

T.R.
GEBZE TECHNICAL UNIVERSITY
GRADUATE SCHOOL OF NATURAL AND APPLIED SCIENCES

**OPTIMIZATION OF EXTRUSION CONDITIONS FOR
PRODUCTION OF THIRD GENERATION CORN SNACKS USING
RESPONSE SURFACE METHODOLOGY**

ÖZLEM AKTÜRK GÜMÜŞAY
**A THESIS SUBMITTED FOR THE DEGREE OF
DOCTOR OF PHILOSOPHY**
DEPARTMENT OF CHEMICAL ENGINEERING

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THESIS SUPERVISOR
PROF. DR. HASAN SADIKOĞLU

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FEN BİLİMLERİ ENSTİTÜSÜ

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EKSTRÜSYON KOŞULLARININ
OPTİMİZASYONU

ÖZLEM AKTÜRK GÜMÜŞAY
DOKTORA TEZİ
KİMYA MÜHENDİSLİĞİ ANABİLİM DALI

DANIŞMANI
PROF. DR. HASAN SADIKOĞLU

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2020



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SUMMARY

Corn grits were extruded under different extrusion process conditions [screw speed (100-200-300 rpm), feed moisture content (37-42-47%) and xanthan gum (XG) ratio (0-0.5-1%)] and produced half-products. Half-products were expanded by two different methods; frying and microwave heating. Firstly, effects of process conditions on the functional and physical properties of corn half products were investigated. Specific volume of half products was low for all extrusion combinations and negative correlated with screw speed, feed moisture and XG ratio. Low degree of starch gelatinization for all half products was obtained because of temperature under 100 °C in last two zones in extruder. Secondly, effects of process conditions on the physical, chemical, textural and microstructural properties of corn extrudates expanded by frying were investigated. Increased screw speed and feed moisture content increased expansion and oil content. Oil content decreased with increase of XG ratio. Low screw speed and high feed moisture content resulted in soft snacks. XG addition resulted in increase of hardness. Surface structure of snacks obtained at higher screw speed and feed moisture content was rough and involved some holes. However XG addition was resulted in smooth surface. Thirdly, effects of process conditions on the physicochemical properties of corn extrudates expanded by microwave heating were investigated. Increasing screw speed increased expansion and luminosity. Increasing feed moisture content resulted in reduced expansion, luminosity and total color difference. Incorporation of XG decreased luminosity and increased total color difference. Screw speed and feed moisture content had a positive effect on apparent viscosity and apparent amylose content but XG ratio had a negative effect on them. Finally, extrusion process optimized for three different corn products. Multi-response process optimization was realized with desirability function approach under response surface methodology. The results obtained in this thesis may be employed as baseline information for further improvement of third generation snack production with several additives and under different production conditions.

Key Words: Extrusion, Hydrocolloid, Xanthan Gum, Third Generation Snacks, Box-Behnken Design, Response Surface Methodology.

ÖZET

Mısır taneleri farklı proses koşullarında ekstrüde edildi [vida hızı (100-200-300 rpm), besleme nem içeriği (%37-%42-%47), ksantan gam oranı (%0-0,5-1)] ve yarı ürünler elde edildi. Yarı ürünler iki farklı metodla genişletildi; kızartma ve mikrodalgada ısıtma. İlk olarak, proses koşullarının mısır yarı ürünlerinin fonksiyonel ve fiziksel özellikleri üzerine etkisi araştırıldı. Yarı ürünlerin spesifik hacmi tüm ekstrüzyon kombinasyonları için düşüktü ve vida hızı, besleme nemi ve XG oranı ile negatif korelasyon gösterdi. Tüm yarı ürünler için nişastanın jelatinleşme derecesi düşük bulunmuştur çünkü ekstrüderdeki son iki bölgedeki sıcaklıklar 100 °C' nin altında tutulmuştur. İkinci olarak, proses koşullarının kızartma ile genişletilen mısır ekstrüdatlarının fiziksel, kimyasal, dokusal ve mikroyapısal özellikleri üzerindeki etkileri araştırıldı. Artan vida hızı ve besleme nemi içeriği genişleme ve yağ içeriğini artırmıştır. XG oranındaki artışla yağ içeriği azalmıştır. Düşük vida hızı ve yüksek nem içeriği atıştırmalıkların yumuşak olmasına neden olmuştur. XG ilavesi sertliğin artmasına neden olmuştur. Daha yüksek vida hızı ve besleme nemi içeriğinde elde edilen atıştırmalıkların yüzey yapısı sert ve bazı delikler gözlenmiştir. Bununla birlikte, XG ilavesi pürüzsüz bir yüzey ile sonuçlanmıştır. Üçüncü olarak, işlem koşullarının mikrodalga ile genişletilen mısır ekstrüdatlarının fizikokimyasal özellikleri üzerindeki etkileri araştırıldı. Vida hızının artırılması ile genişleme ve parlaklık arttı. Besleme nemi içeriğinin arttırılması, genişleme, parlaklık ve toplam renk farkının azalmasına neden oldu. XG' nin eklenmesi, parlaklığı azalttı ve toplam renk farkını arttırdı. Vida hızı ve besleme nemi içeriğinin görünür viskozite ve görünür amiloz içeriği üzerinde olumlu etkisi vardır, ancak XG oranı bunlar üzerinde olumsuz etkiye sahiptir. Son olarak, ekstrüzyon işlemi üç farklı mısır ürünü için optimize edilmiştir. Çok-yanıtlı proses optimizasyonu, yanıt yüzey yöntemi yaklaşımı ile istenenilebilirlik fonksiyonu kullanılarak gerçekleştirilmiştir. Bu tezde elde edilen sonuçlar, çeşitli katkı maddeleri ile birlikte ve farklı üretim koşulları altında üçüncü nesil atıştırmalık üretiminin daha da geliştirilmesi için temel bilgi olarak kullanılabilir.

Anahtar Kelimeler: Ekstrüzyon, Hidrokolloid, Ksantan gam, Üçüncü Nesil Atıştırmalık, Box-Behnken Dizayn, Yüzey Yanıt Yöntemi.

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LIST of ABBREVIATIONS and ACRONYMS

<u>Abbreviations</u> <u>and Acronyms</u>	<u>Explanations</u>
<i>a</i>	: Chromatic component from greenness (-) to redness (+)
ANOVA	: Analysis of variance
<i>b</i>	: Chromatic component from blueness (-) to yellowness (+)
BBD	: Box-Behnken design
BI	: Browning index
DG	: Degree of gelatinization
EI	: Expansion index
<i>L</i>	: Lightness component or luminosity from black (0) to white (100)
RSM	: Response surface methodology
WAI	: Water absorption index
WSI	: Water solubility index
XG	: Xanthan gum
ΔE	: Total color difference

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1. INTRODUCTION

Extrusion cooking is used to create a cooked and shaped food product. In extrusion cooking, food material is subjected to mechanical shearing in a barrel and moistening that convert the starchy material into dough-like material before being forced through a die. Heat is applied directly by electric heaters on the barrel or mechanical shear. Process temperature can be increased 200 °C, this type of extrusion is called as high-temperature short-time process. Generally, extruder types can be categorized into three main classes; hydraulic ram types (piston), roller types and screw types. Screw extruders (Figure 1.1) have single screw, twin or multiple screws. Twin screw extruders are classified as counter-rotating and co-rotating extruders according to screw configuration. They are very useful process due to its versatility, high productivity, relative low cost energy efficiency and lack of effluents [Lazou and Krokida, 2010], [Altan et al., 2008], [Frame, 1994].

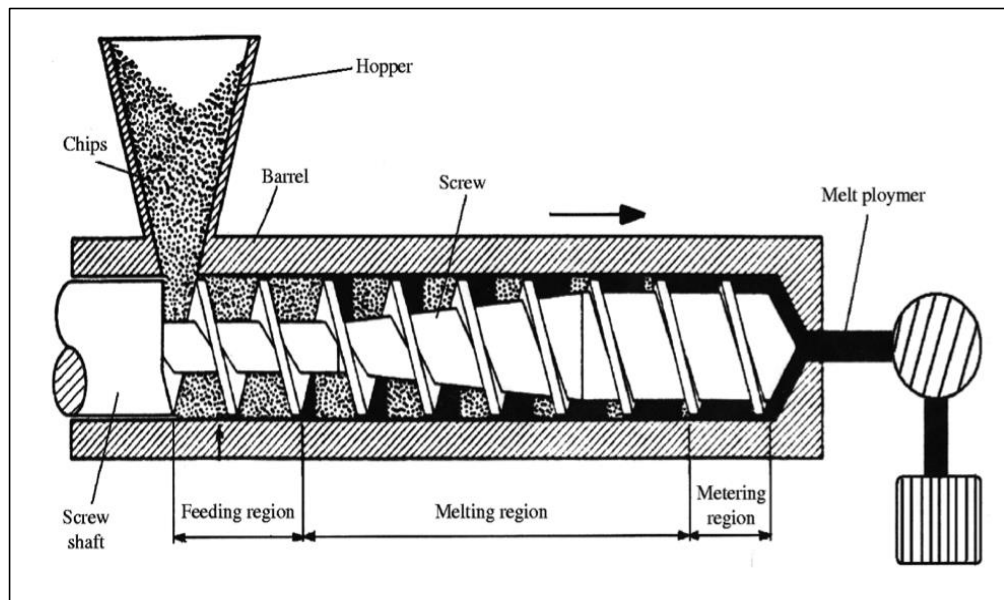


Figure 1.1: Schematic diagram of screw extruder.

Extrusion cooking process occupies an important place in the food industry for the production of snacks, breakfast cereals, breadcrumbs, and animal feeds [Seker et al., 2004]. Snacks have been categorized into three major groups: first generation (conventional potato chips and baked crackers), second generation (directly expanded in extruder die), and third generation (semi- or half-) products. ‘Third generation

snacks' or 'half products' are not expanded directly out of the extruder die. They are formed by extruding cereals at high moisture contents, moderate shear and barrel temperature, and die temperatures below 100 °C. They are dried to a final moisture content of about 10-14% after extrusion. Dried half-products have a long shelf life (at least a year after production) and require less storage space due to unexpanded volume when compared with directly expanded snacks. However, additional process such as frying, hot-air oven, or microwave heating is required for remove moisture and achieve the final texture. During expansion process, the moisture in the half product starts to boil, vapor bubbles are formed and final expansion of half product are provided [Delgado et al. 2012], [Seker, 2011], [Gimeno et al., 2004], [Suknark et al., 1999], [Harper, 1994].

Different chemical and physical changes occur in the food structure during extrusion of the food materials. Starch gelatinization, Maillard reaction, denaturation of vitamins and enzymes, protein denaturation and cross-linking can be given examples of these changes. Extrudate properties are affected by the extruder variables (e.g., screw and barrel design and die geometry), process variables (e.g., feed rate, screw speed and barrel temperature) and the feed composition variables (e.g. water, starch, protein, fiber, lipid, sugar and salt ingredients) [Harper, 1981], [Camire et al., 2011].

Starch based materials are preferred raw material for production of extruded products. Corn is a widespread used cereal in the extrusion industry. Corn can be processed in a variety of granulations, from a coarse grits to a fine flour. [Harper, 1994]. Corn extrudate has been obtained with good physical and functional properties varying process conditions such as feed composition, feed moisture, feed rate, screw speed, barrel temperature [Yu et al., 2012], [Chiu et al., 2013]. Feed composition is one of the most important factors for extrusion process. Incorporation of food additives such as hydrocolloids into feed can help to improve the thermostability and to provide lubrication during extrusion process [Gimeno et al., 2004], [Singh and Singh, 2004].

Xanthan gum (XG) is a hydrocolloid produced commercially by aerobic submerged fermentation from *Xanthomonas campestris* and it has a wide range of applications in food and non-food industries due to its rheological characteristics and physical properties [Butler, 2016]. Furthermore, addition of hydrocolloids such as hydroxypropyl methyl cellulose, carboxyl methyl cellulose, guar gum, gellan gum and

XG are known to restrain oil intake of deep fat fried snacks [Bajaj and Singhal, 2007], [Annapure et al., 1999]. However, addition of hydrocolloids to extrusion process can be effect the quality characteristics of extrudates. Therefore, it will be useful to evaluate hydrocolloid level and the other process conditions by considering multiple quality characteristic.

Response surface methodology (RSM) is frequently preferred in various extrusion studies to associate quality properties of product with process variables [Teba et al., 2017], [Xu et al., 2016]. RSM is a collection of mathematical and statistical methods to evaluate relationships between a group of factors (independent variables) and one or more response variables (affected by the factors). Response function (model equation), contour and graphics of the response surface can be obtained by this technique. RSM is used for developing, improving and optimizing the several processes [Myers and Montgomery, 1995]. Therefore, RSM can be useful tool for development extrusion products.

1.1. Hypothesis

Extrusion process can be practiced with different feed composition and process conditions for improve the extrudate quality. Incorporation of the gums into the feed material significantly affects the physical, chemical and textural properties of the extrudate. In addition to this, additives used in produce the third generation snacks can be change the effectiveness of expansion process (microwave or frying) applied after extrusion. Addition level for gums is important factor for snack structure. Because, above a critical additional level, fiber or gum molecules can be bind the moisture in the melt or they can be break damage the structure of melt during extrusion. Deterioration of food structure negatively affects color, shape and texture properties and these properties are very important for consumers. As a result, determination of effects of gum addition level and the other process conditions is important for desirable product.

1.2. Objectives

The objectives of this study are to:

- i) determine the effects of screw speed, feed moisture and XG level on the water absorption index (WAI), water solubility index (WSI), degree of gelatinization (DG), water activity (a_w), specific volume and color properties of half products and multiple-response process optimization.
- ii) determine the effects of screw speed, feed moisture and XG level on the expansion, oil absorption, hardness, color and microstructural properties of fried extrudates and multiple-response process optimization
- iii) determine the effects of screw speed, feed moisture and XG level on the expansion, color and rheological properties of microwave expanded extrudates and multiple-response process optimization

2. METHODOLOGY

Details of the materials used and methods followed to:

- i) determine the effects of screw speed, feed moisture and XG level on the WAI, WSI, DG, a_w , specific volume and color properties of half products are explained in the “Materials and Methods for Half Products”.
- ii) determine the effects of screw speed, feed moisture and XG level on the expansion, oil absorption, hardness, color and microstructural properties of fried extrudates are explained in the “Materials and Methods for Fried Half Products”.
- iii) determine the effects of screw speed, feed moisture and XG level on the apparent amylose content, expansion, color and rheological properties of microwave expanded extrudates are explained in the “Materials and Methods for Microwave Expanded Half Products”.

3. EFFECTS of EXTRUSION CONDITIONS on FUNCTIONAL and PHYSICAL PROPERTIES of CORN HALF PRODUCTS

Starch based materials are preferred raw material for production of extruded products. Starch plays a major role in expansion due to gelatinization during extrusion cooking of foods. Gelatinization, the conversion of raw starch to a cooked and digestible material by the application of water and heat, is one of the important effects that extrusion has on starch components of foods [Ding et al., 2005]. WAI and WSI can be used to estimate the functional characteristics of extrudates. WAI measures amount of water absorbed by starch and WSI measures the amount of soluble components released from the starch after extrusion. Determination of these characteristics is important for starch gelatinization. Moisturizing is an important process to provide the starch gelatinization with extrusion. Lawton et al. [1972] found that moisture and barrel temperature had the greatest effects on gelatinization of corn starch [Lawton et al., 1972]. Gomez and Aguilera [1983] shown that maximum gelatinization was occurred at about 28-29% moisture content [Gomez and Aguilera, 1983].

The degree of gelatinization were changed depend on the extrusion conditions. Cai and Diosady [1993] found that degree of starch gelatinization increased with increasing screw speed [Cai and Diosady, 1993]. These researchers studied the wheat starch gelatinization profile along the channel of a twin-screw extruder at 25% and 30% moisture contents, screw speeds of 200 and 300 rpm, and barrel temperatures of 100-160 °C. Similar results were observed by Suknark et al. [1999] who studied with twin screw extrusion of tapioca-fish and tapioca-peanut snacks at 90-95-100 °C temperature and 100-250-400 rpm screw speed [Suknark et al., 1999]. Effect of temperature on that degree of starch gelatinization was a similar tendency with screw speed in same research. Similar results were reported by Bhattacharya and Hanna [1987] who observed that an increased barrel temperature and increased the degree of gelatinization [Bhattacharya and Hanna, 1987].

Suknark et al. [1999] demonstrated that when temperatures in the last two zones were <90 °C, half-product with a low degree of gelatinization was obtained and this resulted in hard, poorly expanded products after frying. They found that the

temperature in the last zone and at the die was required to be ≤ 100 °C in order to produce flat, thin, bubble-free extrudates [Suknark et al., 1999]. Degree of gelatinization were affected physical properties such as bulk density, specific volume and color. Case et al. [1992], who studied with extrusion of wheat and corn, reported that as degree of gelatinization increased, the expansion of puffed product increased and bulk density decreased during subsequent frying [Case et al., 1992].

