Predictive equations and response surface analysis for sorghum grain extrudate

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Abstract: The extrusion and extrudate properties measured include expansion indices (or puff indices), bulk density, residence time, solid density, moisture content, maximum stress, water solubility index (WSI), water absorption index (WAI), extruder output, crispness and color. The ranges of the three variables considered were 100 - 160℃ for barrel temperature, 100 - 200rpm for screw speed, and 15 - 25% for feed moisture content (w.b.). The density of extrudate measured varied between 176kg/m^3 and 1100kg/m^3 for the extrusion variables considered. The moisture content of the dry extrudate is between 6% and 7.5% with the mean value of 6.75%. Using the experimental results, predictive equations were developed to relate the influence of processing parameters on the sorghum grain extrudate quality. From the response surface diagram (RSD), it was discovered that the effect of screw speed on density is negligible. Temperature has a significant impact on the density of the extrudate and it has a curvilinear effect on extrudate density and at high temperature, its quadratic effect dominates. The solubility tends to increase linearly with increase in temperature and decreases linearly with increase in moisture content. At temperature below 150℃, the WAI recorded is between 5.1 and 5.3 and it was largely independent of the extrusion moisture content. However, at the temperature higher than 150℃, there is a sharp increase in WAI to a value of six and above. The total specific mechanical energy (SME) varied between 470kJ/kg and 600kJ/kg. SME is directly proportional to screw speed. Comparatively, the screw speed had dominating effects on SME while temperature had a small effect. The sensory evaluation on crispness and color show that the product extruded at the conditioned feed moisture content of 20% and barrel temperature of 150℃ gave the best crispness and color quality.

Keywords: processing parameters, food extrusion, extrudate, quality, sorghum grain

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1 Introduction

Food extrusion is a modern high temperature short time (HTST) cooking process with several unit operations such as a conveying, kneading, heating, mixing and forming in a single unit. Extrusion food processing is widely used to restructure starch and protein based materials to manufacture a variety of ready to eat (RTE) breakfast cereals, pasta, snacks, bread substitutes and pet foods. This process normally involves application of intensive energy to food ingredients at a pressure within a short period of time to form continuous viscous dough.

A proper understanding of how processing parameters affect the thermo-mechanical, rheological and structural transformation of changing food polymers into final product will enhance the development of extrusion cooking technology. The early studies of the extrusion processing of food material mainly focused on the effects of physico-chemical parameters of temperature and extrusion moisture content on product properties (Adekola, 1999). The product properties studied then were color, expansion ratio, bulk density and the viscosity of dilute aqueous dispersion. Subsequent studies showed the importance of process variables that control the mechanical history and residence time of materials such as screw speed, feed rate and die geometry (Adekola et al., 1998).

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Sorghum is produced throughout the tropical, semi-tropical and arid regions of the world. Sorghum is a leading cereal grain and it is mainly used as a principal food in tropical areas of Africa and Asia and often used as raw materials for alcoholic drinks and beverages.

The diversity of sorghum grains is related to their intrinsic properties, which include starch type, non-starch components and phenolic compounds. The latter are genotype dependent which affect the pericarp characteristics such as color and presence of a pigmented testa. This diversity can be valuable for developing new food products by thermoplastic extrusion intended for human consumption (Vargas-Solorzano et al., 2014).

United States is the world largest producer of Sorghum followed by India, Nigeria and Mexico. Sorghum is the only food crop that is reported to contain starch in the anatomical section. The endosperm is a storage organ that is comprised of aleurone layer, peripheral, corneous and floury areas. The aleurone contains proteins, ash and oil. The protein of the germ contains high levels of lysine and tryptophan that are excellent in quality (AAGG, 2010).

