana (5%), water chestnut (15%) and moong flour (10%). Moisture content was estimated using oven method [6] and samples were prepared according to method explained by Gat and Ananthanarayan (Gat et al., 2015<sup>1</sup>).

### Extrusion

Extrusion was carried out as described by Gat and Ananthanarayan (Gat et al<sup>1</sup>). A laboratory-scale-co-rotating twin-screw extruder (KETSE 20/40 Brabender GmbH and Co. KG, Duisburg, Germany) was used for extrusion. It has barrel length to diameter ratio of 20:1. Extrusion conditions were feed moisture 16% and die temperature 160°C.

### Experimental Design

Distance based design of RSM was employed which involved use of two variables at five different stages with six central points. Feed moisture (A) and die temperature (B) were independent. Table 1 shows coded levels for these variables. Extrudate characteristics (overall expansion, bulk density, *L* value, WSI, WAI and hardness) were response variables studied.

### Expansion ratio

Expansion was expressed as articulated by Ding et al (Ding et al<sup>4</sup>) as sectional expansion i.e., ratio of extrudate diameter to the die diameter. Average of ten arbitrary readings was reported as the mean diameter of the extrudate. Die has 4 mm diameter.

### Density

Estimation of density of extrudate was done by method of Alvarez-Martinez et al (Alvarez-Martinez et al<sup>7</sup>). Mean of ten arbitrary readings were expressed as extrudate density.

### *L* value

Hunter Lab Colorimeter (Labscan XE) was employed to measure the color of. *L* value indicates extent of light to dark color characteristic (Francis<sup>8</sup>). Results were tabulated as median of three readings.

**Water solubility index (WSI) and water absorption index (WAI)**

Method of Ding et al (Ding et al<sup>4</sup>) was performed to determine WSI and WAI. Method involves grinding of extrudates to pass through 30 mesh sieve followed by suspending in water along with intermediate stirring. Further centrifugation and evaporation of supernatant was done. WSI and WAI were calculated as explained by above stated author. Mean of three replicates were reported as WSI and WAI.

**Texture**

Gluten free extrudates were analyzed for hardness by the method explained by Ding et al (Ding et al<sup>4</sup>) using 2 mm cylindrical probe. Texture analyzer from Stable Micro System TA-XT2i (Serial No. 4650, TEE version 2.64 UK) was employed for the purpose. Hardness of the extrudate was determined using 2 mm cylindrical probe. Maximum Mean of ten arbitrary readings were expressed as hardness value.

**Statistical analysis**

Design Expert 6.0 (StatEase Inc., Minneapolis, USA) was used to analyze the results and to produce response surface plots. IBM SPSS

statistics was took up to obtain Pearson's correlation coefficient matrix was generated using SPSS. Principal component analysis of data was done using STATISTICA 7.

**3.RESULTS AND DISCUSSION**

**Diagnostic checking of fitted models**

All main, linear, quadratic and interactive effects were calculated for each model. The adequacy of the models was tested using F-ratio and coefficient of determination (R<sup>2</sup>). The models were considered adequate when the calculated F-value was more than table F-value (at 5% level) and R<sup>2</sup> value was more than 80% (Henika<sup>9</sup>). The calculated F-value was also more than the table F-value (at 5% level) for all the responses, indicating the adequacy of the models. All the responses showed values of "Prob (probability) > F" less than 0.05 indicating model terms were significant as shown in table 2. However, most of the models statistically showed their adequacy to describe the effect of variables on the responses, though for getting optimum combination of variables, mapping of the more response plots on each other is difficult.

**Table 1. Coded levels of independent variables used in developing gluten free RTE extruded snack**

Factors	Codes	Levels		
		-1	0	1
Feed moisture (%)	A	12	16	20
Die temperature (°C)	B	140	160	180

**Table 2 Analysis of variance statistics of model terms of mixture design for optimization of gluten free extruded snack**

Parameters	Prob> F	CV	R squared	Adj R squared	Pred R squared	Lack of fit
<b>Expansion</b>	0.0008	6.23	0.90	0.83	0.74	0.79
<b>Density</b>	< 0.0001	8.7	0.98	0.97	0.96	0.63
<b>L value</b>	0.0026	0.83	0.86	0.77	0.55	0.05
<b>Hardness</b>	0.0015	10.9	0.88	0.80	0.61	0.10
<b>WSI</b>	0.0006	2.33	0.91	0.84	0.72	0.76
<b>WAI</b>	< 0.0001	1.95	0.95	0.92	0.86	0.36

(Prob : Probability, WSI : Water soluble index, WAI : Water absorption index)

**Expansion**

The relation between expansion and extrusion parameters (feed moisture and die temperature) is given by following regression equation;

$$\text{Expansion} = 2.11+0.14A-0.44B+0.12AB-0.15A^2+0.11B^2 \dots\dots\dots(\text{Eq1})$$

Figure 1 elaborates the behavior of characteristics of extrudates with respect to feed moisture and die temperature.