In the literature, several studies have been carried out extrusion of corn. Bhattacharya and Hanna [1987] studied that extrusion of ordinary corn (30% amylose) and waxy corn (1% amylose) at 17.8, 20, 30, 40 and 42.2% product moisture contents and different extruder barrel temperatures and they reported that the degree of gelatinization decreased with increasing moisture content but increased with increasing temperature [Bhattacharya and Hanna, 1987]. Fallahi et al. [2016] were used single screw extruder for corn, potato and cassava starches. They demonstrated that corn extrudates had higher degree of structural change during extrusion [Fallahi et al., 2016].

Mezreb et al. [2003] studied that extrusion cooking of corn and wheat flour was carried out in a twin-screw co-rotating extruder and results shown that increase in screw speed resulted in products possessing greater longitudinal expansion, higher water solubility and smaller structural patterns [Mezreb et al., 2003].

Stojceska et al. [2009] investigated that effect of different levels of feed moisture (12–17%) during extrusion cooking, using a co-rotating twin-screw extruder on selected nutritional and physical properties of extruded products and they reported that water feed level affected total dietary fiber content of the extrudates and affected the expansion ratio, bulk density, hardness, water solubility index and color [Stojceska et al., 2009]. Mezreb et al. [2006] were studied that effect of sucrose on the textural properties of corn and wheat extrudates and they demonstrated that expansion decreased and crispiness increased with sucrose addition [Mezreb et al., 2006]. Maga et al. [1991] reported that gellan gum addition to corn grits during extrusion resulted in increase the WAI of extrudates [Maga et al., 1991]. Singh and Singh [2004] were studied that extrusion of corn grits with sodium alginate, which is a hydrocolloid, and they reported that sodium alginate addition resulted in increase of WAI and viscosity values, decrease the WSI and expansion of extrudates [Singh and Singh, 2004].

Carvalho et al. [2009] investigated that effect of particle size on the extrusion of corn meal by a twin-screw extruder and they found that extrudates produced with corn

meal of higher particle sizes expanded more than extrudates produced with smaller particle sizes [Carvalho et al., 2009]. The results have shown that increasing corn meal particle size decreased water absorption index values.

3.1. Materials and Methods for Half Products

Food-grade yellow corn grits (Durmuşoğlu Trade Company, Istanbul, Turkey) were sieved between 7 and 8 meshes. The desired feed moisture on wet basis (37, 42 or 47%) and XG level (0, 0.5 or 1%) of the corn grits were obtained by addition of distilled water and food grade XG (KimbioTek Chemical Substances Trade Company, Istanbul, Turkey), and mixing for at least 30 min in an industrial food mixer. Feed material was kept at 6-8 °C overnight before extrusion process.

An industrial scale twin-screw extruder (Aysa Machine, Istanbul, Turkey) with a slit die (3x18 mm) was used. Screw diameter and length/diameter ratio of extruder were 30 mm and 27:1, respectively. The extruder had 6 heated/cooled zones located on the main barrel. During the extrusion, 6 heat zones were set to 70-70-75-80-85-85 °C, respectively. Material was fed into the extruder inlet port with a volumetric feeder with constant feed rate. Extrusion conditions were established basing on preliminary experiments and literature datas.

Corn grits were extruded at high moisture content (>35%), low die temperature (<100 °C) and moderate screw speed [Seker, 2011], [Suknark et al., 1999]. Thus, partially cooked and non-expanded pellets were obtained. Corn grits could be moisturized up to 47% (maximum value) according to preliminary experiments. Considering these conditions, feed moisture levels were selected as 37%, 42% and 47%. Starch gelatinization of feed material into the extruder is an important factor for production of indirectly expanded pellets. Additive material can be effect the starch gelatinization. When addition level was above 1%, starch gelatinization decreased under 50% according to preliminary experiments. However, Lee et al. (2000) reported that expansion of starch pellets was optimized at ≈50% gelatinization of starch. Considering these conditions, XG ratios were determined as 0%, 0.5% and 1%.

Extrudates were manually cut into 8 cm lengths and dried at 90 °C for 10 min to remove the surface moisture. After they were packaged in freezer bags and stored at -18 °C to final drying. Final drying process was carried out at 50 °C for 12-14 h to 12-

13% moisture content of half products. Half products were stored in freezer bags/glass containers at 4 °C until further experiments.

3.1.1. Proximate Composition of Raw Material

The proximate composition of corn grits was determined according to AOAC [2000] standards: moisture content (Method 930.15), protein content (Method 990.03), fat content (Method 920.39), ash content (Method 942.05). Carbohydrate content was determined as the rest of dry mass to 100%. The experiments were carried out in three parallel.

Corn grits used in this study have 11.26 g 100 g⁻¹ moisture, 6.85 g 100 g⁻¹ protein, 3.38 g 100 g⁻¹ total fat, 0.87 g 100 g⁻¹ ash and 77.64 g 100 g⁻¹ carbohydrate in wet basis.

3.1.2. Experimental Design

A Box-Behnken design (BBD) was used for the design of experiment with three independent variables, including screw speed (X_1), feed moisture content (X_2), and XG level (X_3) [Box and Behnken, 1960]. The levels of each independent variable were established basing on preliminary trials and related literature data. The three levels of process variables were coded as -1 , 0 , and $+1$. Coded and actual values of the independent variables, levels and experimental design are given in Table 3.1. Totally 17 randomized trials with five replicates at the center point were studied. The dependent variables (responses) were selected as WAI, WSI, DG, a_w , specific volume and color properties (L , a and b values). The predictive regression models were constructed for responses by using the Design-Expert software (Version 10.0.3, Stat-Ease Inc., Minneapolis, USA). A second order polynomial model was fitted for three factor design which is given as follows (Equation 3.1):

$$Y = \beta_0 + \sum_{i=1}^3 \beta_i X_i + \sum_{i=1}^3 \beta_{ii} X_{ii}^2 + \sum_{i=1}^3 \sum_{j=1, i < j}^3 \beta_{ij} X_i X_j + \varepsilon \quad (3.1)$$

where Y is the predicted response; X_i and X_j are coded values of the process variables (screw speed, feed moisture and XG level); β_0 the constant coefficient, β_i the linear coefficient, represent, β_{ii} the quadratic coefficients, β_{ij} the interaction coefficients and ε the residual error.

Table 3.1: Coded and uncoded values for the experimental design.

Runs	Coded values			Uncoded Values		
	Screw speed	Feed moisture	XG ratio	Screw speed	Feed moisture (%)	XG ratio
	(rpm) X_1	(%) X_2	(%) X_3	(rpm)		(%)
1	0	0	0	200	42	0.5
2	+1	+1	0	300	47	0.5
3	0	+1	-1	200	47	0
4	0	+1	+1	200	47	1
5	-1	0	+1	100	42	1
6	0	0	0	200	42	0.5
7	0	0	0	200	42	0.5
8	0	0	0	200	42	0.5
9	+1	0	+1	300	42	1
10	0	-1	-1	200	37	0
11	+1	0	-1	300	42	0
12	-1	+1	0	100	47	0.5
13	-1	-1	0	100	37	0.5
14	0	-1	+1	200	37	1
15	-1	0	-1	100	42	0
16	0	0	0	200	42	0.5
17	+1	-1	0	300	37	0.5

3.1.3. Water Absorption Index and Water Solubility Index

WAI and WSI of half products were determined by a modification of the method of Chang and Ng [2011]. The half products were ground and sieved through 200 μm sieve. One g of ground half product was placed in a tared centrifuge tube and distilled water (10 ml) at 25 °C was added. The tubes were kept for 10 min and were inverted three times each at 0, 5 and 10 min. After 10 min, the suspensions were centrifuged (Elektromag M 4812 P laboratory centrifuge, Istanbul, Turkey) at 3000 rpm for 25

min. The supernatant was decanted into a tared aluminum pan and dried at 60 °C for 24 h. The weight of the pellet remaining in the centrifuge tube was noted. The results were calculated as in Equation (3.2) and Equation (3.3):

$$WAI(g/g) = \frac{\text{Weight of pellet}}{\text{Sample weight (db)}} \quad (3.2)$$

$$WSI(\%) = \frac{\text{Weight of dried supernatant}}{\text{Sample weight (db)}} \times 100 \quad (3.3)$$

The results were reported as the average of three separate measurements.

3.1.4. Degree of Gelatinization

The half products were ground and sieved through 180 µm sieve. Then ground samples were dried at 50 °C for 12 h. DG of ground dried samples was determined according to the spectrophotometric method [Birch and Priestley, 1973]. Ground dried samples (0.2 g) were placed in a centrifuge tube. Then water (98 mL) and 10 M-KOH solution (2 mL) were added and gently agitated for 10 min. The slurry was centrifuged at 3000 rpm for 15 min. Aliquots of the supernatant (1mL) were neutralized with 0.5 M HCl (0.4 mL), made to 10 mL with distilled water and mixed with 0.1 mL of iodine reagent (1 g iodine and 4 g potassium iodide per 100 mL distilled water). Fifteen min after addition of iodine reagent, absorbance, A_1 was read at 600 nm in a spectrophotometer (Lambda 35, Perkin Elmer, Shelton, USA), against a reagent blank. The estimation was repeated using 95 ml water and 5 mL 10M-KOH solution, and 1.0 mL 0.5 M-HCl for neutralization to obtain absorbance, A_2 . DG was calculated by the ratio of A_1 and A_2 . Three measurements were made on each half product, and the average value was used.

3.1.5. Water Activity

Water activity of ground half products was measured at 25 °C on Aqua Lab 3TE water activity meter (Decagon Devices Inc., Pullman, WA). Ten different 8 cm

extrudates were ground for each sample and all measurements were performed in triplicate.

3.1.6. Specific Volume

Specific volume of the half products was determined using a glass bead (0.1 mm diameter) displacement method. Specific volume (mL/g) was calculated by dividing the volume displaced by the weight of the extrudates. [Bhatnagar and Hanna, 1995]. The results were reported as the average of ten separate measurements.

3.1.7. Color Properties

Color values of half products were measured by using a Konica Minolta CR-400 Chroma Meter (Konica Minolta Sensing, Inc., Osaka, Japan) with an illuminant D65 and the 10° observer angle, according to Hunter Lab system (*L*: lightness, *a*: redness, and *b*: yellowness). Before measuring, the colorimeter was calibrated against a standard white tile. Ten measurements were performed on each sample and averaged.

3.1.8. Statistical Analysis and Optimization

The levels of significance of all terms in the polynomials were determined by Analysis of Variance (ANOVA) Technique. Model development was applied by using significance levels at $p < 0.01$, $p < 0.05$ and $p < 0.1$. Adequacy of developed models were tested using the model analysis, lack of fit test and coefficient of determination (R^2). Three-dimensional surface plots were generated by keeping the one independent variable at the middle value and they were established for understand how the responses alter with respect to the extrusion factors.

Process variables were optimized numerically with response surface of desirability function using statistical Design-Expert software. In this optimization method, each response is transformed into a desirability value, called d_i . The scale for d_i is between 0 and 1. If d_i is close to 1, response has an ideal value. However, if d_i is close to 0, response has not an acceptable value. Quality parameters such as color and shelf life were the most important major parameters for third generation snack foods.

Therefore, optimum conditions for extrusion of corn grits were determined to obtain minimum WAI and a_w (for higher shelf life), maximum L and b value (for light and yellowness). Other responses and process variables were kept in range during the optimization.

3.2. Results and Discussion for Corn Half Products

Experiments were carried out according to BBD and experimental data (Table 3.2) were fitted to Equation (3.1) to develop models for all the responses. ANOVA results (Table 3.3) showed that the models developed for all the responses were statistically significant ($p \leq 0.01$ and $p \leq 0.05$). Lack of fit F -values were non-significant ($p > 0.05$) for all models. Final equation of the fitted model and R-squared values were demonstrated in Table 3.4. The predicted R-squared values was found reasonable agreement with the adjusted R-squared values for all the parameters.

3.2.1. Effects of Extrusion Conditions on WAI and WSI

WAI and WSI can be used to estimate the functional characteristics of extrudates. WAI measures amount of water absorbed by starch and WSI measures the amount of soluble components released from the starch after extrusion. The WAI values for the half products ranged between 4.77 ± 0.93 and 6.76 ± 0.14 g/g (Table 3.2). The linear fitted model showed that screw speed and feed moisture content had significantly negative effect whereas XG had a positive effect on WAI values. WSI was significantly influenced by screw speed, feed moisture and XG content (Table 3.3). Lower screw speed increases input of thermal energy due to high residence time [Seker, 2005]. So level of starch degradation and WAI value may increase. The results are in accordance with previous works [Stojceska et al., 2008], [Yagci and Gogus, 2008]. WSI increased as a function of screw speed. Ainsworth et al. [2007] reported that increased shear effects at higher screw speeds may lead to enhanced starch gelatinization and so the molecular weight of starch granules decreased hence increased WSI [Ainsworth et al., 2007]. Sobukola et al. [2013] and Mezreb et al. [2003] reported that increase in WSI with increase in screw speed [Sobukola et al., 2013] [Mezreb et al., 2003].

Table 3.2: Experimental results of half products for response surface analysis.

Runs	Half product quality responses							
	WAI	WSI (%)	DG (%)	a _w	Specific Volume (mL/g)	<i>L</i>	<i>a</i>	<i>b</i>
1	5.57 ± 0.58	4.60 ± 0.17	54.80 ± 1.29	0.70 ± 0.006	1.55 ± 0.070	50.50 ± 1.37	5.46 ± 0.32	22.38 ± 1.00
2	4.77 ± 0.93	4.34 ± 0.17	54.21 ± 1.76	0.62 ± 0.005	1.36 ± 0.051	63.82 ± 1.00	5.99 ± 0.33	29.00 ± 0.69
3	5.15 ± 0.13	4.25 ± 0.18	46.33 ± 0.58	0.75 ± 0.003	1.45 ± 0.076	55.62 ± 1.41	6.54 ± 0.39	25.64 ± 1.13
4	5.24 ± 0.18	4.15 ± 0.11	48.75 ± 0.90	0.67 ± 0.007	1.52 ± 0.040	62.90 ± 0.82	6.29 ± 0.42	29.12 ± 0.52
5	6.27 ± 0.42	4.43 ± 0.11	56.49 ± 0.83	0.70 ± 0.004	1.53 ± 0.028	50.89 ± 3.19	5.24 ± 0.40	21.53 ± 1.16
6	5.86 ± 0.50	4.60 ± 0.10	54.49 ± 0.36	0.69 ± 0.009	1.57 ± 0.090	51.48 ± 0.94	5.12 ± 0.30	23.48 ± 0.58
7	5.84 ± 0.18	4.65 ± 0.04	55.37 ± 1.34	0.71 ± 0.002	1.58 ± 0.096	52.71 ± 1.30	5.26 ± 0.38	22.52 ± 0.94
8	5.59 ± 0.12	4.60 ± 0.13	55.55 ± 0.71	0.70 ± 0.007	1.59 ± 0.022	51.35 ± 0.87	5.26 ± 0.15	23.57 ± 0.62
9	5.70 ± 0.13	4.70 ± 0.05	58.37 ± 1.15	0.73 ± 0.007	1.63 ± 0.036	59.01 ± 1.13	4.99 ± 0.44	26.44 ± 1.23
10	6.35 ± 0.23	5.10 ± 0.10	52.23 ± 0.39	0.73 ± 0.001	1.68 ± 0.085	51.81 ± 1.30	4.59 ± 0.43	21.04 ± 0.91
11	5.32 ± 0.29	4.79 ± 0.02	52.62 ± 0.68	0.69 ± 0.003	1.76 ± 0.052	62.23 ± 1.29	5.28 ± 0.48	27.08 ± 0.94
12	5.44 ± 0.29	4.04 ± 0.15	57.63 ± 1.14	0.68 ± 0.005	1.80 ± 0.095	46.24 ± 0.86	6.31 ± 0.33	20.11 ± 0.46
13	6.75 ± 0.22	4.94 ± 0.15	60.91 ± 0.96	0.71 ± 0.007	1.80 ± 0.052	43.17 ± 1.53	4.59 ± 0.43	18.08 ± 1.23
14	6.76 ± 0.14	5.16 ± 0.18	53.76 ± 1.02	0.76 ± 0.003	1.81 ± 0.052	43.54 ± 0.73	3.92 ± 0.29	16.57 ± 0.60
15	5.61 ± 0.13	4.63 ± 0.14	54.44 ± 0.91	0.80 ± 0.005	1.83 ± 0.091	47.74 ± 1.00	5.35 ± 0.53	20.87 ± 0.99
16	5.58 ± 0.29	4.65 ± 0.05	55.46 ± 1.28	0.71 ± 0.005	1.55 ± 0.030	51.18 ± 0.95	5.59 ± 0.31	21.98 ± 0.68
17	5.00 ± 0.24	5.25 ± 0.07	63.36 ± 0.60	0.66 ± 0.004	2.18 ± 0.094	41.64 ± 1.30	4.15 ± 0.59	16.74 ± 0.87

Table 3.3: Statistical analyzed data (F values) for half product quality responses.