Influence of cooking conditions on the product qualities and extrusion have been generally studied and modeled (Adekola, 2015b). In a recent study, two sorghum genotypes (red, tannin; white, non-tannin), were evaluated for their potential use in breakfast cereals. Two levels of whole grain sorghum flour (550 g/kg dry mix or 700g/kg dry mix) were processed per genotype using a pilot-scale, twin screw extruder. A whole grain oat-based cereal was used as a reference. White sorghum cereals (WSC) had significantly ($p<0.05$) higher starch, brightness (L*), and yellowness (b*) than red sorghum cereals (RSC). RSC had higher protein and bulk density than the WSC. Overall acceptability and texture of sorghum cereals did not differ significantly from the oat reference, although appearance and aroma liking were significantly reduced. Therefore, non-tannin sorghum has potential to be used in the breakfast cereal industry with minimal impact on nutritional profile and sensory properties (Mkandawire et al., 2015).

In a similar study, milled barley and sorghum grains were separated into three size fractions (fine, <0. mm; medium, 0.5 - 1mm; coarse, >1mm) and extruded at two maximum temperatures (100 ℃; 140 ℃). Mechanical resistance and specific mechanical energy during extrusion was significantly higher for fine fractions, and extrusion at high temperature resulted in higher mechanical resistance. Pressure generated during extrusion was higher for the fine fraction in sorghum but lower in barley. Expansion index was highest for the fine fraction for barley, but did not differ significantly between sorghum fractions or with extrusion temperature. For all samples, extrusion at low temperature resulted in a higher final paste consistency and lower water absorption index, but there was no significant effect on water solubility index (WSI). Fraction size showed a significant effect on WSI in sorghum but not in barley (Al-Rabadi et al., 2011)

Mathematical modeling and simulation of the effects of extrusion processing parameters on extrudate is becoming more popular for scale-up purposes. In the light of this, two-dimensional flow simulation in food extruder die for intermeshing co-rotating twin-screw extruder was performed by solving Navier-Stokes equation and continuity equation for non-Newtonian fluid using finite element computer package, ANSYS/FLOTRAN (Adekola, 2015a).

The study determined the nature of flow, heat and pressure distribution in the die and to ascertain the effect of screw speed on process parameters such as temperature, pressure and flow rate in the dies (Adekola, 2014). Experimental results obtained for the die geometries statistically correlate with the simulation results.

The studies on effects of process parameters on extrusion efficiency for many grains such as corn, wheat, rice and others are in literature (Mahasukhonthachat et al., 2010, Chakraborty et al., 2011 and Myat et al., 2014). However, there is no reported work on using response surface analysis and predictive equations to relate the processing parameters to sorghum extrudate quality. Therefore, this paper intends to study the effects of processing parameters such as barrel temperature, screw speed and feed moisture content on extrudate quality for sorghum using a twin screw co-rotating food extruder using predictive equation and response surface analysis method.

2 Materials and methods

2.1 Materials

To determine the effects of the processing parameters on the extrudate quality, France-made Clextral BC45 twin-screw extruder (Figure 1) was used. The three processing parameters considered were screw speed, temperature of barrel at the last section and feed moisture content. The feed material used is white grain sorghum flour.