Figure 1.a shows expansion behavior of gluten free extrudates as a function of feed moisture and die temperature. As can be seen from the figure, increase in feed moisture has led to an increase in overall expansion of the extrudates. Increase in die temperature increased expansion followed by a slight decrease in expansion value of the gluten free extrudates. These observations are in agreement with the work reported in the literature (Meng et al<sup>10</sup>). The expansion was found to be highest at 16% feed moisture and 160° C die temperature.

Pressure difference between the barrel before die and outside atmosphere is responsible for expansion of extruded food material. Low moisture foods tend to be more viscous than foods with higher moisture. It leads to lower the pressure difference for higher moisture foods which yields a less expanded product. Singh et al(Singh et al<sup>11</sup>) reported that feed with low moisture content are inclined to increase the shearing rate and residence time which is beneficial in terms of higher degrees of gelatinization and expansion.

**Density**

Density is a chief material property of the extrudate which along with expansion indicates the puffing extent of extrudates. It is inversely proportion to the expansion ratio.

Figure 1.b represents density of extrudate as a function of feed moisture and die temperature As can be seen from the figure, bulk density of gluten free extrudate increased from 0.20 to 1.1 g/cm<sup>3</sup> with increase in moisture content from 12 to 20%, while increasing die temperature 140 to 180 °C bulk density was lowered initially giving lowest density at the optimum temperature followed by increase in its value. This range of bulk density from 0.20 to 1.1

g/cm<sup>3</sup> correlates well with a decrease in expansion ratio from 1.565 to 2.7. Generally product having low bulk density is desirable. Ding et al (Ding et al<sup>4</sup>) has reported temperature to be responsible for the degree of superheating of water in the extruder. Their rise encourages bubble formation and leads to reduced density of the product. He also explained the significant interaction between moisture content, temperature and density. Plasticizing phenomenon may occur as feed moisture increases leading to reduction in elasticity of the dough. Gradually, mechanical energy drops down, gelatinization decreases and so expansion yielding an extrudate with undesirable high density.

The regression equation (Eq.2) for density:

$$\text{Density} = 0.25-0.13A+0.25B-0.12AB+0.18A^2+0.14B^2 \dots\dots\dots(\text{Eq.2})$$