Source	F-value							
	WAI	WSI	DG	a _w	Specific volume	<i>L</i>	<i>a</i>	<i>b</i>
Fitted model	Linear	Linear	Quadratic	Quadratic	Quadratic	Quadratic	Linear	Quadratic
Screw speed (SS)	28.06 ^a	60.24 ^a	0.24	77.99 ^a	62.02 ^a	152.07 ^a	5.13 ^b	64.56 ^a
Feed moisture content (FM)	136.87 ^a	750.20 ^a	155.75 ^a	42.35 ^a	87.81 ^a	238.55 ^a	272.86 ^a	183.07 ^a
XG ratio (XG)	12.05 ^a	6.07 ^b	39.47 ^a	26.14 ^a	44.61 ^a	0.11	7.66 ^b	0.17
SS*FM	-	-	19.70 ^a	0.43	130.40 ^a	74.32 ^a	-	38.76 ^a
SS*XG	-	-	7.83 ^b	84.69 ^a	62.33 ^a	8.26 ^b	-	0.63
FM*XG	-	-	0.45	52.28 ^a	1.61	49.21 ^a	-	23.41 ^a
SS ²	-	-	200.52 ^a	18.63 ^a	45.43 ^a	1.29	-	1.31
FM ²	-	-	4.06	24.91 ^a	2.26	15.31 ^a	-	11.29 ^b
XG ²	-	-	169.95 ^a	140.89 ^a	228.90 ^a	58.65 ^a	-	17.03 ^a
Lack of Fit	1.17	3.88	3.86	0.60	4.91	3.12	0.75	1.85
Total Model	58.99 ^a	272.17 ^a	64.33 ^a	51.05 ^a	74.97 ^a	66.04 ^a	95.21 ^a	37.64 ^a

Significance level: ^a $p \leq 0.01$, ^b $p \leq 0.05$.

Table 3.4: Predicted models for the experimental data of half products.

Functional and physical properties		R ²
WAI	$Y = 5.75 - 0.29X_1 - 0.65X_2 + 0.19X_3$	0.9316
WSI (%)	$Y = 4.64 + 0.13X_1 - 0.46X_2 - 0.041X_3$	0.9843
DG (%)	$Y = 55.11 - 0.11X_1 - 2.92X_2 + 1.47X_3 - 1.47X_1X_2 + 0.92X_1X_3 + 0.22X_2X_3 + 4.56X_1^2 - 0.65X_2^2 - 4.20X_3^2$	0.9881
a _w	$Y = 0.70 - 0.024X_1 - 0.018X_2 - 0.014X_3 - 0.0025X_1X_2 + 0.035X_1X_3 - 0.027X_2X_3 - 0.016X_1^2 - 0.018X_2^2 + 0.044X_3^2$	0.9850
Specific volume (mL/g)	$Y = 1.82 - 0.099X_1 + 0.12X_2 + 0.084X_3 - 0.20X_1X_2 + 0.14X_1X_3 - 0.022X_2X_3 - 0.12X_1^2 + 0.026X_2^2 - 0.26X_3^2$	0.9897
<i>L</i>	$Y = 51.44 + 4.83X_1 + 6.05X_2 - 0.13X_3 + 4.78X_1X_2 - 1.59X_1X_3 + 3.89X_2X_3 - 0.61X_1^2 - 2.11X_2^2 + 4.14X_3^2$	0.9884
<i>a</i>	$Y = 5.29 - 0.13X_1 + 0.99X_2 - 0.16X_3$	0.9565
<i>b</i>	$Y = 22.79 + 2.33X_1 + 3.93X_2 - 0.12X_3 + 2.567X_1X_2 - 0.32X_1X_3 + 1.99X_2X_3 - 0.46X_1^2 - 1.35X_2^2 + 1.65X_3^2$	0.9798

Increasing feed moisture content caused a decrease in WAI (Figure 3.1a) and WSI (Figure 3.1c). Seker and Hanna [2005] found that there was a decrease in WAI and WSI of corn starch extruded when moisture content increased from 40 to 70% [Seker and Hanna, 2005]. Natabirwa et al. [2018] reported that WAI value of bean extrudates increased with increases in feed moisture, except at feed moisture above 19% [Natabirwa et al., 2018]. In our study, feed moisture content interval was higher than 19% and barrel temperature was lower than <100 °C for non-expanded half product, hence, similar effect on WAI may not be observed. High moisture content in extrusion process acts as a plasticizer thus decreases the water absorption by starch molecules. A direct comparison of WAI values in the literature is difficult due to the difference in processing conditions and raw material used. The decrease in WSI with increase in feed moisture content was in agreement with previous works [Yagci and Gogus, 2008], [Ding et al., 2005]. It was proposed that extrusion at low moisture conditions is caused greater shear degradation of starch.

The addition of XG to corn grits increased the WAI values (Figure 3.1b). XG may have interacted with the starch components of the corn during extrusion and resulted in increase of WAI. It is seen the literature that starch can participate in synergistic interactions with hydrocolloid. These interactions have been shown to take place between amylose of solubilized starch and the hydrophilic portion of the hydrocolloid [Ari Akin and Miller, 2017], [Adamu and Jin, 2002], [Philips and Williams, 1995]. Apart from this interaction, XG may have increased WAI due to its fiber structure. Hashimoto and Grossmann [2003] reported that increased fiber level caused to higher water absorption capacity of extrudates [Hashimoto and Grossmann, 2003]. Increase in WAI values is consisted with the previous findings [Hanna et al., 1997], [Maga and Fapojuwo, 1988]. Maga et al. [1991] reported that gellan gum addition increased WAI values of extrudates [Maga et al., 1991]. Another study about fenugreek gum addition to pea-rice blends shown that WAI values affected the by increasing inclusion level of gum [Ravindran et al., 2011]. Also, increase in WAI values may be due to interact or react of XG with other components of corn such as proteins and lipids [Singh and Singh, 2004]. These results were indicated that the hydrocolloid is still capable of absorbing water. WSI measures the degree of starch conversion which is the amount of soluble polysaccharides released from the starch components after extrusion [Ding et al., 2005]. WSI of half products was negative influenced by XG level. The results of the present study are also in line with literature

[Rashid et al., 2015]. This pattern is difficult to explain although it was in compliance with the observations on WAI results. Structural modifications involving fiber would be responsible for increasing WAI and decreasing WSI [Hashimoto and Grossmann, 2003].

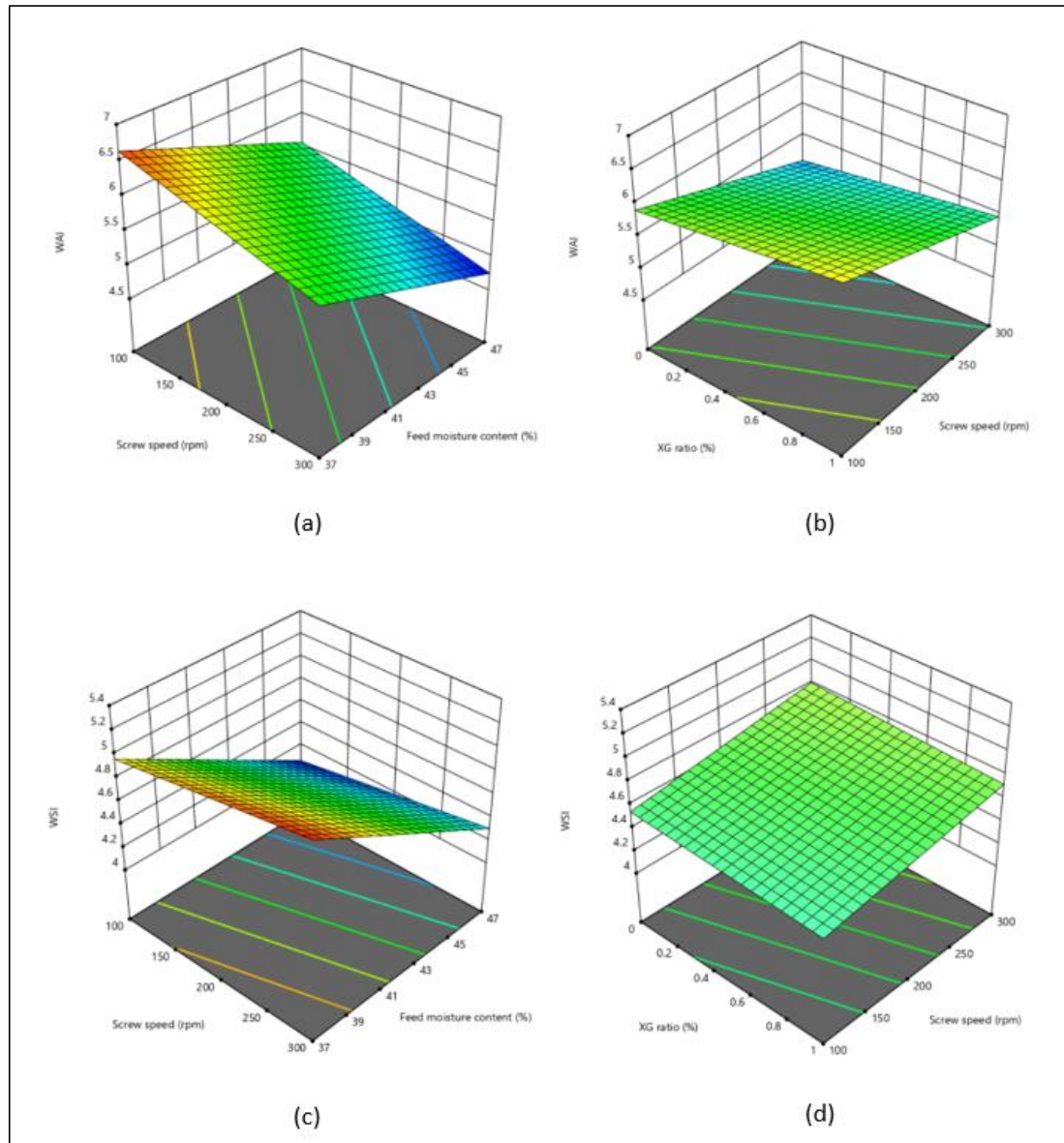


Figure 3.1: Response surface diagrams illustrating the effects of screw speed, feed moisture and XG level on water absorption index (a-b), water solubility index (c-d)

3.2.2. Effects of Extrusion Conditions on Degree of Starch Gelatinization

DG of half products ranged from 46.33% to 63.36% (Table 3.2). The coefficient estimates of DG model (Table 3.4) showed that screw speed had a positive quadratic effect whereas feed moisture had a negative linear effect. Figure 3.2a shows the effect of screw speed and feed moisture on the DG of half products. Increasing XG level, increased DG significantly in linear term and decreased in quadratic term. Interaction of screw speed and feed moisture had negative effect on the DG and interaction of screw speed and XG had a positive effect on the DG (Table 3.4).

Gelatinization, the conversion of raw starch to a cooked and digestible material by the application of water and heat, is one of the important effects that extrusion has on starch components of foods [Ding et al., 2005]. When temperatures in last two zones were $<100^{\circ}\text{C}$, half product with low DG was obtained and this was required to produce flat, thin and bubble-free extrudates. DG decreased with increases in screw speed, expect at screw speed above 200 rpm, at lower XG content. Lower screw speed increases input of thermal energy due to high residence time and so level of starch degradation may be increase similar to WAI values [Yagci and Gogus, 2008]. However, DG increased with increases in screw speed from 200 to 300 rpm (Figure 3.2a). Higher screw speed than 200 rpm enhance the starch gelatinization due to increase in shear degradation effect. Sobukola et al. [2013] and Suknark et al. [1999] stated that increased shear effect at higher screw speeds lead to higher gelatinization of starch molecules in extrusion cooking [Sobukola et al., 2013], [Suknark et al., 1999].

Feed moisture effect on the DG and WAI were similar in this study. This was an expected result because WAI can be used as gelatinization index [Ding et al., 2006]. High moisture content in extrusion process acts as a plasticizer thus reduces full expansion and degradation granules of starch [Liu et al., 2011].

The addition of XG up to 0.5% increased DG (Figure 3.2b). The higher DG with addition of XG may be attributed to water retention capacity providing better interaction with the starch components of corn grits during extrusion. Singh and Singh [2004] reported that increase in water retention capacity with addition of gum (sodium alginate) during extrusion process [Singh and Singh, 2004]. Similar results for addition of various hydrocolloids were found [Maitre et al., 2017], [Arambula et al.,

1999]. However, excess distribution of hydrocolloid in the starch intergranular spaces may reduce interparticle contacts or restrain the hydrogel network [Zhuang et al., 2010]. Therefore, XG ratios above 0.5%, might interrupt the rate of hydration and gelatinization of starch molecules.

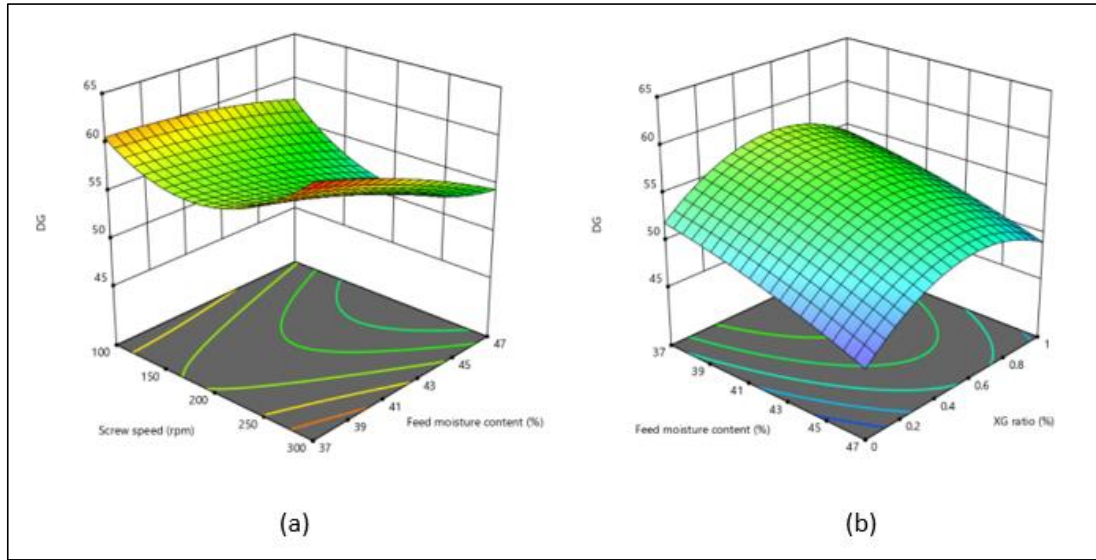


Figure 3.2: Response surface diagrams illustrating the effects of screw speed, feed moisture and XG level on degree of gelatinization (a-b).

3.2.3. Effects of Extrusion Conditions on Water Activity

Water activity is defined as the ratio between the pressure of a solution to that of pure water under the same condition [Koop et al., 2000]. It measures the existing free water in a material and it is important factor for shelf life. Food products with a low a_w have a longer shelf life whereas materials with a high a_w are at a greater risk for quick spoilage. Lowe and Kershaw [1995] found that water activity of less than 0.6 can guarantee a longer shelf life for most food products [Lowe and Kershaw, 1995]. In this study, a_w values of half products ranged between 0.62 ± 0.005 and 0.80 ± 0.005 (Table 3.2). Statistically, F-values in Table 3.3 shown that the effect of linear and quadratic terms of screw speed, feed moisture and XG addition was significant ($p \leq 0.01$). Furthermore, interaction of screw speed and XG had a positive effect on the a_w , and interaction of feed moisture and XG had negative effect on the a_w (Table 3.4).

Increasing in screw speed decreased the a_w values (Figure 3.3a). This result was in agreement with the literature [Fallahi et al., 2013]. Higher screw speed enhances

water absorption by starch due to increase in shear degradation effect. Thus free water in half products was decreased.

The decrease in a_w values with increasing feed moisture content could be attributed to the dispersion of starch and other water binding components in excess water, the increased absorption of free water. Hence, a_w values of half products may reduce.

The addition of XG up to 0.5% decreased a_w values (Figure 3.3b). The hydrophilic character of gums is expected to modify water absorption in extrusion process. He et al. [2015] reported that water absorption values were particularly increased by XG addition to corn starch [He et al., 2015]. This could be because a higher hydrocolloid–starch interaction retain more water molecules. Thus a_w value of corn half products showed a tendency to decrease. However, a_w value increased with increases in XG ratio above 0.5%. Similar effect of XG ratio on DG was also observed in this study. Excess distribution of hydrocolloid in the intergranular spaces may reduce the interparticle contacts or interrupt water binding capacity of starch [Zhuang et al., 2010].