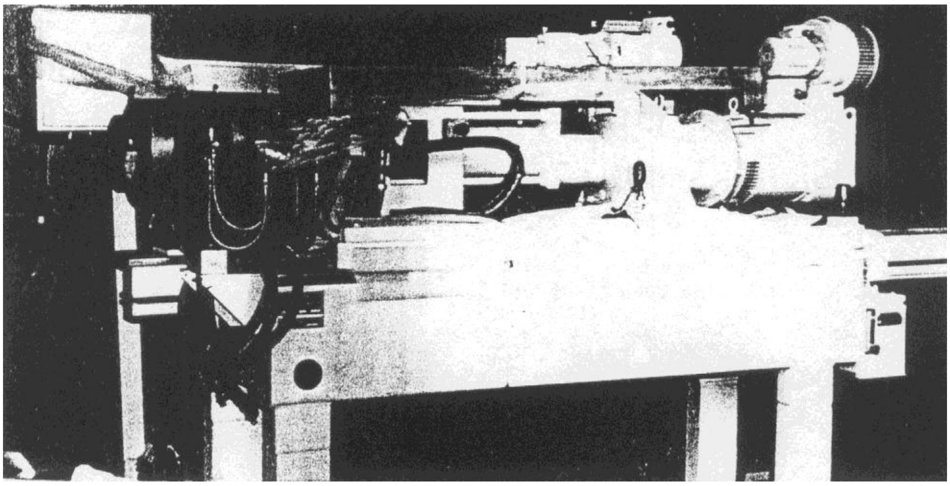

Figure 1 Clextral BC-45 twin-screw extruder

2.2 Experimental design

Based on the review of available literature, a five-level, three-variable, orthogonal central composite rotatable design was used to design the experiment (Myer, 1971). The independent variables are the barrel temperature at the last section, screw speed and feed moisture content. These variables were coded at the levels of -1.682, -1, 0, +1 and 1.682. Table 1 shows the level of variables in the experimental plan.

Table 1 Experimental Design for a Three–variable System

Coded level	Actual level		
x_j (j = 1,2,3)	Temperature, X_1	Screw speed, X_2	Feed Moisture, X_3
	℃	rpm	%
1.682	160	200	25
1	150	185	23
0	130	150	20
-1	110	115	17
-1.682	100	100	15

The total number of experiments is 23. The ranges of independent variables (in actual values) were 100 - 160℃ for barrel temperature, 100 - 200rpm for screw speed, and 15 - 25% for feed moisture content (w.b.). The response function (y) was related to input variable (X) by a second-degree polynomial in Equation 1 below using the method of least squares. The significance of all the terms was determined by the F-test at a probability level 0.01. The significance of the correlation coefficient, r was judged at a probability level of 0.01.

$$y = b_0 + \sum_{i=1}^{n} b_i x_i + \sum_{i=1}^{n}\sum_{j=1}^{n} b_{ij} x_i x_j + \varepsilon \qquad (1)$$

$$i \le j$$

Where n is the number of variables,: i and j are integers,: b_o, b_i and b_{ij} are the coefficients of the polynomial,: ε is the random error.

2.3 Machine and equipment

The experiments carried out in this work made use of the BC45 twin screw co-rotating extruder. It is a modern machine for industrial production. The Clextral Company of France manufactured the extruder. The machine accessories include the feed mixer, the extrudates cutter, the conveying system and the dryer. The barrel is divided into three sections, namely the conveying section, the compression section and the reverse screw section.

The screw sections are divided into seven with the total length screw of 750mm. From the hopper end, the lengths of the sections are 200mm, 200mm, 100mm, 100mm, 50mm, 50mm and 50mm respectively. The corresponding screw pitch is 50mm, 50mm, 35mm, 35mm, 35mm, 25mm and -15mm (because the direction is reversed) respectively (Figure 2). The die for the BC45 extruder has a single hole for extrusion. Changing the speed of the cutter can produce different shapes and size of extrudates. The width of the slot at the screw end for the dough to flow through to the die is 5mm. The diameter of the tapered die exit hole is 4mm. The entire system is computer controlled and the values of extrusion parameters can be varied through the control panel.

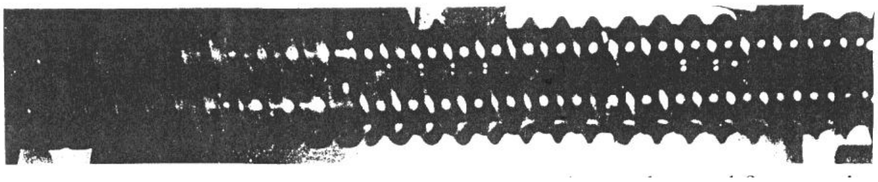

Figure 2 Screw configuration for the BC45 Clextral extruder used for experiments

The advantage of this machine is that it is very efficient in operation, it can work for several hours and the system parameters can be fixed and maintained as desired.