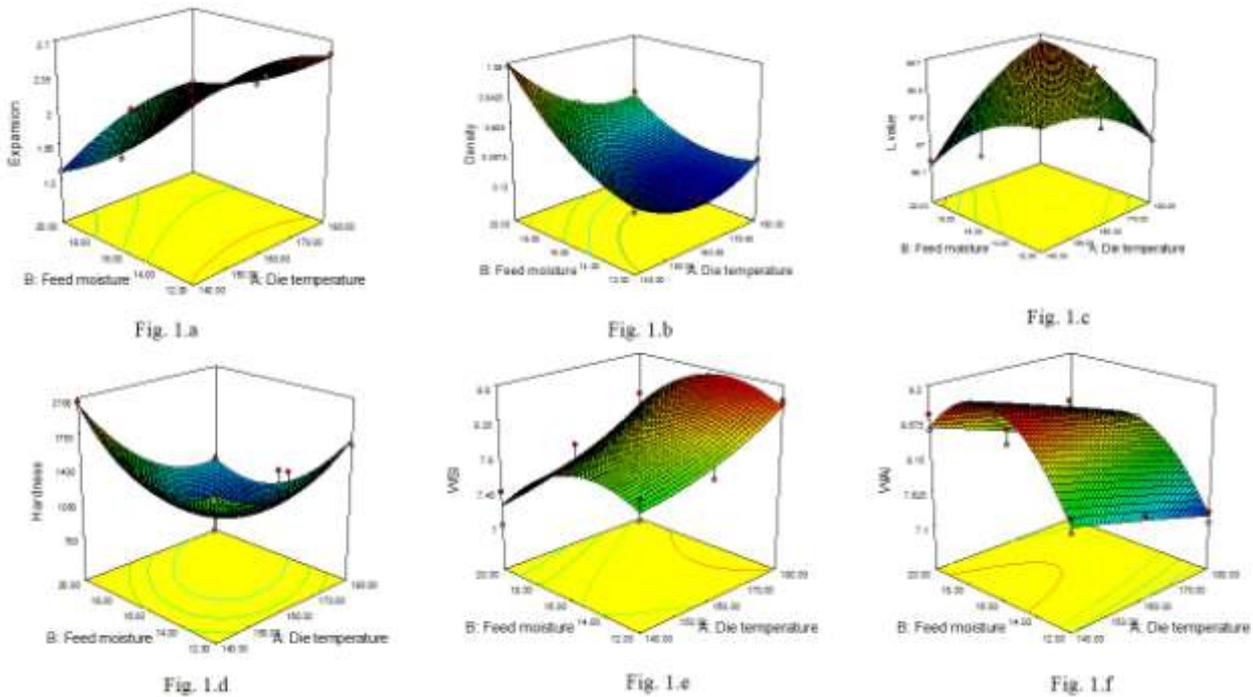
**Color**

As said by Francis (Francis<sup>8</sup>), color is crucial in deciding the acceptability of food products before getting consumed. *L* (luminosity) value indicates the brightness of the product; higher value of which is requisite. The regression equation (Eq. 3) involves:

$$L \text{ value} = 68.96+0.43A-0.024B+1.22AB-0.54A^2-0.59B^2 \dots\dots\dots(\text{Eq.3})$$

As can be seen from figure 1.c, *L* value initially increased and further decreased as feed moisture escalated. Similar behavior was observed with respect to increase in die temperature. This behavior is due to the distribution of material in low density high expanded snacks through the surface which gives rise to the reflection and increases the luminosity of the surface (Gat et al<sup>1</sup>).

Figure 3 clearly illustrates the decrease in *L* value at higher temperatures indicating the formation of darker expanded product. As stated by Chevanan et al (Chevanan et al<sup>12</sup>), color modifications occurring during high temperature processing are due to Maillard reactions and protein denaturation. They have stated color change of the extruded products as a symbol of lysine loss.



**Fig 1. Characteristics of extrudate as a function of feed moisture and die temperature (1.a: expansion ratio, 1.b: density, 1.c: L value, 1.d: hardness, 1.e: WSI, 1.f: WAI)**

**Hardness**

The regression equation (Eq.4) for hardness is given as:

$$\text{Hardness} = 937.79 - 217.27A - 52.48B - 264.24AB + 303.10A^2 + 366.90B^2 \dots \dots \dots \text{(Eq.4)}$$

Figure 1.d explains about the behavior of hardness as a function of feed moisture and die temperature. As can be seen from the figure, a rise in the value of hardness of gluten free extrudate has been observed with the increase in feed moisture as well as die temperature. Extrudate hardness is affected by moisture content of feed. It has shown to be directly proportional to product hardness from previous studies (Seth et al<sup>13</sup>). Higher moisture content decreases the shear of the plasticized mass inside the extruder, reducing the gelatinized starch and prevents the growth of air bubbles of the snacks. This leads to the increase in hardness of the extrudate. Similar effects on hardness had been reported in the previous studies (Singh et al<sup>11</sup>).

At low levels of feed moisture and die temperature, extrudate has high density and low expansion and has a compact structure for the probe to penetrate its surface which gives

rise to increase in hardness. Whereas, highly expanded extrudate has lower densities and are porous in nature exerting low force on the probe to penetrate hence led to decrease in hardness of the extrudate.

**Water solubility index (WSI) and water absorption index (WAI)**

WSI determines the extent starch undergoes conversion during extrusion process. Van den Eijnde et al (Eijnde et al<sup>14</sup>) has explained this conversion phenomenon on starch conversion i.e. gelatinization by the application of water and heat. Figure 1.e elaborates how WSI of the gluten free extrudate varies as a function of feed moisture and die temperature. WSI of the extrudate is affected significantly by both feed moisture and die temperature. Rise in feed moisture was observed to decrease WSI significantly, while it was found to be enhanced by increase in die temperature. These observations are consistent with previous studies (Ding et al<sup>4</sup>, Singh et al<sup>11</sup>). It has been reported that extrusion temperature and feed moisture to be responsible for change in soluble starch (Camire<sup>3</sup>). They found out that as extrusion temperature increased water solubility index increased.