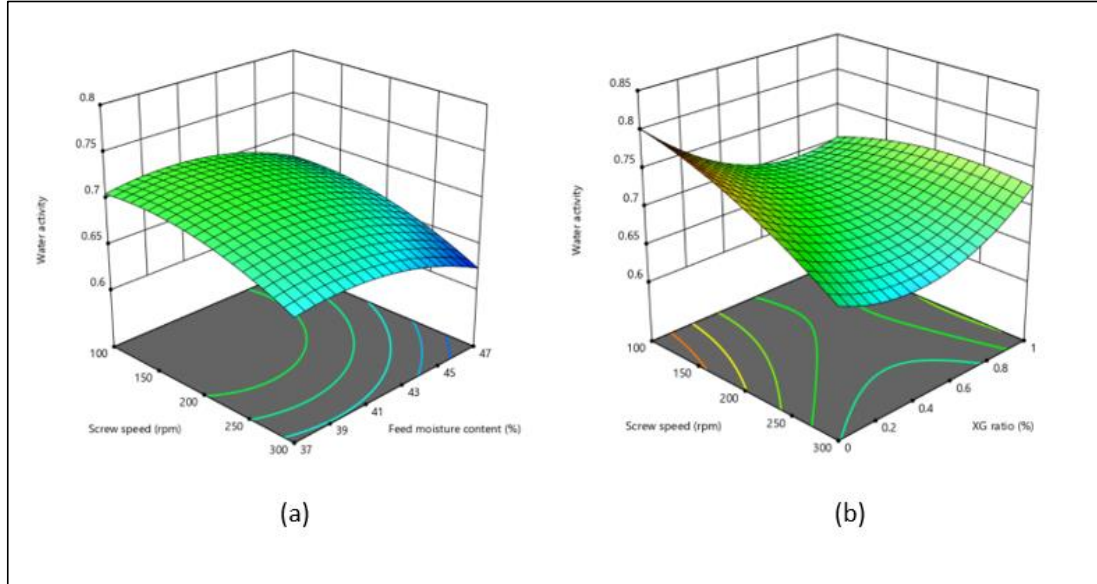


Figure 3.3: Response surface diagrams illustrating the effects of screw speed, feed moisture and XG level on water activity (a-b).

3.2.4. Effects of Extrusion Conditions on Specific Volume

Specific volume is a measurement of volumetric expansion, which is the sum of radial and axial expansions. It is directly related with “degree of cooking” for expanded extrudates [Gonzalez et al., 2004]. In this study, specific volume of half products ranged between 1.15 ± 0.070 and 2.18 ± 0.094 mL/g (Table 3.2). The reason for low specific volume values may be temperature under 100 °C in last two zones for production of flat, thin and bubble-free half products. According to Table 3.4, negative linear and quadratic effects of screw speed, negative interaction effects of screw speed with feed moisture on the specific volume was observed. Statistically, feed moisture had a significant negative linear effect on the specific volume whereas XG level significant positive linear and negative quadratic effect on the specific volume. Furthermore, interaction of screw speed and XG had positive effect on the specific volume.

Increasing in screw speed decreased the specific volume of half products (Figure 3.4a). This result was in agreement with the literature for corn extrudates [O’shea, Arendt, and Gallagher, 2014]. Xie et al. [2012] reported that an increase of the screw speed increases the intensity of mechanical treatment, which results in greater macromolecular degradation and thus lower melt viscosity and this mechanical treatment was more important at lower temperature [Xie et al., 2012]. In our study, lower melt viscosity may be result in decrease of specific volume of half products at lower barrel temperature (<100 °C).

Specific volume of half products was found to be most dependent on feed moisture. Specific volume of the half products decreased when moisture content increased (Figure 3.4a). Similar tendency was obtained by Perez et al. [2008] for corn extrudates [Perez et al., 2008]. The high dependence of specific volume on feed moisture would reflect its influence on elasticity characteristics of the starch-based material. An increase in moisture may be reduce melt viscosity and, as melt viscosity decreases, product structure becomes thinner.

The addition of XG up to 0.5% increased specific volume of half products. However decrease in specific volume with the addition of XG above 0.5% was observed (Figure 3.4b). Thakur and Saxena [2000] reported some hydrocolloids (XG, carboxymethyl cellulose, guar and arabic gum) showed negative quadratic effect on expansion of the extrudates [Thakur and Saxena, 2000]. The decrease in the specific

volume with addition of XG may be attributed to the interaction of the gum with the corn starch which may have inhibited the volumetric expansion. Furthermore, XG ratios above 0.5% decreased DG and this may reduce the volumetric expansion.

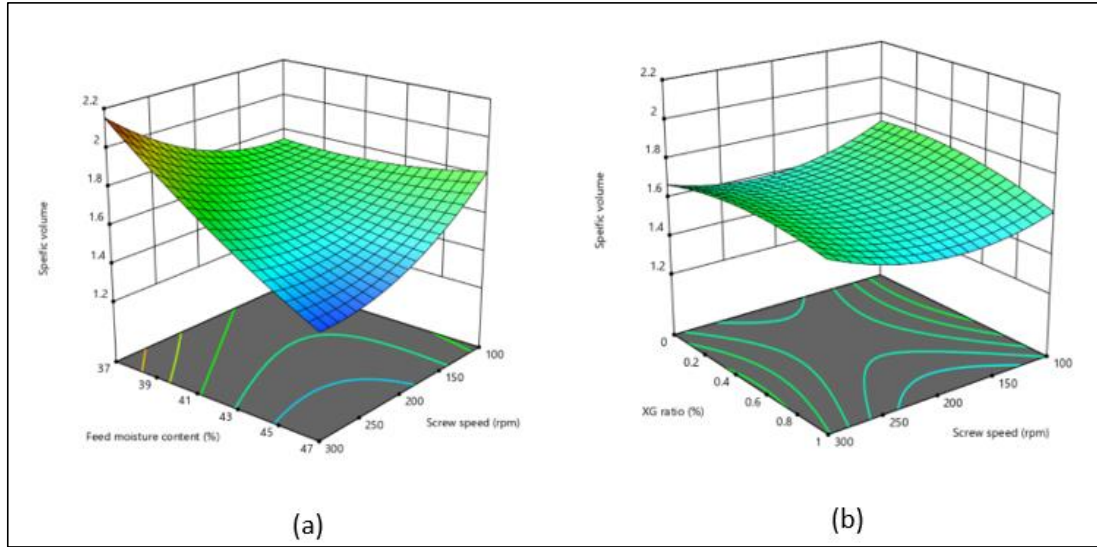


Figure 3.4: Response surface diagrams illustrating the effects of screw speed, feed moisture and XG level on specific volume (a-b).

3.2.5. Effects of Extrusion Conditions on Color Properties

Color is an important physical property for extrudate products. Statistically, positive linear effect of screw speed on the L values of half product was observed. Interaction of screw speed and XG had a negative effect on the L values. Feed moisture was an important variable for the half product L value, as its positive linear and negative quadratic terms were significant.

Increasing screw speed caused higher L values or lighter half product (Figure 3.5a). This result is in accordance with previous work in the literature [Sun and Muthukumarappan, 2002]. Increasing screw speed decreased the residence time. The shorter residence time would decrease the heating time of the dough inside the extruder and result in lighter products [Liu et al., 2000]. Screw speed had a linear negative effect on redness of half products (Figure 3.5c). Positive linear coefficient of screw speed indicated that yellowness (b values) increased with increasing the screw speed (Figure 3.5e). This effect of screw speed on a and b values can also be linked to lower residence time in barrel, thus less cooking of sample [Gulati et al., 2016].

Increasing the moisture content resulted in higher L and b values (Figure 3.5). At higher moisture conditions, the effect of temperature is reduced which results in lighter sample. Interaction of screw speed and feed moisture had positive effect on L and b values of half products. However feed moisture had positive effect on a values (Figure 3.5e). Liu et al. [2000] reported that increasing feed moisture content increased the redness of oat extrudates due to reduced expansion. We observed that increasing the feed moisture reduced volumetric expansion and correspondingly increased the a values.

XG level significant positive quadratic effect on the lightness and yellowness of half products. Brown et al. [2015] stated that increasing hydrocolloid level (XG and carboxymethyl cellulose) resulted in lighter extrudates. Interaction of feed moisture and XG level had positive effect on the lightness and yellowness of half products. Increasing XG level, decreased a values significantly in linear term. Zhuang et al. [2010] reported that addition of gum reduced redness of rice extrudates. Decrease in redness with addition of gum may be attributed to providing water retention capacity and decrease the cooking temperature during extrusion.

3.2.6. Optimization and Model Validation

From the numerical analysis, best extrusion conditions were 300 rpm screw speed, 47% feed moisture and 0.44% XG content having a desirability value 0.91. Predicted values of WAI (4.78), WSI (4.32), DG (54.15%), a_w (0.62), specific volume (1.52), L (64.18), a (6.16) and b (29.64) were found. Extrusion was carried out using optimum processing conditions and experiments were performed. The experiments carried out triplicate and the average values with standard deviation were presented in Table 3.5. The predicted values registered non-significant differences from the experimental results ($p > 0.05$). This shows that suitability of the models to predict various responses was confirmed.

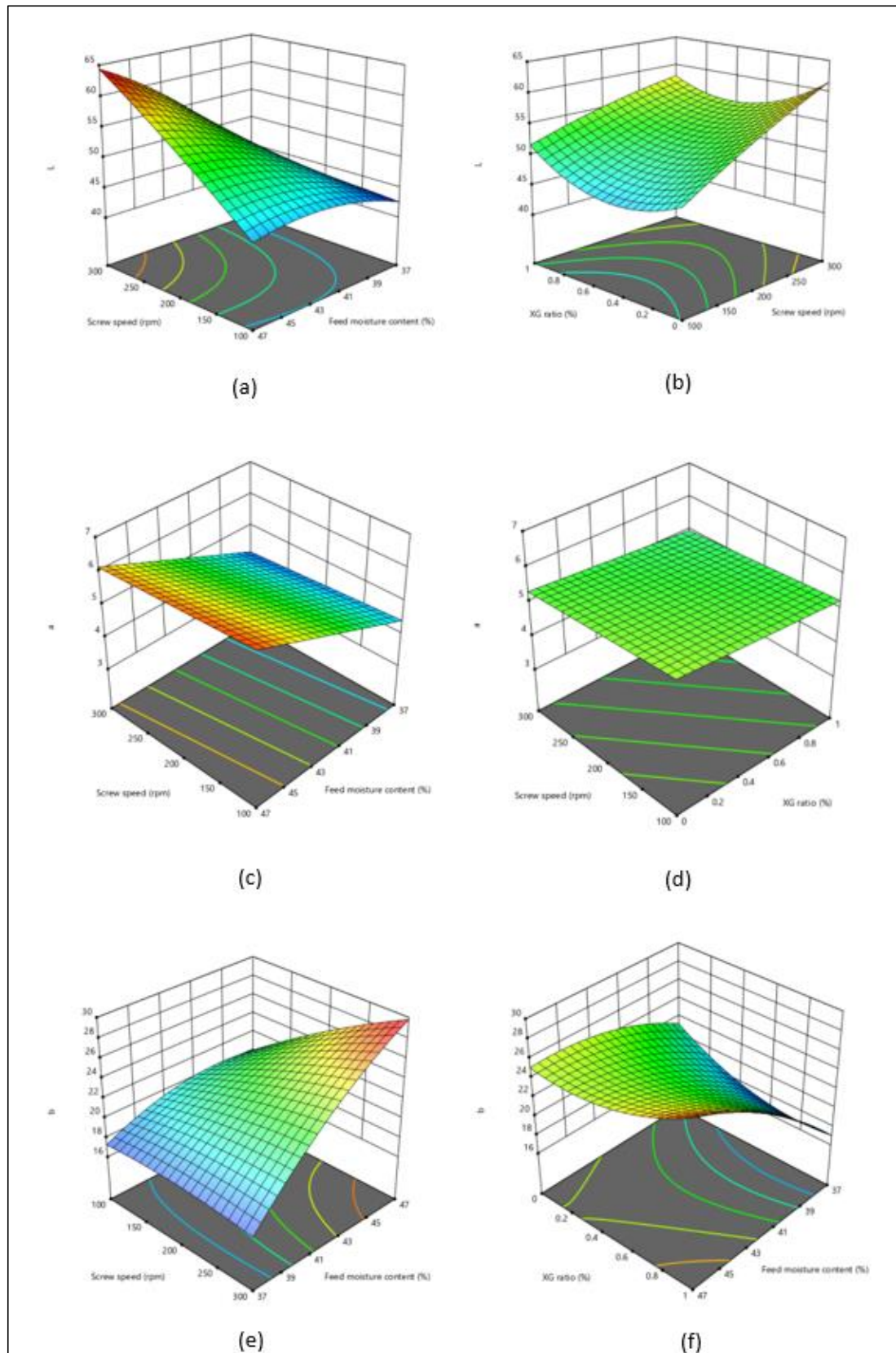


Figure 3.5: Response surface diagrams illustrating the effects of screw speed, feed moisture and XG ratio on L (a-b), a (c-d) and b values (e-f) of corn half products.

Table 3.5: Predicted and experimental values at optimum processing conditions.

Responses	Predicted values	Experimental values ^a
WAI	4.78	4.68 ± 0.18
WSI (%)	4.32	4.28 ± 0.03
DG (%)	54.15	55.76 ± 0.58
a _w	0.62	0.63 ± 0.01
Specific volume (mL/g)	1.52	1.44 ± 0.03
<i>L</i>	64.18	61.90 ± 0.73
<i>a</i>	6.16	5.98 ± 0.34
<i>b</i>	29.64	27.73 ± 1.12

^a Mean of three replications with standard deviations

4. EFFECTS of EXTRUSION CONDITIONS on EXPANDED CORN HALF PRODUCTS by FRYING

Snacks which defined as a small and lightweight food are commonly consumed irrespective of age, gender or social status. Potato chips and masa products such as corn chips are produced by frying of dough. However moisture content of dough is high and it requires great drying time, drying cost and process time with lower production capacity in case of preparation from dough. Indirect expanded products such as third generation snacks are produced by extrusion of starchy food at moderate barrel temperature and low pressure to avoid expansion. Final expansion of dried half products can be performed by deep fat frying process.

Frying can be considered a complex process due to coupled heat and mass transfer between food and frying medium [Farkas et al., 1996]. Temperatures used for frying are above the boiling point of water, generally in the 150–200 °C range. Heat of oil evaporate water in product and oil is absorbed, thus expansion is achieved. Oil absorption of half products during frying process is affected by initial half product composition, extrusion conditions and frying process conditions such as frying time, oil type, oil temperature [Noorakmar et al., 2012], [Osman et al., 2000]. In addition to this, surface structure of food directly affects the oil absorption. In terms of health, lower oil content in deep fat fried foods is very important for the consumer. Use of additives can be an effective and functional method to obtain low-fat fried extrudates. Addition of hydrocolloids such as hydroxypropyl methyl cellulose, carboxyl methyl cellulose, guar gum, gellan gum and XG are known to restrain oil intake of deep fat fried snacks [Bajaj and Singhal, 2007], [Annapure et al., 1999].

Osman et al. [2000] investigated that effect of two extrusion variables, screw speed and extrusion temperature, on oil absorption characteristics of a deep-fat-fried extrudate, when other process variables such as extrudate moisture and frying conditions are held constant [Osman et al., 2000]. According to the their results, lowest oil absorption (19.9%) was obtained with an extruder screw speed of 218.6 rpm and a cooking temperature of 117.8 °C. They reported that water absorption index was reached a maximum at a screw speed of 221.9 rpm and a cooking temperature of 109 °C. Suknark et al. [1999] studied about production of two high protein snack

products from tapioca starch and fish muscle, and from tapioca starch and partially defatted peanut flour (PDPF) using a twin screw extrusion process followed by deep fat frying and they found that increasing temperature and screw speed resulted in increased expansion, and decreased bulk density and shear strength [Suknark et al., 1999]. Microstructural analysis of the extrudates also used to gain an understanding of what happens inside the extrudates after the process of frying during storage. Dar et al. [2014] found that presence of porous structure and air cells confirm the efficient removal of moisture from the surface of extrudates during the process of frying which may be the reason for the desirable crispiness of the extrudates and decreased hardness [Dar et al., 2014].

Noorakmar et al. [2012] studied that effects of sweet potato flour addition to tapioca starch on the physical properties of fried extruded fish crackers and they reported that bulk density and WSI increased with the increase in orange sweet potato flour addition [Noorakmar et al., 2012]. However, WAI, linear expansion, expansion ratio, volume expansion and oil absorption decreased as the amount of orange sweet potato increased.

The use of hydrocolloids in directly expanded extrudates has been reported in literature. However any study about effect of hydrocolloid incorporation into feed used for third generation snacks (expanded by frying) has not so far been reported. Osman et al. [2001] studied effects of screw speed and temperature of extruder on oil absorption but there is not study about effect of moisture content of feed during extrusion on quality characteristics of fried extrudates [Osman et al., 2001]. In this respect, this section of thesis about effects of feed moisture level and XG addition to half product on oil absorption property can be contribute to further explanation about mechanism of oil absorption of extrudates during frying process and their quality characteristics.