2.4 Experimental procedure

The white grain sorghum flour used for the experiment was procured from the Extension Unit of North East Agricultural University, Changchun. The proximate analysis (AOAC, 1995) and data given by the Agricultural University Food Research Laboratory for the grain sorghum flour (100 g) were as follows: moisture 11.5 g, carbohydrate 70.4 g, protein 9.2 g, ash 1.2 g, fat 3.5 g, tannin 0.06 g, and dietary fiber 4.1 g.

The moisture content of the sample was determined using AOAC method (1995). 5g of sample was weighed and transferred to pre-dried covered dish. The sample was dried in an electric oven at 130 ℃ until a constant dry weight of the sample was obtained. Known amount of water (10-25 mL) was added to the flour to bring the moisture content to the desired moisture content for different experimental runs. The samples were then refrigerated in sealed containers for 24 hours. Prior to extrusion, the samples were taken out and allowed to equilibrate with the room temperature.

For the experiments, the extruder throughput was put at 50 kg/h. Calibration of the feed rate and the water addition rate were carried out before each experimental run to ensure accuracy of the feed rate and water rate. The water addition rate was varied. These were done by having test runs for two minutes and the samples thus collected are measured. The value was then extrapolated to give the value on hour basis. Each experimental run was replicated thrice and the values reported were the average of the replicates.

The conduct of the experiment involves:

1. Test running the extruder for 30 – 45minutes to ensure smooth operation. At the same time, the barrel is thoroughly washed with water. The test run also allows the extruder barrel to be heated to the designed temperatures for extrusion.
2. Loading the feed hopper with pre-conditioned feed material.

3. Feeding the extruder with the feed material and water.
4. The remaining process depends on the peculiarity of the experiment.
5. Finally, the extrudates are collected for post-extrusion treatment and analysis.

2.5 Extrusion and extrudate properties measurements

2.5.1 Expansion indices (or puff indices)

The determination of longitudinal (or axial) LEI, radial (or sectional) SEI, and volumetric VEI indices are done according to the following expression (Alvarez-Martinez et al., 1988). Ten replicates were made to get the average.

For LEI, it is defined as :

$$LEI = \frac{V_e}{V_d} \qquad (2)$$

and

$$V_d = \frac{\dot{Q}_d}{S_d} \qquad (3)$$

where V_e is the extrudate velocity after expansion,: V_d is the extrudate velocity in the die,: S_d is the cross sectional area of the die, and $\dot{Q}_d$ is the volumetric flow rate through the die. A mass balance equation for the extruder will give the expression:

$$\dot{m}_d = \left[\frac{1-\dot{M}_e}{1-\dot{M}_d}\right]\dot{m}_e \qquad (4)$$

where $\dot{m}_d$ is the mass flow rate of the dough entering the die,: $\dot{m}_e$ is the mass flow rate of the extrudate, M_d and M_e are the dough and extrudate moisture content (w.b.). Also:

$$\dot{Q}_d = \frac{\dot{m}_d}{\rho_d} \qquad (5)$$

Substituting Equation 3, Equation 4 and Equation 5 into Equation 2

$$LEI = \frac{V_e \rho_d S_d}{\dot{m}_e}\left[\frac{1-M_d}{1-M_e}\right] \qquad (6)$$

Where, ρ_d is the density of the dough behind the die, which is determined to be $1250\ \mathrm{kg/m^3}$ (Adekola et al, 1998; Adekola, 2014). The actual velocity of the extrudate from the die, V_e is

$$V_e = L_{se}\dot{m}_e \qquad (7)$$

Where, L_{se} is the specific length of the extrudate defined as the length of the extrudate per unit mass (m/g). By substituting Equation 7 into Equation 6 and knowing that $S_d = \pi D_d / 4$ gives :

$$LEI = \left[\frac{\pi D_d^2}{4}\right] L_{se}\rho_d \left[\frac{1-M_d}{1-M_e}\right] \qquad (8)$$

Where, D_d is the diameter of the die.