WAI and WSI parameters are generally related to degree of starch degradation (Gomes et al<sup>15</sup>). WAI depend on the presence of relatively intact molecules, which have not lost their ability to bind water. Lower the degree of starch degradation more will be amount these intact molecules and hence higher will be their WAI. Similarly inverse effect is observed with respect to WSI.

Starch swells in contact with excess water and acquires increased volume which is measure as WAI. Figure 1.f shows the behavior of WAI of extrudate under effect of extrusion conditions. Feed moisture and die temperature had a significant effect on WAI. It increased with increasing feed moisture but decreased with die temperature. This decrease was probably due to decomposition or degradation of starch (Altan et al<sup>16</sup>). Similar result was found by Meng et al (Meng et al<sup>10</sup>). Ding et al (Ding et al<sup>4</sup>) also found starch dextrinization to be accountable for decrease in WAI of extrudate.

The regression equations (Eq.5 and 6) relating WSI and WAI with extrusion parameters are represented as:

$$WSI = 7.94-0.035A-0.18B+0.34AB.....(Eq.5)$$

$$WAI = 7.99+0.33A-0.26B.....Eq.6)$$

### Optimisation of independent variables and validation of model

RSM was employed to find out optimum combination of extrusion process parameters (feed moisture and die temperature) for the production of extrudates using base material of composite of gluten free flours (rice, sorghum, unripe banana, water chestnut and mung flour) with a twin-screw extruder. The principle criteria of optimization were selected as elaborated in table 4 for the responses. By using the given criteria, the solution obtained was feed moisture (16%) and die temperature (160°C) which satisfied all constrains. Extrudates were produced using these optimum conditions which yielded the best product in terms of expansion, density, hardness, color, WSI, WAI and color. The measured response values were very close to the predicted values, confirming the adequacy of the models. Also, the validation of the model was reconfirmed by the lower t test values as depicted in table 3. These optimum conditions can be employed to produce extrudate with superior qualities.

**Table 3 Optimization of constraints and validation of model**

Constraints	Goal	Limits		Model Predicted Value	Experimental Value	T test value
		Lower	Upper			
Feed moisture (%)	In range	12	20	15.47	16	-
Die temperature(°C)	In range	140	180	162.42	160	-
Expansion	Maximize	1.37	2.56	2.19	2.56±0.11	0.28
Density (g/cm <sup>3</sup> )	Minimize	0.18	1.05	0.14	0.20±0.06	0.29
L value	Maximize	66.35	69.67	68.97	70.5±1.5	0.29
Hardness (gm)	Minimize	753.45	2064.34	925.46	777.81±23.35	0.29
WSI	Maximize	7.01	8.65	8.41	7.73±0.21	0.25
WAI	Maximize	7.14	8.99	8.62	8.75±0.45	0.29

(WSI : Water solubility index, WAI : Water absorption index)

### Principal Component Analysis (PCA)

PCA enables a considerable reduction in a number of variables studied and understating the structure of there lationship between measuring parameters that give complimentary information. The eigen values for successive factors are displayed on “screeplot” (Figure2.a). As can be seen from the figure, two principal components are accounting for the 100% variation in data (50.16% by PC1 and 49.84 by PC2). Quality results show that the first two principal components, accounting for 100% of the total variability, can be considered as the perfect for

data representation in case of dependent and independeant variables in production of gluten free extrudates.

Figure 8 shows the scatter plot of samples using the first two principal components (PCs) issued from the PCA of the data matrix is obtained. The number of factors retained in the model for proper classification of experimental data were determined by application of Kaiser and Rice’s rule (Otto<sup>17</sup>). This criterion retains only principal components with eigen values > 1. As can be seen, there is a neat separation of the observed samples, according to the assays used.