4.1. Materials and Methods for Fried Half Products

Half products were deep fried at 180 °C for 60 second in sunflower oil using an electronic fryer (SDF-3817, Sinbo, Turkey) for expansion. After frying process, third generation corn snacks were drained on paper towels to remove excess oil. They were stored in zip lock bags (in glass containers) at room temperature until further experiments.

4.1.1. Experimental Design

The same experimental design as in section 3.1.1 was used.

4.1.2. Expansion Index

The EI was calculated by dividing the average transversal area of the fried (expanded) products by the average transversal area of the half (non-expanded) products. Results were given as the mean of ten replicates.

4.1.3. Oil Content

Oil content of ground fried extrudates was determined in triplicate by Soxhlet extraction system.

4.1.4. Textural Measurement

A texture analyzer (Lloyd Ins., England, UK) with a 250 N load cell, coupled with Nexygen Plus software (Lloyd Ins, England, UK) was used for determination of hardness of fried products. A three-point bending method was performed on a fried snack (8 cm length). Snack was compressed with a blade 3 mm thick at a crosshead speed of 2 mm/s. A force-time (distance) curve was generated and the highest value of force was taken as the value for hardness. Measurements were performed during the same day to prevent significant moisture pick-up from the air. Results were given as the mean of ten replicates.

4.1.5. Color Properties

Color properties of corn snacks were determined by measuring with Konica Minolta CR-400 Chroma Meter and color values were expressed by Hunter L , a and b values. The instrument was calibrated before the sample measurement with a standard white tile. Results were given as the mean of ten replicates. Browning index (BI) (Equation 4.1) were calculated from the L , a and b values;

$$BI = \frac{100(x - 0.31)}{0.17} \quad (4.1)$$

Where

$$x = \frac{(a + 1.75L)}{(5.654L + a - 3.012b)} \quad (4.2)$$

4.1.6. Microstructure Properties

Microstructures of fried corn snacks were observed by scanning electron microscopy (SEM) (XL-30 SFEG, Philips, Eindhoven, Holland). Surface oil was removed from fried corn snacks by using petroleum ether for at least 24 h before the analysis. Because it can be negatively affect the quality of the images obtained from SEM. Previous studies shown that removal of surface oil with petroleum ether did not have any effect on the sample structure for the SEM analysis [Primo-Martin and Van Dauter, 2011]. Samples were dried after treatment with petroleum ether. For external surface examination, samples pieces were coated with gold by using SEM sputter coater (Model SC7620, Quorum Technologies Ltd., Lewes, UK) before the analysis.

4.1.7. Statistical Analysis and Optimization

Statistical significance of each factor was determined by ANOVA technique. Regression analysis was carried out to fit second-order polynomial model with using the Design-Expert software. Model development was applied by using significance levels at $p < 0.01$, $p < 0.05$ and $p < 0.1$. The goodness of fit of predictive models was estimated using lack of fit test, R-squared and adjusted R-squared values. Addition to ANOVA analysis, standardized Pareto charts (at $p = 0.05$) were generated using with Statgraphics program (version 18 X64, Statistical Graphics Corporation, Rockville, MD, USA) for better observation the magnitude and importance of an effect of factor on the each response. Three-dimensional surface plots were formed considering two process variables at a time (the other process variable is kept at the second level).

Numerical optimization approach was selected to optimization of multiple responses. Color, hardness and oil content were important factors affect the quality of third generation snacks. Hence, optimal extrusion conditions were identified to acquire the lowest values for hardness, oil content and BI and highest values for yellowness and lightness. Other responses and process variables were kept in range during optimization. Experiments at optimum process conditions were performed in triplicate. Experimental data and predictive values obtained from the models were compared.

4.2. Results and Discussion for Fried Half Products

Experimental results (in Table 4.1) were fitted to Equation (3.1) to develop models for all the responses. Table 4.2 shows the coefficient of equations, *F*-value, *R*-squared and adjusted *R*-squared values of developed models. All models for seven responses were found as statistically significant, with $p \leq 0.001$. Lack of fit values of all models were non-significant, with $p > 0.05$. Lack of fit test compares the deviation of actual points from the fitted surface, relative to pure error. If a model has non-significant lack of fit value, it can be used as predictive model. Hence, it can be concluded that developed models in our study sufficiently explained the relationship between responses and process variables. Figure 4.1 shows Pareto charts for each response. There is a reference line in these charts. If bars extends the reference line, effect is potentially important and length of each bar shows magnitude of the effects for the main factors or their interactions. The sign of the bar (grey (+) or blue (-)) indicates that the factor has a positive or negative effect on the response.

Table 4.1: Coded, uncoded variables for the Box-Behnken design and experimental results of fried half product responses.

Runs	Coded variables			Uncoded variables			Experimental Results						
	X ₁	X ₂	X ₃	Screw speed (rpm)	Feed moisture (%)	XG ratio (%)	EI	Oil content (%)	Hardness (N)	<i>L</i>	<i>a</i>	<i>b</i>	BI
1	0	0	0	200	42	0.5	1.69 ± 0.06	8.46 ± 0.21	23.88 ± 3.57	49.45 ± 0.84	12.58 ± 0.84	21.60 ± 0.53	74.87 ± 2.38
2	+1	+1	0	300	47	0.5	1.73 ± 0.06	8.87 ± 0.11	7.58 ± 0.47	51.29 ± 0.67	11.83 ± 0.22	22.36 ± 0.40	73.01 ± 1.14
3	0	+1	-1	200	47	0	1.92 ± 0.07	8.48 ± 0.11	10.87 ± 1.15	51.62 ± 0.86	10.62 ± 0.97	21.10 ± 0.69	66.68 ± 2.93
4	0	+1	+1	200	47	1	1.73 ± 0.02	8.82 ± 0.23	11.5 ± 0.74	56.62 ± 1.48	10.80 ± 0.67	23.56 ± 0.27	66.78 ± 2.55
5	-1	0	+1	100	42	1	1.52 ± 0.07	6.91 ± 0.11	22.85 ± 0.34	50.42 ± 1.05	10.62 ± 0.77	20.94 ± 0.48	68.11 ± 2.55
6	0	0	0	200	42	0.5	1.71 ± 0.03	8.46 ± 0.27	24.84 ± 1.33	49.09 ± 0.80	12.51 ± 0.48	21.97 ± 0.60	76.73 ± 1.75
7	0	0	0	200	42	0.5	1.67 ± 0.02	8.60 ± 0.07	24.59 ± 3.42	49.71 ± 1.04	12.39 ± 0.63	21.99 ± 0.63	75.44 ± 2.17
8	0	0	0	200	42	0.5	1.69 ± 0.03	8.61 ± 0.06	24.9 ± 0.92	49.55 ± 0.72	12.55 ± 0.49	21.91 ± 0.50	75.70 ± 1.91
9	+1	0	+1	300	42	1	1.79 ± 0.03	9.25 ± 0.29	27.44 ± 3.19	57.17 ± 0.89	9.90 ± 0.66	24.02 ± 0.51	66.13 ± 1.35
10	0	-1	-1	200	37	0	1.45 ± 0.05	7.57 ± 0.18	33.28 ± 2.47	51.06 ± 1.33	12.16 ± 0.57	22.09 ± 0.68	72.99 ± 1.52
11	+1	0	-1	300	42	0	1.81 ± 0.02	7.56 ± 0.10	22.13 ± 0.44	50.63 ± 0.92	11.56 ± 1.07	21.99 ± 0.53	72.56 ± 2.69
12	-1	+1	0	100	47	0.5	1.79 ± 0.05	10.05 ± 0.16	13.50 ± 0.43	50.24 ± 1.53	10.87 ± 1.49	21.07 ± 0.73	69.20 ± 3.02
13	-1	-1	0	100	37	0.5	1.33 ± 0.04	6.81 ± 0.25	30.13 ± 1.08	45.94 ± 0.84	12.90 ± 0.56	20.20 ± 0.35	77.26 ± 1.55
14	0	-1	+1	200	37	1	1.70 ± 0.04	7.19 ± 0.24	36.80 ± 0.89	51.52 ± 1.02	10.27 ± 0.73	21.41 ± 0.67	67.32 ± 2.44
15	-1	0	-1	100	42	0	1.54 ± 0.02	8.55 ± 0.16	19.89 ± 1.59	49.88 ± 1.03	10.80 ± 1.24	21.10 ± 0.85	69.81 ± 3.91
16	0	0	0	200	42	0.5	1.74 ± 0.02	8.52 ± 0.16	23.78 ± 0.92	49.66 ± 0.74	12.44 ± 0.60	21.80 ± 0.52	74.96 ± 1.87
17	+1	-1	0	300	37	0.5	1.88 ± 0.06	8.88 ± 0.36	42.26 ± 1.44	50.69 ± 1.29	11.06 ± 0.52	23.28 ± 0.56	76.08 ± 1.42

4.2.1. Effects of Extrusion Conditions on Expansion Index

Expansion of product provides open cell structure and affect quality of products. EI of fried extrudates ranged from 1.33 ± 0.04 to 1.92 ± 0.07 depending on extrusion conditions (Table 4.1). According to Table 4.2, screw speed and feed moisture level significantly affected the EI positively, but interaction of these two factor affect the EI negatively. As illustrated in Figure. 4.1a, the most pronounced effect on EI was belonged to screw speed. EI increased with increase in screw speed (Figure 4.2a). Thickness of extrudates have critic effect on volumetric expansion of third generation snack [Suknark et al, 1999]. The higher screw speed can be caused thinner half product. In our previous work, increasing screw speed resulted decrease in specific volume of half products [Gumusay et al., 2019]. In other words, thickness of extrudates decreased and so EI of corn snacks when screw speed increased. This mechanism could be clarified by the deep fat frying applied after extrusion process. Samples were immersed in oil at 180 °C and heat transfer occurred from the oil to the food during frying. Water vaporization takes place from surface of food and oil migrates into the places left by the water evaporation. Colonna et al. [1989] reported that the expansion of extrudates resulted from rapid water evaporation inside starch matrix when the frying temperature become higher than the glass transition temperature [Colonna et al., 1989]. In this study, midpoint temperature of thin extrudates may have increased more quickly and may have reached the water vaporization point. However, thick half products may not expanded due to slow rise of midpoint temperature. Suknark et al. [1999] were reported same results for tapioca snacks [Suknark et al, 1999]. Similar effect of feed moisture level on EI was observed, thin extrudates prepared with high moisture content have also high EI or more expansion after frying with same reason explained above (Figure 4.2).

Table 4.2: Coefficients from the predictive model equation for fried half product responses.

Variables	Expansion Index	Oil Content (%)	Hardness (N)	<i>L</i>	<i>a</i>	<i>b</i>	Browning Index
Intercept	1.69	8.53	23.54	49.49	12.49	21.91	75.54
X_1	0.13***	0.28***	1.63***	1.66***	-0.10*	1.041***	ns
X_2	0.10***	0.72***	-12.38***	1.32***	-0.28***	ns	-2.25***
X_3	ns	ns	1.55***	1.57***	-0.44***	0.46***	-1.71***
X_1X_2	-0.15***	-0.81***	-4.51***	-0.93***	0.70***	-0.45***	1.25**
X_1X_3	ns	0.83***	ns	1.50***	-0.37***	0.55***	-1.18**
X_2X_3	-0.11***	0.18**	ns	1.14***	0.52***	0.78***	1.44**
X_1^2	ns	ns	ns	ns	-0.54***	ns	ns
X_2^2	ns	ns	ns	0.36*	-0.29***	ns	-1.18**
X_3^2	ns	-0.55***	ns	2.85***	-1.24***	ns	-5.92***
Model (<i>F</i> -value)	48.74 ^a	83.55 ^a	218.39 ^a	84.17 ^a	80.51 ^a	44.21 ^a	33.07 ^a
r^2	0.9663	0.9908	0.9924	0.9908	0.9904	0.9637	0.9770
Adjusted r^2	0.9461	0.9789	0.9879	0.9791	0.9781	0.9419	0.9475
Lack of Fit (<i>p</i> -value) ^b	0.2214	0.0671	0.0624	0.1001	0.0539	0.1531	0.2426

X_1 , Screw speed (rpm); X_2 , Feed moisture (%); X_3 , XG ratio (%).

^a Significant at a $p < 0.0001$ level.

^b want the selected model to have non-significant lack of fit ($p > 0.05$)

*Significant at $0.05 \leq p < 0.1$.

**Significant at $0.01 \leq p < 0.05$.

*** Significant at $p < 0.01$; ns, non-significant (p -values are greater than 0.1).

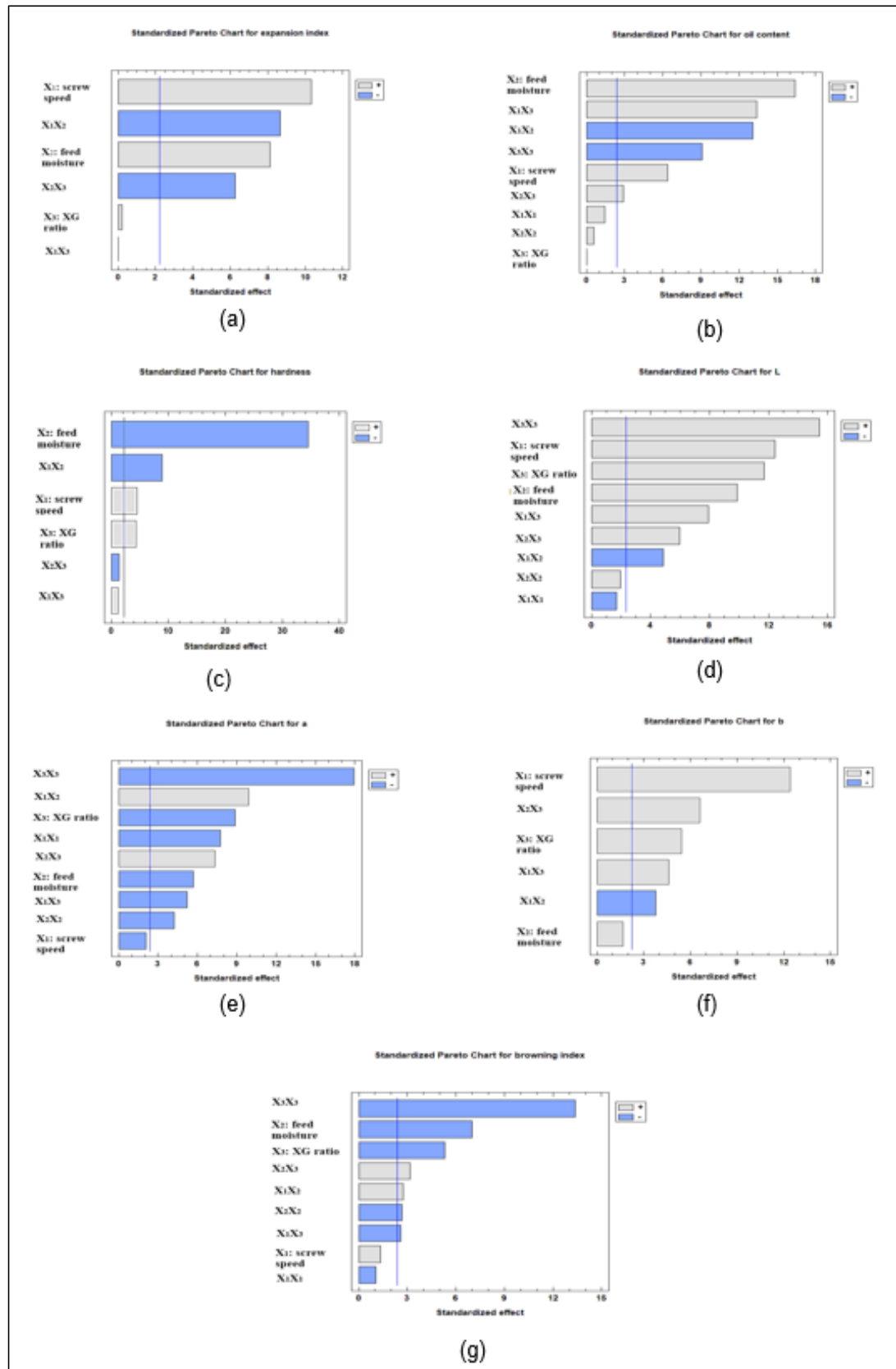


Figure 4.1: Pareto charts for the standardized effects: expansion index (a), oil content (b), hardness (c), L value (d), a value (e), b value (f) and browning index (g).