The sectional expansion index is given as:

$$SEI = \frac{S_e}{S_d} = \frac{\left(\pi D_e^2/4\right)}{\left(\pi D_d^2/4\right)} = \left[\frac{D_e}{D_d}\right]^2 \qquad (9)$$

Where, S_e is the cross-sectional area of the extrudate, D_e is the diameter of extrudate.

The diameters of the extrudates were measured with vernier calipers. The volumetric expansion index is given as the product of the sectional and longitudinal expansion indices.

$$VEI = SEI \cdot LEI \qquad (10)$$

2.5.2 Bulk density

Bulk densities of the extrudate were obtained by volumetric displacement procedure (Hwang and Hayakawa, 1980), using glass beads as the filler medium. Glass beads with diameters ranging from 100 - 105 μm

were used. Nine pieces of extrudate, with each 3.5cm long were used. Three layers of extrudates completely covering with glass beads were made in sequence. The bulk density of extrudate was calculated using the Equation

$$\rho_{ex} = \frac{W_{ex}}{W_{gb}} \rho_{gb} \qquad (11)$$

Where, W_{ex} is the weight of the extrudate, W_{gb} is the weight of the glass beads, ρ_{gb} is the density of the glass beads. The density of the glass beads was determined using the volume displacement method. The density of the glass beads was calculated using the equation

$$\rho_{gb} = \frac{W_{gb}}{V_{gb}} \qquad (12)$$

Where, V_{gb} is the volume of the glass beads. Three replicates gave the average value for bulk density.

The alternative method used was to calculate the mean diameter and weight of the extrudate per unit length. It was assumed that the extrudate are perfectly cylindrical in shape. The volume of the cylindrical shape was then calculated. The weights per unit length (about 5 cm) were measured by using sensitive electronic weighing machine. Prior to measurement, extrudate samples were allowed to equilibrate with the laboratory atmosphere for about six days to bring them to uniform moisture content. Ten replicates of the diameter and weight gave the mean values reported. The results obtained from these methods are comparable.

2.5.3 Residence time

The residence time of the dough in the extruder are determined by introducing colored dye tracer in the feed at the entrance of the extruder. The residence time is determined by timing the time it takes the colored dough to be extruded. Different screw speeds were used during the experiment. Fifteen replicates gave the average value.

2.5.4 Solid density

The solid densities of the extrudate were determined by using air comparism multi-volume pycnometer. The extrudate samples were grounded to pass through 80 mesh sieve and placed in pycnometer cup. Masses were recorded and sample volumes were determined. The densities were calculated as mass per unit volume

2.5.5 Moisture content

The moisture content of the extrudate were determined by drying them in a conventional air-vacuum oven at 103 ℃ until a constant weight was obtained.

2.5.6 Specific mechanical energy (SME)

The total specific mechanical energy input during extrusion is estimated by using the following expression (Hseih et al., 1990; Hwang and Hayakawa, 1980).

$$SME = \frac{rs(run)}{rs(rated)} \times \frac{\%torque(run)}{100} \times \frac{mp(rated)}{pc} \qquad (13)$$

Where, ‘rs’ is rpm of screw, ‘mp’ is motor power and ‘pc’ is production capacity

2.5.7 Maximum stress, σ_m

The maximum stress of the dry extrudate was obtained as the ratio of maximum force applied during shearing and the corresponding cross sectional area of the extrudate. The maximum force during shearing was obtained using Instron Universal Testing machine. The machine was operated at the crosshead speed of 500 mm/min. Five replicates gave the average value reported.

2.5.8 Water-solubility index (WSI) and water-absorption index (WAI)

The water-solubility index and water-absorption indexes were measured (Anderson et al., 1969). The extrudate were milled to particle size of between 200 and 250 μm. A 2g sample was dispersed in 20g of distilled water. The resulting lumps were broken using a glass rod. The mixture was then stirred for about 20min., and afterwards rinsed into centrifuged tubes made up to 30g. The TGL – 16 centrifuge model was used. It was operated at the speed of 1600rpm for about 15 min. The supernatant was decanted to determine its solids content and the sediment was weighed. The indices for WSI and WAI are:

$$WSI(\%) = \frac{wdis}{wdrs} \times 100 \qquad (14)$$

Where, 'wdis' is the weight of dissolved solids in supernatant, 'wdrs' is the weight of dry solids

$$WAI = \frac{wse}{wds} \qquad (15)$$

Where, 'wse' is the weight of sediment and 'wds' is the weight of dry solids

Three replicates of the measurement were carried out.