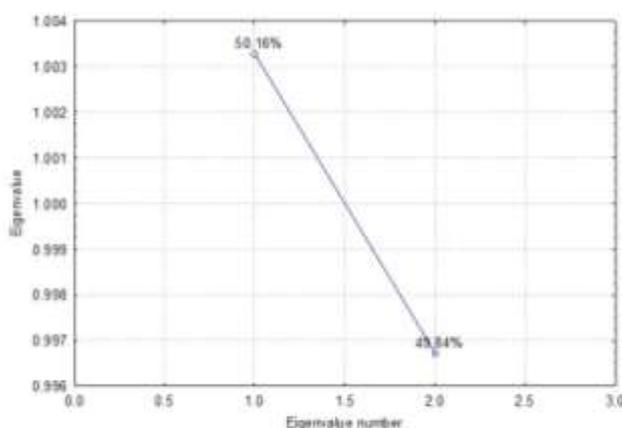


Fig. 2.a

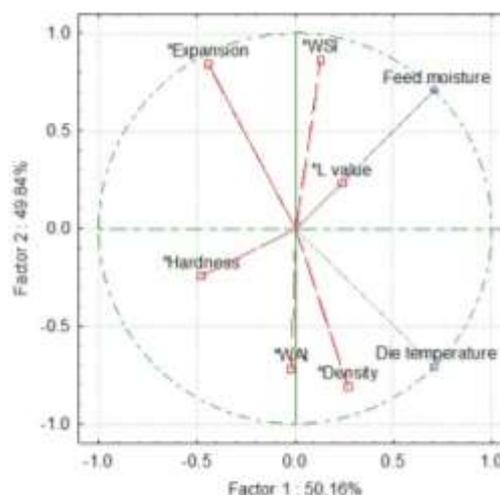


Fig. 2.b

Fig. 2. Principal Component Analysis (2.a: Scree plot, 2.b: Loading plot of dependent and independent variables)

Table 4 Correlation coefficients between system parameters and product responses

	Die temperature	Feed moisture	Expansion	Density	L value	Hardness	WSI	WAI
Die temperature	1	.003	-.091	.800**	.046	-.107	.538*	.057
Feed moisture		1	-.515	-.256	.237	-.569*	-.188	.219
Expansion			1	-.322	.374	-.269	.167	.424
Density				1	-.450	.451	-.585*	-.451
L value					1	-.770**	.531	.630*
Hardness						1	-.103	-.752**
WSI							1	.184
WAI								1

\*\*Correlation is significant at the 0.01 level (2-tailed).

\*Correlation is significant at the 0.05 level (2-tailed).

Figure 2.b demonstrates principal component plot of dependent and independent variables employed in distance based RSM for the production of gluten free extruded snack. The properties which are placed near to each other on the plot were positively correlated while those which are placed in opposite directions were negatively correlated. This analysis showed two axes explaining 100% variability. The first and the second PCs described 50.16 and 49.84% of the variance respectively. Table 4 represents correlation coefficients between system parameters and product responses. It is clear from this table that expansion was positively correlated with *L* value, WAI and WSI (Correlation coefficient 0.305, 0.581 and 0.636 respectively) and a negative correlation occurs with density and hardness (correlation coefficient -0.908 and -0.157 respectively) as shown in table 4. This indicates that extrudate having low density and hardness is well expanded during the process which is obvious as shown by high correlation between product expansion and hardness as well as density. Statistical analysis revealed that *L* value is positively correlated with expansion, WSI and WAI (correlation coefficient 0.305, 0.660 and 0.131 respectively) while correlated in negative with density and hardness (correlation coefficient -0.560 and -0.859 respectively). It was clear from analysis that hardness was negatively correlated (correlation coefficient -0.157, -0.286 and -0.357) with expansion, WSI and WAI respectively, while it was positively correlated (0.471) with density.

The first PC is well characterized by expansion, hardness in the negative quadrant and feed moisture and die temperature in the positive quadrant. The negative correlation between these parameters are well explained by negative correlation coefficients for expansion and hardness with feed moisture and die temperature as shown in table 4. Also, feed moisture being placed in the same quadrant by this PC with *L* value and WSI have positive correlation coefficients (0.237 and 0.538 respectively). Same is the case with die temperature and density positioned very close

to each other has high value of correlation coefficient (0.800) which is highly significant. Second PC splits expansion, WSI and feed moisture from WAI, density and die temperature. Being placed in opposite quadrants negative correlation is explained by table 4 showing negative value of correlation coefficients. WSI and WAI located far apart and opposite to each other are negatively correlated with high value of correlation coefficient (-0.752) which is highly significant.

#### 4. CONCLUSION

Product responses of gluten free formulated extrudate on twin-screw extrusion process were dependent on process variables. Feed moisture and die temperature had significant effect on various extrudate properties, with feed moisture having the greatest influence on the properties of the extrudate. Low density (a desirable characteristic of expanded product) was achieved at medium levels of feed moisture (16 %) and moderate die temperature (160 °C). These results suggest that the physical and functional characteristics of extruded products made of a composite of various gluten free flours can be exploited further in the development of nutritious and healthy extruded products.

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#### Conflicts Of Interest

Authors do not have any conflicts of interest.

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