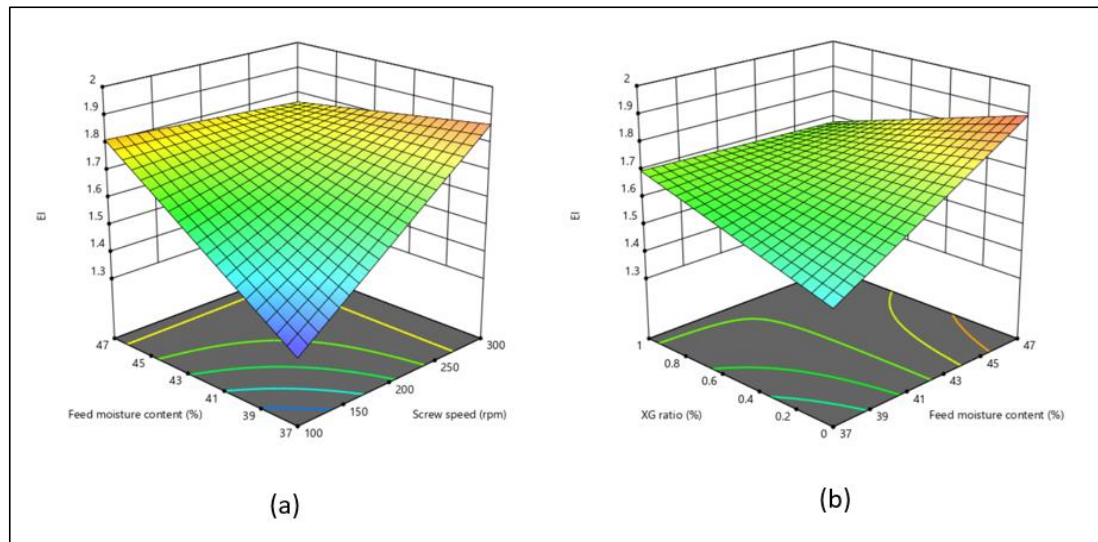


Figure 4.2: Three dimensional surface diagrams illustrating the effects of screw speed, feed moisture and XG ratio on expansion index (a-b).

4.2.2. Effects of Extrusion Conditions on Oil Absorption

Oil content of corn snacks were changed from 6.81% to 10.05% due to extrusion conditions (Table 4.1). The Standardized Pareto chart (Figure 4.1b) revealed that the most significant factor on oil content was feed moisture content and oil content of corn snacks was significantly affected by interaction of all factors. The coefficients estimated from Table 4.2 demonstrated that screw speed and feed moisture level had a positive linear effect but XG ratio had a negative quadratic effect on oil content of corn snacks (Figure 4.3b).

The increase in oil absorption with screw speed and feed moisture level could be due to expansion of the final product. Surface area for oil transfer increased when expansion of extrudate increased, hence oil absorption on the surface increased. Noorakmar et al. [2012] reported that oil absorption was greater for more expanded products in their studies. It could be attributed to more oil engaged in the surface layer of the bigger air cells when expansion occurs [Suhaila and Norhasyimah, 1994]. Osman et al. [2000] found that increasing screw speed increased oil absorption of fried extrudates [Osman et al., 2000]. In addition to this, increase of screw speed resulted in increase of starch gelatinization of corn half products in our previous work [Gumusay et al., 2019]. Thus, larger air cells can be formed with sufficient starch gelatinization during extrusion and these cells would lead to absorption more oil during frying.

XG addition to corn snack showed significant quadratic effect on reduction of oil content (Figure. 4.3b and Table 4.2). Oil absorption during frying depends on surface characteristic. Hydrophobic character of surface causes to increase the oil absorption. XG is known as a hydrophilic colloid. Decrease of oil content with addition of XG can be attributed to its hydrophilic character [Pinthus and Saguy, 1994]. On the other hand, in the extrusion process heat and mechanical stresses can be resulted in the thermal gelation of XG, and this can be formed oil-resistant film around the corn half product. This film forming behavior of XG may be another reason for the observed reduction in oil absorption of corn snacks during frying process [Bajaj and Singhal, 2007]. Annapure et al. [1999] researched that effect of various hydrocolloids on the oil absorption properties of a deep fat fried food known as sev (produced with chickpea flour) and they reported that hydrocolloids can be effectively used to preventing the oil uptake in frying of products with dough structure [Annapure et al., 1999].

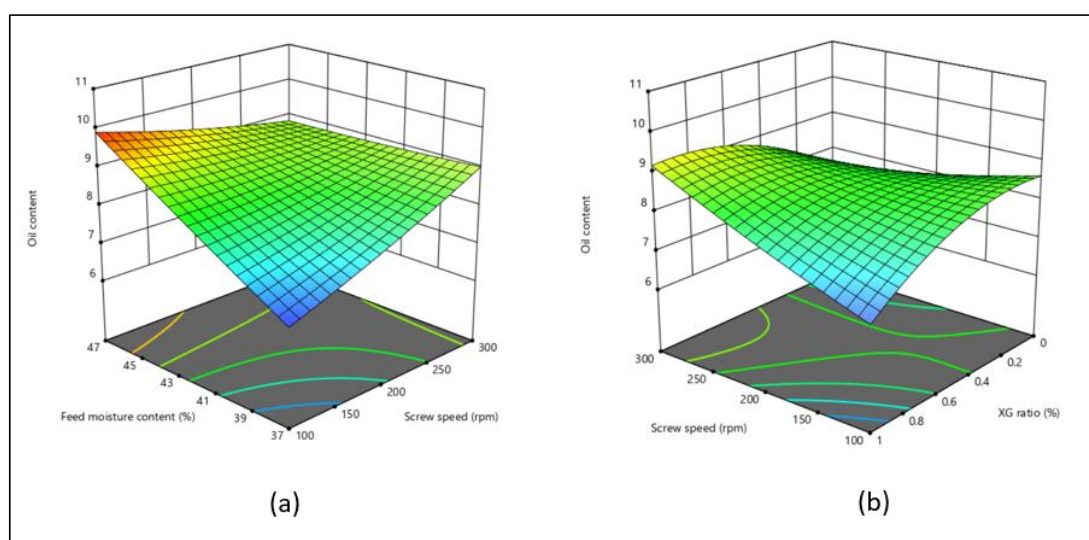


Figure 4.3: Three dimensional surface diagrams illustrating the effects of screw speed, feed moisture and XG ratio on oil content (a-b).

4.2.3. Effects of Extrusion Conditions on Textural Characteristics

Consumers judge quality and freshness of product by its mechanical properties, hardness, which known as resistance to compression, is an important mechanical property. In this study, hardness was measured to investigate the extrusion conditions on the textural properties of corn snacks. Hardness values were ranged from $7.58 \pm$

0.47 to 36.80 ± 0.89 N. Hardness was positively affected by screw speed and XG ratio, according to coefficients of regression model in Table 4.2. As shown in the Figure 4.1c, feed moisture was the most significant (negative) effect on hardness of corn snacks. Interaction of screw speed and feed moisture level had negative effect on hardness.

The hardness is generally associated with cell structure of the product. Increasing moisture content of feed resulted in lower hardness (Figure 4.4a), they also have higher EI. Shear strength shows inverse relationship with expansion of samples [Grenus et al., 1993] and high breaking strength is result of large cells with thicker cell walls [Onwulata et al., 2001]. Fried extrudates prepared with high moisture content have lower hardness due to their higher expansion and lower cell wall thickness. On the other hand, increasing screw speed did not reduced hardness of fried products (Figure 4.4a) even if it provided higher expansion after frying. Increasing screw speed enhanced WSI of extrudates [Gumusay et al., 2019]. Higher specific mechanical energy provided with increased screw speed and shear rate degrades starch molecules [Seker, 2005] and increases WSI that indicates starch degradation. Degradation of starch molecules may have increased hardness of fried samples. Since increasing feed moisture content did not increased WSI in our previous work, elevating moisture content did not enhanced hardness [Gumusay et al., 2019].

Incorporation of XG in corn extrudates resulted in increased the hardness (Figure 4.4b). This result is in accordance with literature study about addition of Mesona Blumes gum to rice extrudates [Zhuang et al., 2010]. They reported that hardness values of extrudates increased to 5% substitution level. Absorption of water with XG may have toughened extrudate and then increased hardness of fried extrudate [Zhuang et al., 2010]. Increase in hardness with XG addition can be depend on interactions between amylose part of starch and the hydrophilic part of the hydrocolloid [Adamu and Jin, 2002]. XG can compete starch for water and addition of XG to feed reduces the water absorption by starch during extrusion process [Borries-Medrano et al., 2016]. This competition may reduce the starch gelatinization and increase the hardness of third generation corn snacks.

XG fiber structure can be affect pore size and the density of extrudate. It is seen the literature that extrudates containing higher content of dietary fiber tend to have lower pore size, higher product density and greater hardness [Ainsworth et al., 2007].

In addition to this, XG addition had negative effect on oil absorption of corn snacks. Dueik et al. [2014] reported that starch based snack with higher oil content were softer.

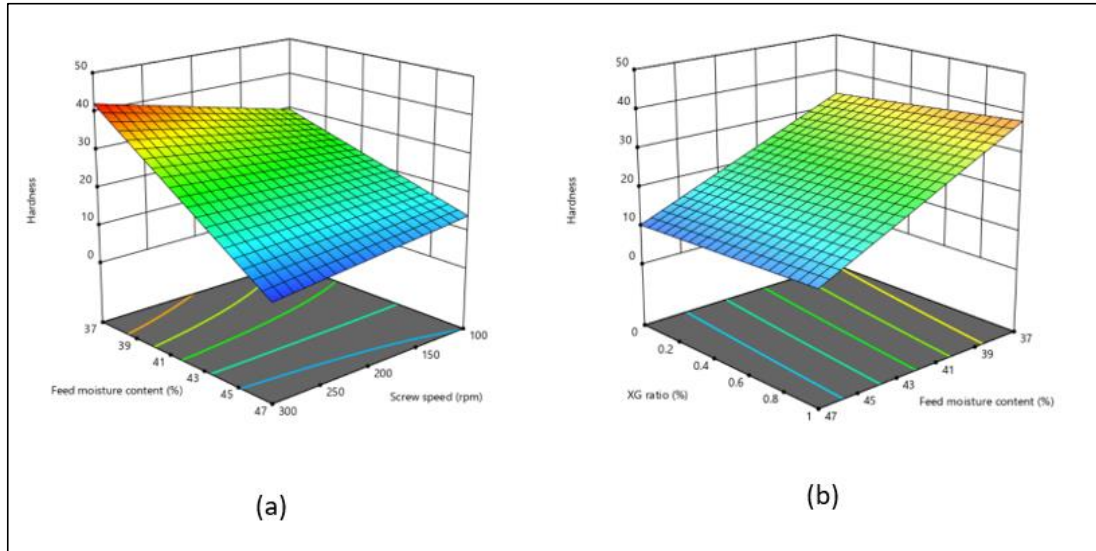


Figure 4.4: Three dimensional surface diagrams illustrating the effects of screw speed, feed moisture and XG ratio on hardness (a-b).

4.2.4. Effects of Extrusion Conditions on Color Characteristics

The color values (L , a and b) and BI of third generation corn snacks are given in Table 4.1. L values, indicating luminescence or lightness, were increased when screw speed, feed moisture and XG levels increased (Figure 4.5). In our previous work, we reported that the increase in lightness of half products with increase in screw speed can be due to lesser residence time at higher screw speed result in lower degradation of the pigments and lighter products [Gumusay et al., 2019]. Screw speed had quadratic negative effect on a values and positive linear effect on b values of corn snacks. This effect of screw speed on redness and yellowness can also be related to lower residence time and less cooking of sample. Lower residence time decreases starch gelatinization. Lower starch gelatinization of the half products prior to frying process can be negatively affected the cooking of sample during frying [Kawas and Moreira, 2001]. The formation of Maillard reaction and the other browning products was decreased. Therefore redness of corn snacks can be negatively affected by screw speed.

L values in corn snacks increased with increasing feed moisture content (Figure 4.5a). However moisture content had negative effect on a and BI values (Figure 4.5).

Higher moisture content reduce temperature of feed during extrusion and Maillard reaction decreases. Excess water can be done plasticizer effect on extrusion process with increase in feed moisture content and water absorption capacity of starch can also be decreased [Liu et al., 2011]. Lower starch gelatinization caused less cooking in frying process. Hence redness and BI decreased and lightness increased in corn snacks.

XG addition to corn snack showed positive quadratic effect on *L* values and positive linear effect on *b* values (Figure 4.5). Redness and BI of corn snacks decreased with increasing XG addition (Figure 4.5). Interactions between XG and amylose of solubilized starch can be result in decrease the starch gelatinization and cooking level of half product [Adamu and Jin, 2002]. Therefore lightness and yellowness positively affected whereas redness and BI negatively affected by inclusion of XG.

4.2.5. Effects of Extrusion Conditions on Microstructure

Examination of surface topography with SEM will provide us the perspective to interpret the effects of process factors. Figure 4.6b shows surface of snack at higher screw speed was rougher, and even some holes appeared. At lower screw speed (Figure 4.6a) surface was smooth. Increasing screw speed provided fried products with higher expansion due to evaporation of water. In addition, oil content of snacks increased with increase of screw speed. In deep fat frying process, water inside the food evaporates and oil migrates into the food. In our study, all half products dried to a constant initial moisture content before frying process. However, extrusion factors critically affect the water replacement by oil. WAI is a functional characteristic for extrudates.

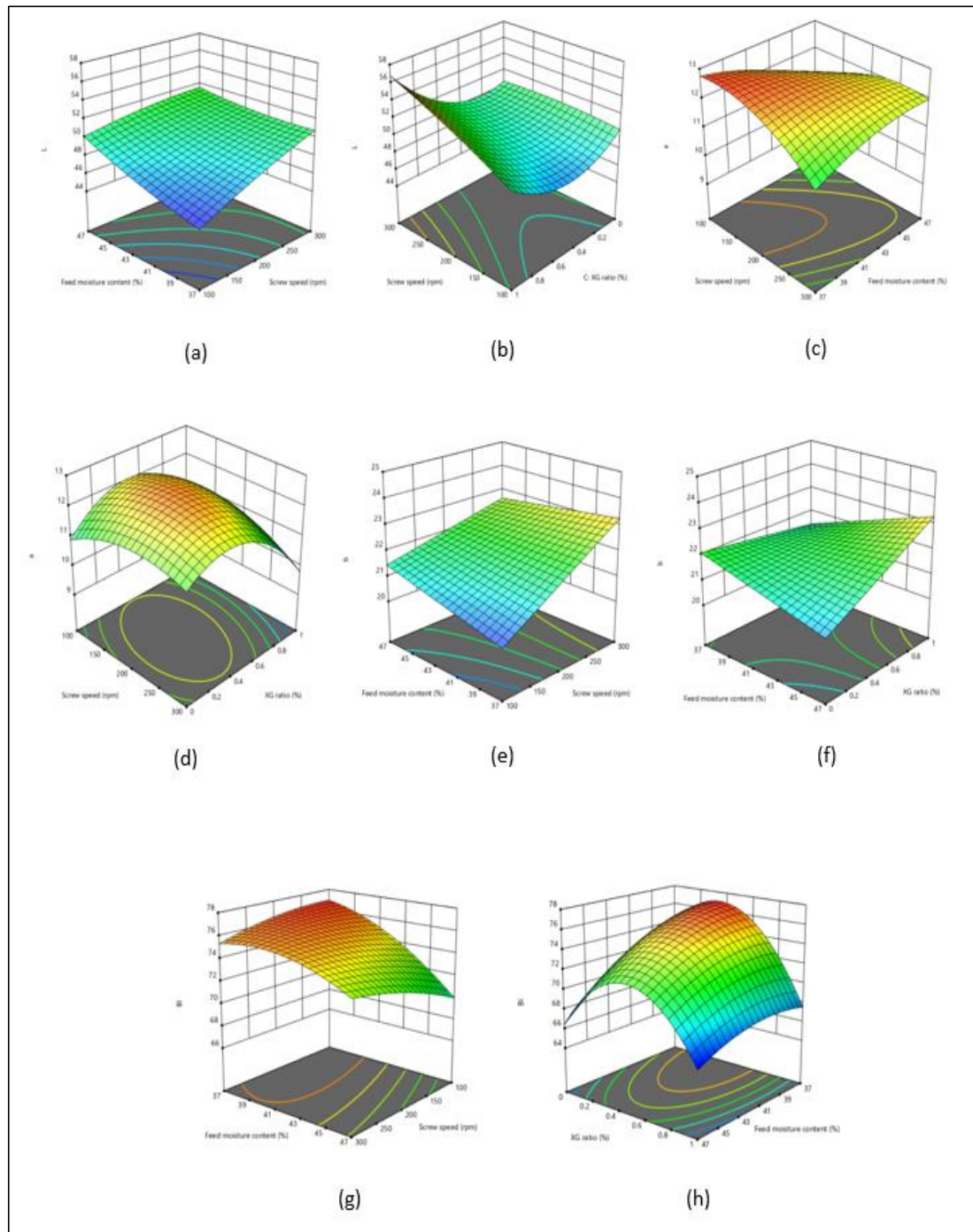


Figure 4.5: Three dimensional surface diagrams illustrating the effects of screw speed, feed moisture and XG ratio on L (a-b), a (c-d), b values (e-f) and browning index (g-h) of third generation corn snacks.