2.5.9 Extruder output

The extruder output for each die were determined by collecting the sample of the extrudate for 5min and weighing after drying at 60℃ for 24h. The extrudate mass flow rate on dry basis was calculated (kg/h). On the volumetric basis, the flow rate was calculated by dividing the mass flow rate by the extrudate solid density.

2.5.10 Crispness and color

A taste panel of five was set up to evaluate the quality of the extrudate in terms of crispness and color of the extrudate. The scoring was from one to ten with the highest quality being ten while the lowest quality is one in that descending order.

2.5.11 Temperature, speed and moisture content

Furthermore, the Clextral extruder used for this experiment is equipped with sensors to measure and record temperatures during the experiment. The speeds of the extruder screw were set to required levels and measured accurately by the sensors attached to the extruder. The extrudate moisture contents were also determined (AOAC, 1995)

3 Results and discussion

The three processing parameters considered were: screw speeds (100 - 200rpm), temperature of barrel at the last section (100 - 160℃) and conditioned feed moisture content (15 - 24%). The feed material used was white grain sorghum flour. The numbers and levels of each parameter were shown in section 2.2 on experimental design in this paper.

3.1 Effects of temperature, screw speed and moisture contents on bulk and solid densities

The density of extrudate obtained varied between 176kg/m^3 and 1100kg/m^3 for the extrusion variables considered. The moisture content of the dry extrudate was between 6% and 7.5%. The experimental results obtained show the density of the extrudates at different extrusion moisture contents and barrel temperature. The results reveal an increasing density with increasing moisture content. It is noticeable that there was a sharp change in the density over a small temperature increment such as 20℃ increment from 140℃ to 160℃. These changes may be due to the complete disruption of the molecular structure of corn particle and gelatinization of their starch granules.

Low density was obtained with higher temperature (above 140℃). Low density is desirable in extrusion, because it enhances the extrudate quality. Conversely, high-density value was obtained for low barrel temperature (100 - 120℃). At low temperature, the corn particles are not completely disrupted and a poorly expanded and uncooked extrudate is produced with high density.

From the response surface diagram (RSD) (Figure 3), the effect of screw speed on density is negligible at constant feed rate. Also, there is a drastic reduction in density with an increase in temperature from 100℃ to 145℃, above 145℃, the rate of decrease in density became rather small. This strongly suggests that temperature has a significant impart on the density of the extrudate. Based on the experimental results, and using regression analysis, an equation was developed to show the relationship between density of extrudate and processing factors of the barrel temperature and the screw speed as below:

$$\rho = 7799.3256 - 98.3798X_1 - 0.9314X_2 + 0.3160X_1^2 + 0.0008X_1X_2 + 0.0025X_2^2 \tag{16}$$

Where, ρ is the density of the extrudate (kg/m^3), X_1 is the barrel temperature (℃), X_2 is the screw speed (rpm). The correlation coefficient at probability level of 0.01 for the polynomial is 0.986

The regression equation shows the dominance of the first order over the second order. The barrel temperature shows a negative linear effect whereas its quadratic effect is positive. This suggests that temperature has a curvilinear effect on extrudate density and at high temperature, its quadratic effect dominates.