In the literature, it is known that low water absorption can be result the formation of compact starch structures that limit water penetration [Colonna et al., 1989]. In our previous study, we have showed that water absorption index of corn half products decreased with increase of screw speed [Gumusay et al, 2019]. Similar results were obtained in the literature [Osman et al., 2000]. They were reported that low water

absorption resulted in high oil absorption [Osman et al., 2000]. It may be suggested that the generation of compact starch structures that negative affect the water absorption did not offer resistance to oil absorption. These results show us that oil absorption may not be a simple quantitative water-oil replacement phenomena on the surface. Feed moisture level had similar effect on the surface of fried corn snacks. At higher feed moisture level, roughness and holes at surface were increased (Figure 4.6c). With increasing of feed moisture content, expansion, oil absorption increased while hardness decreased. Oginni et al. [2015] reported that holes on the surface can be caused the higher oils content for fried matrices [Oginni et al., 2015]. According to Figure. 4.6e-f, addition of XG (at 1% level) caused the smooth surface. Dueik et al. [2014] shown that high fiber content caused to reduction in oil content during frying [Dueik et al., 2014]. Roughness of surface was lower at XG added corn snack sample due to decrease of oil absorption. In addition to this, increase of hardness with XG addition was related to low oil absorption into the snacks from the surface.

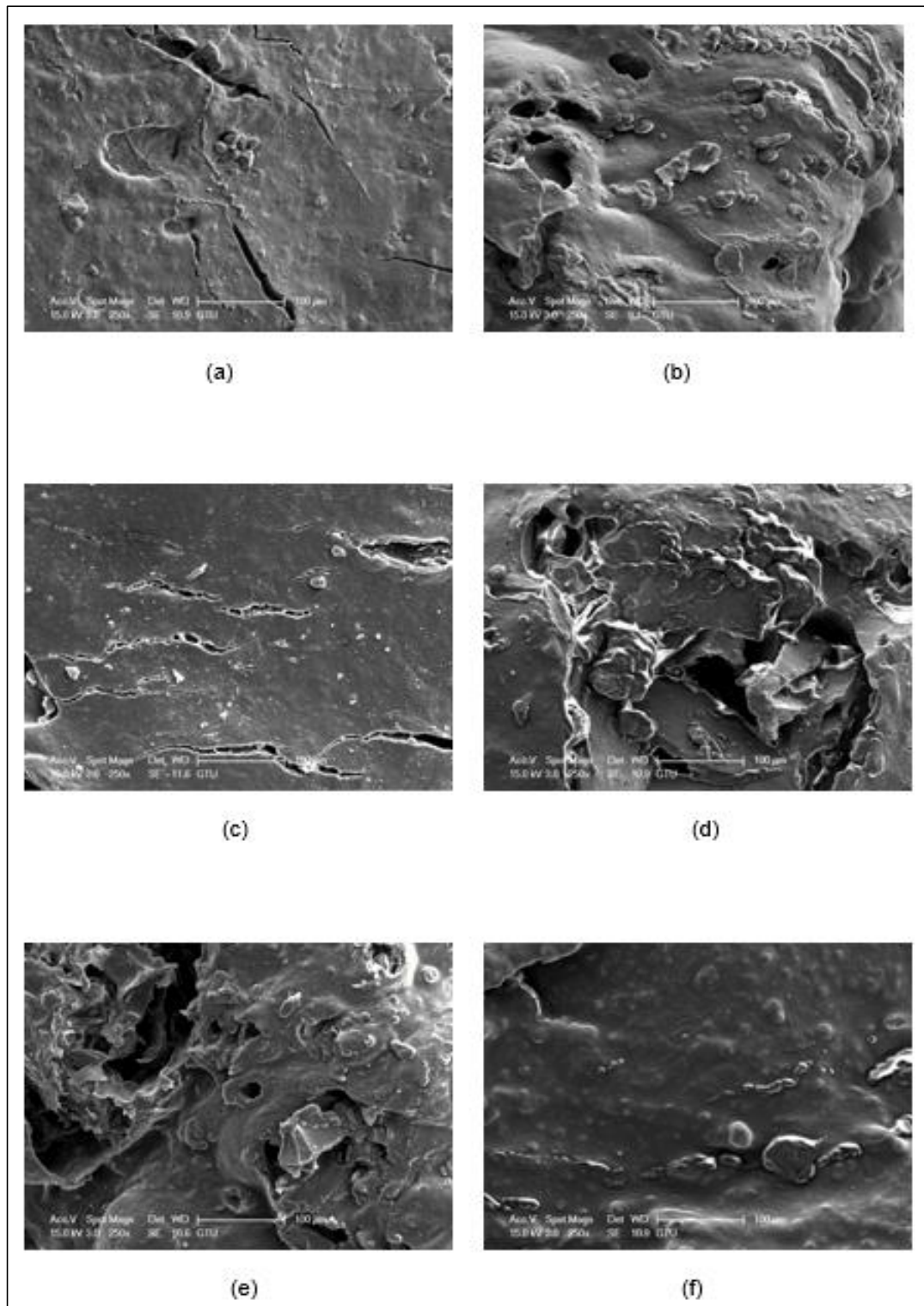


Figure 4.6: Scanning electron micrographs of the surface of fried corn snacks. All the images were taken at 250x magnification. (a) screw speed 100 rpm, feed moisture 47%, XG ratio 0.5 % (Run no 12); (b) screw speed 300 rpm, feed moisture 47%, XG ratio 0.5 % (Run no 2); (c) screw speed 200 rpm, feed moisture 37%, XG ratio 0 % (Run no 10); (d) screw speed 200 rpm, feed moisture 47%, XG ratio 0% (Run no 3); (e) screw speed 100 rpm, feed moisture 42%, XG ratio 0% (Run no 15); (f) screw speed 100 rpm, feed moisture 42%, XG ratio 1 % (Run no 5).

4.2.6. Optimization and Model Validation

Desirability function approach was used to optimize the process conditions for extrusion by numerical optimization. The optimum solution was attained at the screw speed of 246 rpm, feed moisture content of 47% and XG ratio of 1%. Desirability value was found 0.76. The corresponding results for optimized extrusion process were determined as EI 1.67, oil content 9.07%, hardness of 10.94 N, L value of 57.69, a value of 10.74, b value of 23.81 and BI 66.05. Extrusion process was performed at optimum conditions. The experiments were performed triplicate and results were given in Table 4.3. No significant difference was found between experimental results and predicted values ($p > 0.05$). This shows that the adequacy of the developed models was confirmed for identifying the relationship between the process variables and responses.

Table 4.3: Predicted values and experimental results for fried extrudates at optimum extrusion conditions.

Response	Predicted values	Experimental results
Expansion Index	1.67	1.73 ± 0.02
Oil content (%)	9.07	9.37 ± 0.35
Hardness (N)	10.94	9.39 ± 1.23
L	57.69	56.78 ± 0.99
a	10.74	10.61 ± 1.00
b	23.81	23.59 ± 0.62
Browning Index	66.05	66.83 ± 1.77

5. EFFECTS of EXTRUSION CONDITIONS on EXPANDED CORN HALF PRODUCTS by MICROWAVE HEATING

Heat produced by microwave energy internally transfers to food by the molecular friction of the electrical dipoles and charged molecules under an oscillating field of specific frequency [Mudgett, 1982]. Superheated steam generated by heat is facilitated to expansion of extrudates and formation of porous structure [Gimeno et al., 2004]. Microwave heating for expansion process of half products may have more advantages compared to deep fat frying. Because, microwave expanded snacks have lower fat content. In addition to this, microwave expanded snacks can be easily and fast prepared at home or office.

Starch in the cereals is the main component to contribute the expansion phenomena. Starch gelatinization provided by extrusion has an important positive effect on expansion during microwave heating. Because microwave energy is used for expansion rather than starch gelatinization, thus more expanded snack can be produce [Lee et al., 2000].

Extrusion process conditions (feed moisture content, screw speed, barrel temperature) and additives used in extrusion process have a critical importance for quality characteristics of microwave expanded snacks. Ernoult et al. [2002] reported that liquid fat addition to feed material had a negative effect on microwave expansion [Ernoult et al., 2002]. According to Bastos-Cardoso et al. [2007], low barrel temperature and high feed moisture content increased the luminosity of the microwave expanded pellets [Bastos-Cardoso et al., 2007]. Same researchers reported that addition of corn starch to feed prepared with whole potato flour increased expansion and viscosity. Aguilar-Palazuelos et al. [2006] reported that expansion increased with increase of barrel temperature [Aguilar-Palazuelos et al., 2006]. In the same study viscosity increased when feed moisture content increases to >23%. Delgado-Nieblas et al. [2012] used with winter squash flour to produce the half products and they reported that increasing of winter squash flour increased total color difference whereas expansion decreased [Delgado-Nieblas et al., 2012].

Incorporation of the gums into the feed material significantly improved the physical properties of the microwave-expanded samples [Gimeno et al., 2004].

Gimeno et al. [2004] denoted that 1% addition level for xanthan gum (XG) and carboxymethyl cellulose is sufficient for improve the shape and texture of microwave expanded corn extrudates [Gimeno et al., 2004]. Addition level for fiber or gums is an important factor for snack structure. Because, above a critical additional level, fiber or gum molecules can be bind the moisture in the melt or they can be break damage the structure of melt during extrusion [Moraru and Kokini, 2003]. Effects of gum additional level and extrusion process conditions can be evaluate together with response surface methodology approach. Furthermore, RSM can be help us to achieve a better product considering multiple quality characteristic with desirability function.

5.1. Materials and Methods for Microwave Expanded Half Products

Extrudates were manually cut into 2 cm lengths and dried to 12–13% moisture content at 50 °C for 12–14 h. Corn half products were expanded in a microwave oven (Samsung, ME711K) with a frequency 2450 MHz and power density of 800 W. All samples were not evenly expanded before 30 sec heating time. Some half products were started to burn after 30 sec in microwave. Thus, microwave heating time was determined as 30 sec according to preliminary tests.

5.1.1. Experimental Design

The same experimental design as in section 3.1.1 was used.

5.1.2. Rheological Properties

Rheological properties were measured with Brookfield Rheometer (Model RS4 SST, USA). Microwave expanded corn snacks were milled and sieved (200 μm). The milled samples were dried at 50 °C overnight. One gram sample was mixed with 30 mL distilled water and then sample solution heated at 85 °C for 25 minutes. Then suspension was cooled to room temperature and measurements were practiced with parallel plate probe at constant room temperature (25 °C). The shear stress and shear rate (0-300 s^{-1}) data were recorded during 400 second. Then data analyzed with using the software program.

5.1.3. Apparent Amylose Content

The apparent amylose content of samples was determined by iodine-based colorimetric method according to Hoover and Ratnayake [2005] experimental procedure [Hoover and Ratnayake, 2005]. Amylose (from potato) and amylopectin (from maize) mixtures at different ratios were prepared for calibration curve. The results were given as the average of three measurements.

5.1.4. Expansion Index

Expansion index of microwave expanded corn snacks was determined according to literature procedure of Bastos-Cardoso et al. [2007].

5.1.5. Color Properties

Color properties of microwave expanded corn snacks were determined by measuring with Konica Minolta CR-400 Chroma Meter. Color values were recorded as L , a and b values according to Hunter Lab system. The total color difference (ΔE) (Equation 5.1) and BI (Equation 4.1) were calculated from the L , a and b values of corn snacks;

$$\Delta E = \sqrt{(L - L_0)^2 + (b - b_0)^2 + (a - a_0)^2} \quad (5.1)$$

5.1.6. Statistical Analysis and Optimization

ANOVA technique was conducted to investigate the significance of each independent variables on responses. Multiple regression analysis was applied to fit a second order polynomial model with the Design-Expert software. Model adequacies were analyzed in terms of lack of fit test, R-squared and adjusted R-squared values. Response surface plots were generated to characterize graphically the effect of the independent process variables on each response. The effect of two variables on the response was examined with response surface plots while the third variable was kept at the middle level.

Desirability function tool of RSM was used to generate optimal extrusion conditions (screw speed, feed moisture and XG additional level). The optimum extrusion conditions were determined to obtain the maximum values for expansion index and luminosity and minimum value for the total color difference. Other responses and process variables were kept in range during optimization. Experiments at optimum process conditions were performed in triplicate. Experimental data and predictive values obtained from the models were compared.

5.2. Results and Discussion for Microwave Expanded Half Products

Experimental results shown in Table 5.1 and they were fitted to Equation (3.1). F-values, lack of fit (*p*-values), R^2 and adjusted R^2 values of the developed models were indicated in Table 5.2. *p*-values of all models were less than 0.001 (significant) and lack of fit (*p*) values were of all models greater than 0.05 (non-significant).

5.2.1. Effects of Extrusion Conditions on Rheological Properties

Rheological measurements show that all suspensions had power law properties. Ostwald-de Waele model with strong correlation ($R^2 > 0.985$) between shear rate and shear stress was applied. Apparent viscosity (η , at 25s^{-1} of shear rate) was calculated with this model Equation (5.2) and results given in Table 5.1.

$$\eta = K\gamma^{n-1} \quad (5.2)$$

where K is the consistency index (Pa s), γ is the shear rate (s^{-1}), n is flow behavior index and η is apparent viscosity (Pa s). All suspensions exhibited shear-thinning behavior ($n < 1$). Apparent viscosity ranged between 3.80 ± 0.94 and 42.11 ± 1.24 . According to coefficients in the Table 5.2, screw speed and feed moisture had positive linear and quadratic effects on the apparent viscosity of expanded corn snacks. However, XG ratio had negative quadratic effect on the apparent viscosity. All interactions except feed moisture content and XG ratio interaction had a positive effect on viscosity.

Wetted starch is gelatinized with heat, it absorbs water and then increases viscosity of suspension by force of friction heat obtained with screw rotation. If shearing of starch exceeds some level at low moisture content, starch is degraded, its water absorption and viscosity decreases. Increasing the screw speed reduce residence time and solubility at high moisture content [Seker, 2005], [Seker, 2004]. Thus, apparent viscosity of extrudates heated at microwave increased with elevation of screw speed due to reduction of solubility (Figure 5.1a).

Starch needs water for gelatinization. When moisture content is low, starch is degraded and this causes low viscosity. High feed moisture acts as a lubricant and cause decreases of the shear degradation of starch and increase viscosity so apparent viscosity increased with elevation of feed moisture content as result of reduction of solubility at higher moisture content (Figure 5.1a). Increasing moisture content during extrusion reduce gelatinization and viscosity of starch but it can be compensated with microwave cooking.

Lower thermal energy can be resulted in lower starch degradation. High feed moisture acts as a lubricant and this caused to decrease in the shear degradation of starch. Aquilar-Palazuelos et al. [2006] reported that low starch degradation during extrusion caused to greater viscosity for expanded pellets [Aquilar-Palazuelos et al., 2006]. Correlation between viscosity and feed moisture content may be attributed to increase of glass transition temperature of starch with decrease of feed moisture [McPherson et al., 2000]. Thus, low feed moisture content resulted in higher starch degradation during extrusion and lower viscosity for expanded samples after extrusion. On the other hand, comparison of our results with our previous study results [Gumusay et al., 2019], which about effect of moisture content on water absorption index, shows that microwave heating of extrudates may have increased viscosity of samples just extruded at every moisture content.

Figure 5.1b shown that apparent viscosity decreased with increase of XG ratio. Similar results were observed in the literature when hydrocolloids added to feed during extrusion [Maitre et al., 2017], [Singh and Singh, 2004]. Enhancement of water absorption with increase of XG in extrusion of feed may have caused higher starch degradation and reduction of viscosity. Therefore, viscosity of microwave expanded of corn extrudate decreased with increase of XG level.

Table 5.1: Coded and uncoded values for the experimental design and experimental results for the microwave expanded half product responses.