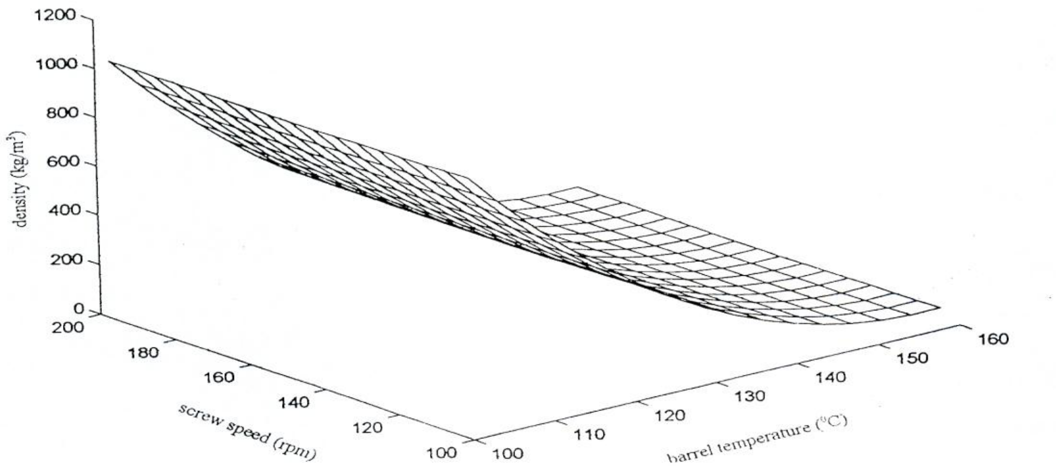

Figure 3 Response surface diagram (RSD) for extrudate density at different screw speeds and barrel temperatures

3.2 Effects of temperature and moisture contents on water solubility index

The water-solubility and water-absorption results are depicted in Figure 4 and Figure 5 respectively. The solubility tends to increase linearly with increase in temperature and decreases linearly with increase in moisture content. At temperature below 150℃, the WAI recorded is between 5.1 and 5.3 and to a large extent is independent of the extrusion moisture content. However, at the temperature higher than 150℃, there is a sharp increase in WAI to a value of six and above.

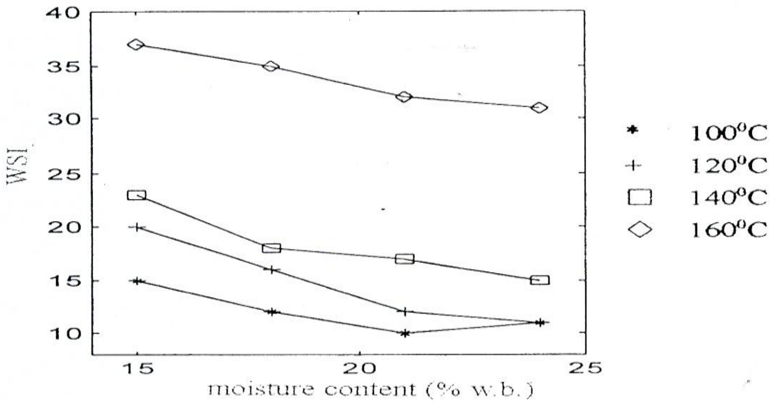

Figure 4 Water-solubility index of extrudates at different extrusion moisture contents and barrel temperatures

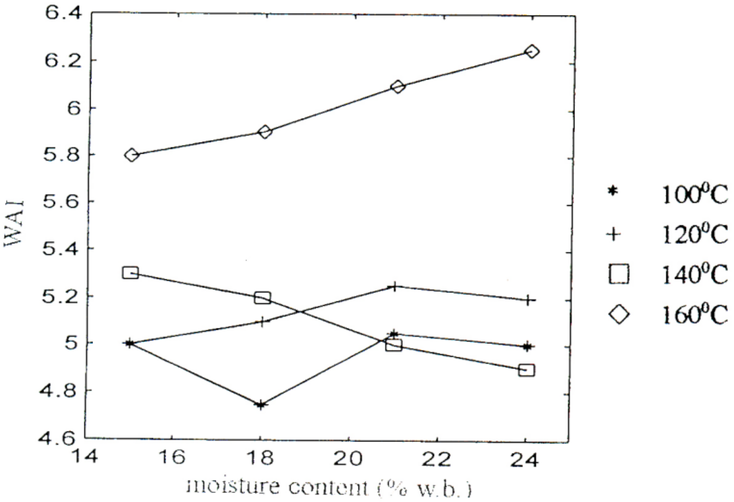

Figure 5 Water-absorption index of extrudate at different extrusion moisture contents and barrel temperatures

3.3 Effects of temperature and screw speed on SME

The total specific mechanical energy (SME) varied between 475kJ/kg and 1000kJ/kg. SME is directly proportional to screw speed. At low screw speed, the SME is low and vice-versa (Figure 6). Comparatively, the screw speed has dominating effects on SME while temperature has a small effect. The regression equation below relates barrel temperature (X_1), and screw speed (X_2) to SME.

$$\mathrm{SME} = \begin{array}{l} 606.372 - 2.856X_1 - 0.037X_2 \\ + 0.017X_1^2 - 0.006X_1X_2 + 0.005X_2^2 \end{array} \qquad (17)$$

Where, X_1 is the barrel temperature, X_2 is the screw speed. The correlation coefficient, r at probability level of 0.01 for the polynomial equation is 0.974

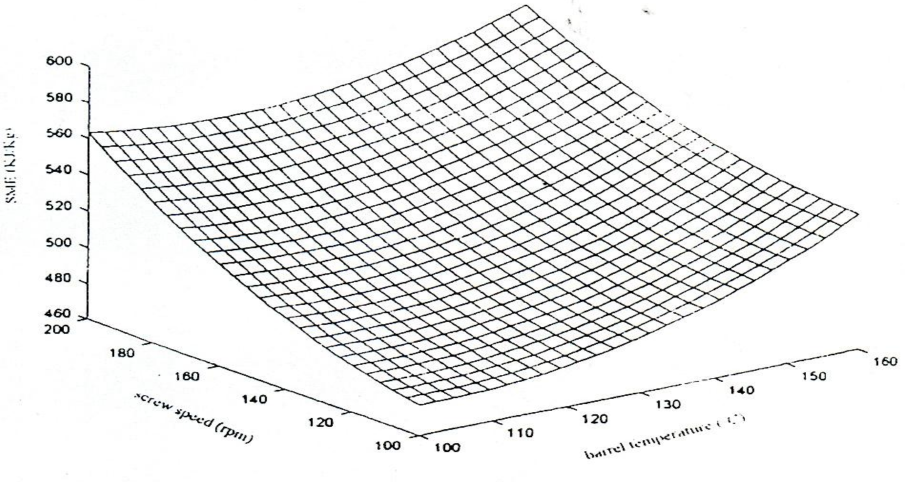

Figure 6 Response surface diagram for SME during extrusion of yellow corn flour at different barrel temperatures and screw speeds

4 Conclusions

In sorghum grain food extrusion operation carried out in this work, screw speed, barrel temperature and feed moisture content significantly affect the quality of extrudate and extrusion operation. From this work, the following conclusions can be made:

(1). At low barrel temperature, the value of extrudate density is high. Low density was obtained with temperature higher than 140 ℃. Conversely, high density value can be obtained for low barrel temperature between 100 ℃ and 120 ℃.

(2). At low temperature, the corn particles are not completely disrupted and a poorly expanded and uncooked extrudate is produced with high density.

(3). The effect of screw speed is negligible on density.

(4). The water solubility index (WSI) increases linearly with increase in temperature and decreases linearly with increase in moisture content.

(5). Screw speed has dominating effects on SME while temperature has small effects.

(6). At low screw speed, SME is low, and vice-versa.

(7). The study shows an increasing density with increasing moisture content. These changes are due to the complete disruption of the corn particle and gelatinization of their starch granules.

(8). Sorghum grain extruded at the conditioned feed moisture content of 20% and barrel temperature of 150 ℃ gave the optimum value for crispness and color quality.

(9). Understanding the mechanical and rheological properties of food dough will help in obtaining data and information necessary for research and development purposes.

(10). Focus should also be in the area of co-extrusion of sorghum with other cereals and additives to provide more nutritional snacks.

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