Runs	Coded variables			Uncoded variables			Experimental results					
	X ₁	X ₂	X ₃	Screw speed (rpm)	Feed moisture (%)	XG ratio (%)	Apparent Viscosity (Pa s)	Amylose content (%)	EI	Luminosity	BI	ΔE
1	0	0	0	200	42	0.5	15.26 $\pm$ 1.65	30.63 $\pm$ 1.96	1.76 $\pm$ 0.03	63.12 $\pm$ 0.93	55.61 $\pm$ 0.99	11.37 $\pm$ 0.47
2	+1	+1	0	300	47	0.5	42.11 $\pm$ 1.24	31.55 $\pm$ 1.77	2.10 $\pm$ 0.23	66.30 $\pm$ 0.92	52.23 $\pm$ 1.73	4.95 $\pm$ 0.45
3	0	+1	-1	200	47	0	27.10 $\pm$ 1.86	32.55 $\pm$ 2.89	1.62 $\pm$ 0.02	61.79 $\pm$ 0.99	48.21 $\pm$ 1.04	7.21 $\pm$ 0.71
4	0	+1	+1	200	47	1	16.93 $\pm$ 3.19	27.30 $\pm$ 2.24	1.55 $\pm$ 0.04	58.94 $\pm$ 0.94	52.67 $\pm$ 1.33	9.24 $\pm$ 0.42
5	-1	0	+1	100	42	1	9.25 $\pm$ 2.86	22.47 $\pm$ 1.76	1.71 $\pm$ 0.16	58.26 $\pm$ 1.01	57.27 $\pm$ 0.73	16.57 $\pm$ 0.97
6	0	0	0	200	42	0.5	12.36 $\pm$ 2.16	32.47 $\pm$ 2.31	1.80 $\pm$ 0.05	63.32 $\pm$ 0.59	55.55 $\pm$ 0.51	10.97 $\pm$ 0.62
7	0	0	0	200	42	0.5	13.08 $\pm$ 1.45	30.30 $\pm$ 1.41	1.75 $\pm$ 0.04	63.31 $\pm$ 1.25	54.57 $\pm$ 0.97	10.84 $\pm$ 0.52
8	0	0	0	200	42	0.5	12.17 $\pm$ 1.02	29.80 $\pm$ 1.71	1.80 $\pm$ 0.03	64.39 $\pm$ 1.39	55.55 $\pm$ 0.92	10.97 $\pm$ 1.30
9	+1	0	+1	300	42	1	23.10 $\pm$ 2.14	29.30 $\pm$ 1.73	1.53 $\pm$ 0.03	68.49 $\pm$ 1.16	44.56 $\pm$ 0.90	9.84 $\pm$ 0.86
10	0	-1	-1	200	37	0	3.80 $\pm$ 0.94	36.55 $\pm$ 2.35	1.69 $\pm$ 0.01	67.92 $\pm$ 1.34	44.89 $\pm$ 1.36	17.9 $\pm$ 0.57
11	+1	0	-1	300	42	0	17.10 $\pm$ 1.69	37.97 $\pm$ 2.06	1.75 $\pm$ 0.02	70.51 $\pm$ 0.81	46.36 $\pm$ 1.03	9.11 $\pm$ 0.83
12	-1	+1	0	100	47	0.5	16.30 $\pm$ 1.72	24.05 $\pm$ 2.47	1.72 $\pm$ 0.11	56.05 $\pm$ 1.15	55.85 $\pm$ 1.74	12.45 $\pm$ 1.01
13	-1	-1	0	100	37	0.5	17.13 $\pm$ 2.11	29.05 $\pm$ 1.35	2.30 $\pm$ 0.05	63.16 $\pm$ 1.64	58.56 $\pm$ 1.34	20.90 $\pm$ 1.19
14	0	-1	+1	200	37	1	12.00 $\pm$ 1.79	28.47 $\pm$ 1.44	1.55 $\pm$ 0.05	68.11 $\pm$ 1.29	45.33 $\pm$ 1.13	17.57 $\pm$ 0.82
15	-1	0	-1	100	42	0	18.00 $\pm$ 2.16	32.13 $\pm$ 2.47	1.72 $\pm$ 0.05	61.03 $\pm$ 0.80	52.33 $\pm$ 1.64	15.17 $\pm$ 0.99
16	0	0	0	200	42	0.5	14.65 $\pm$ 1.94	30.05 $\pm$ 1.06	1.81 $\pm$ 0.17	64.00 $\pm$ 1.20	54.61 $\pm$ 1.36	11.55 $\pm$ 1.59
17	+1	-1	0	300	37	0.5	14.56 $\pm$ 1.43	37.05 $\pm$ 1.77	1.74 $\pm$ 0.11	71.83 $\pm$ 1.11	42.41 $\pm$ 1.05	14.03 $\pm$ 1.13

Table 5.2: Coefficients from the predictive model equation for microwave expanded half product responses.

Variables	Apparent viscosity	Apparent amylose content	EI	Luminosity	BI	ΔE
Intercept	13.50	30.54	1.78	64.15	55.18	11.14
X ₁	4.52***	3.52***	-0.04***	4.83***	-4.81***	-3.40***
X ₂	6.87***	-2.33***	-0.04***	-3.49***	2.22***	-4.57***
X ₃	ns	-4.33***	-0.06***	-0.93***	1.01***	0.48***
X ₁ X ₂	7.10***	ns	0.24***	ns	3.13***	ns
X ₁ X ₃	3.69***	ns	-0.0525***	ns	-1.69***	ns
X ₂ X ₃	-4.59***	ns	ns	ns	1.01**	0.59**
X ₁ ²	5.46	ns	0.13***	ns	ns	0.82***
X ₂ ²	3.56	ns	0.05***	ns	-2.64***	1.13***
X ₃ ²	-2.10**	ns	-0.23***	ns	-4.77***	0.72***
Model (<i>F</i> -value)	39.62 ^a	183.19 ^a	76.38 ^a	157.70 ^a	99.98 ^a	217.19 ^a
<i>r</i> ²	0.9807	0.9769	0.9899	0.9733	0.9923	0.9964
Adjusted <i>r</i> ²	0.9560	0.9716	0.9770	0.9671	0.9824	0.9918
<i>Lack of Fit</i> (<i>p</i> -value) ^b	0.2121	0.9385	0.3751	0.1869	0.2048	0.2288

X₁, Screw speed (rpm); X₂, Feed moisture (%); X₃, XG ratio (%).

^a Significant at a $p < 0.0001$ level.

^b want the selected model to have non-significant lack of fit ($p > 0.05$)

*Significant at $0.05 \leq p < 0.1$.

**Significant at $0.01 \leq p < 0.05$.

*** Significant at $p < 0.01$; ns, non-significant (p -values are greater than 0.1).

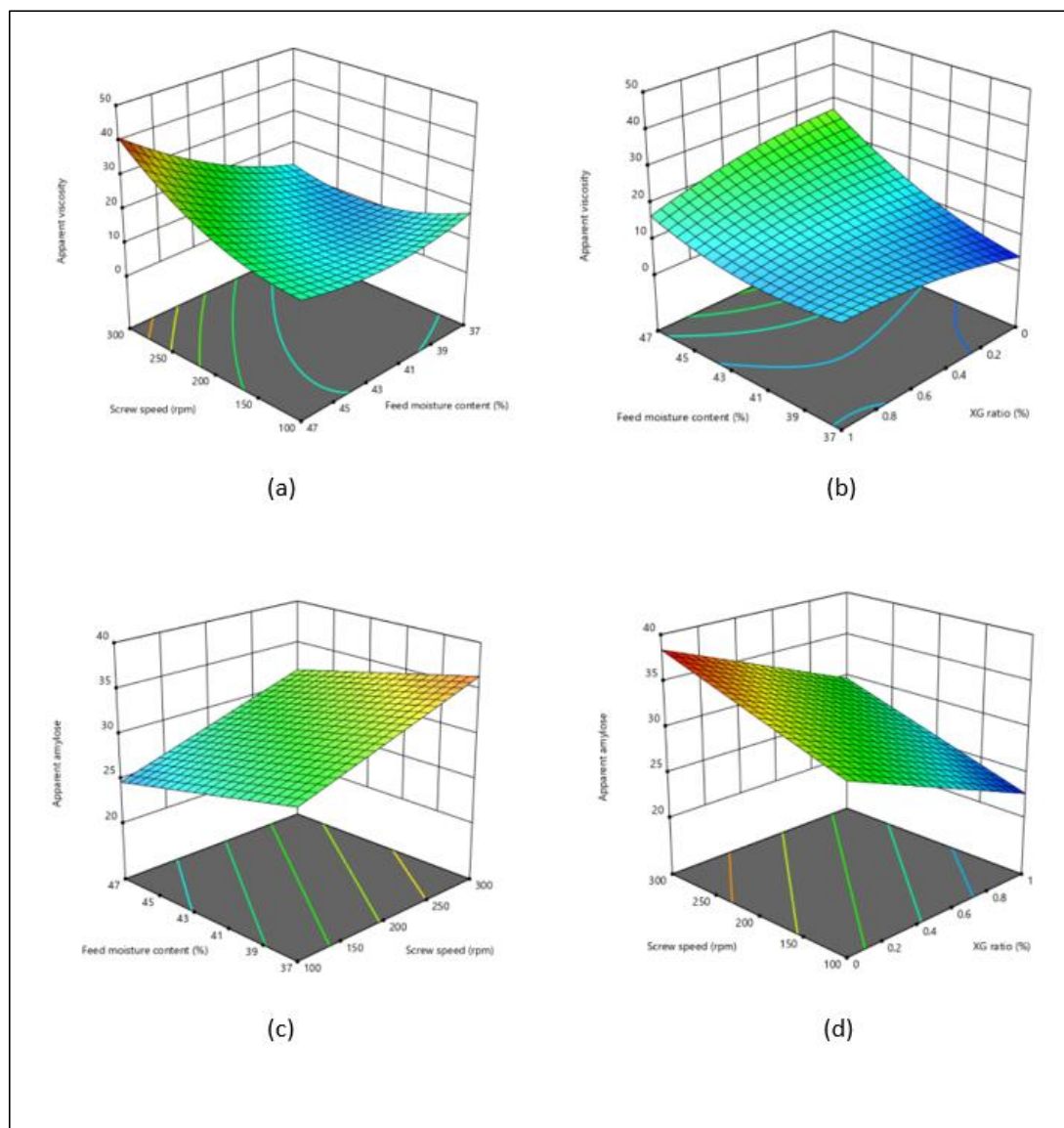


Figure 5.1: Three dimensional surface diagrams illustrating the effects of screw speed, feed moisture and XG ratio on apparent viscosity (a-b) and apparent amylose content (c-d).

5.2.2. Effects of Extrusion Conditions on Apparent Amylose Content

Apparent amylose content of microwave expanded snacks ranged from 22.47% to 37.97%. According to model coefficients from Table 5.2, screw speed had positive linear effect on apparent amylose content of corn snacks. Higher screw speed resulted in decrease of residence time and starch degradation at high moisture content. Increase of apparent amylose content with increase of screw speed was an expected result. Since starch degradation is low, amylose has a longer structure and it has more iodine binding capacity. However, feed moisture content had negative effect on amylose

content of corn snacks (Figure 5.2c) and it shows that degradation of starch is not just one factor affecting apparent amylose content. Increasing moisture content may have increased interference concentration and resulted in measurement of decreased apparent amylose content. Htoon et al. [2009] reported that gelatinized form of amylose under extrusion process with high feed moisture may undergo degradation resulting in shorter amylose molecules with a reduced iodine binding capacity.

Apparent amylose content of expanded extrudates was decreased with increases of XG concentration (Figure 5.1d). XG may have complex with amylose or it may have act as interfering material and reduced apparent amylose content. Wang et al. [2007] reported that the thickening effect of XG, which is a water soluble hydrocolloid, may cause increase of effective amylose concentration in continuous phase while XG-starch mixtures gelatinize.

5.2.3. Effects of Extrusion Conditions on Expansion Index

EI data of microwave expanded snacks varied from 1.55 ± 0.04 to 2.30 ± 0.05 (Table 5.1). According to Table 5.2, a negative linear effect and positive quadratic effect of screw speed on EI was observed. Interaction of screw speed with feed moisture had positive effect on EI whereas interaction of screw speed with XG ratio had negative effect on EI. Lee et al. [2000] reported that better microwave expansion of corn starch pellets was obtained at 52% degree of gelatinization and they observed that pellets rarely expanded at lower than 50% and or at higher than 65% degree of starch gelatinization [Lee et al., 2000]. In our previous work, degree of gelatinization of corn half products decreased to 50% from 63% with increase in screw speed [Gumusay et al., 2019]. In this study, EI of microwave expanded corn half product was increased with increase in screw speed (Figure 5.2a). At higher screw speed, residence time may be decreased and so input thermal energy may be decreased. Thus, higher screw speed caused to decrease of gelatinization degree to 50%. Increase of expansion at higher screw speed was an expected result in view of gelatinization degree.

Figure 5.2a shows negative effect of feed moisture content on expansion of corn pellets. Towar-Jimenez et al. [2015] reported that EI values of microwave expanded third generation snack increased with decrease in feed moisture content [Towar-Jimenez et al., 2015]. In our study, similar results were obtained. In extrusion process,

high feed moisture acts as a lubricant and this caused to decrease the shear force, temperature and gelatinization. Thus starch gelatinization may be decreased. If sufficient starch gelatinization is not achieved, full expansion of the half product can not be achieved in the microwave heating. Because pellets had insufficient gelatinization (<50%), air cell formation can not occur and sudden release of internal pressure cannot actualized during microwave heating for expansion [Lee et al. 2000]. Kebede et al. [2010] attributed that alteration in EI was related to the starch gelatinization during extrusion [Kebede et al., 2010]. In our previous study, gelatinization degree of half product decreased with increase of feed moisture content. This result corroborated that reduction of expansion of half product during microwave with increase of feed moisture content. Moraru and Kokini [2003] reported that expansion is a complicated phenomena and it generally occurs at low feed moisture content [Moraru and Kokini, 2003].

XG addition had negative linear and quadratic effect on EI value of microwave expanded snacks (Table 5.2). According to Figure 5.2b, expansion increased with increase of XG addition level to 0.5%, however expansion decreased at higher than 0.5% value of XG ratio. As seen in literature, fiber or gums added to feed material during extrusion affect the expansion dependent on addition concentration of them [Delgado-Nieblas et al., 2012], [Gimeno et al., 2004], [Moraru and Kokini, 2003], [Onwulata et al., 2001], [Lue and Huff, 1991]. In our previous study, starch gelatinization degree increased to 50% when XG ratio increased to 0.5%. However, we observed that gelatinization degree of half product decreased, when XG ratio was higher than 0.5% [Gumusay et al., 2019]. Maitre et al. [2017] reported that addition of gum may be increase the water absorption capacity, provide better interaction with starch components and increase the starch degradation [Maitre et al., 2017]. However the higher level of gum in the starch intergranular space may be reduce interparticle contacts and restrain gel formation [Zhuang et al., 2010]. Another reason may be water competition between gum and starch. Towar-Jimenez et al. [2016] reported that gum with fiber structure affected starch gelatinization as starch molecules become less soluble and tend to aggregate [Towar-Jimenez et al., 2016]. Thus, insufficient gelatinization attributed to reduction of expansion. Delgado-Nieblas et al. [2012] reported that expansion of third generation snack produced with winter squash addition decreased due to high content of dietary fiber structure [Delgado-Nieblas et al., 2012]. Lue et al. [1991] stated that high fiber content can disrupt cell wall

structure, decrease the air bubbles inside the wall and therefore prevents the expansion [Lue et al., 1991].

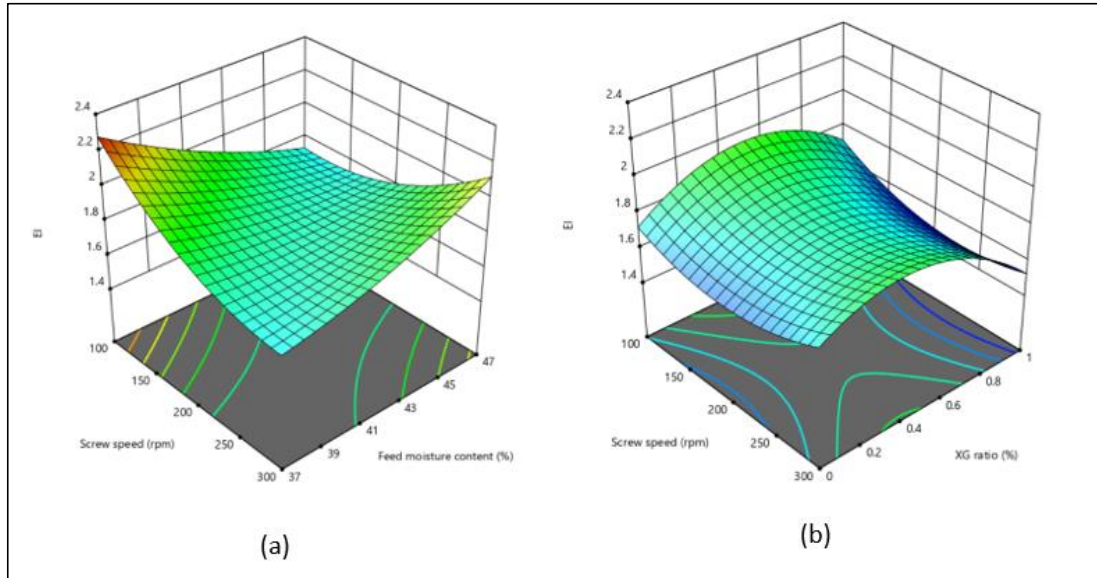


Figure 5.2: Three dimensional surface diagrams illustrating the effects of screw speed, feed moisture and XG ratio on expansion index (a-b).

5.2.4. Effects of Extrusion Conditions on Color Properties

Luminosity, BI and ΔE inform that severity of heat treatment during extrusion and microwave expansion. Therefore color properties are important factors affecting the consumer choice. Luminosity values of expanded snacks ranged between from 56.05 ± 1.15 and 71.83 ± 1.1 (Table 5.1). Screw speed had a positive linear effect on luminosity whereas XG ratio and feed moisture content had a negative linear effect. According to coefficients in Table 5.2, positive linear effect and negative quadratic effect of feed moisture on the BI was observed. However, screw speed had a negative linear effect on BI and ΔE . Figure 5.3a, 3c and 3e shown that luminosity of microwave expanded snacks increased but BI and ΔE decreased with increase of screw speed. We observed same effect of screw speed on the luminosity of half product (non-expanded) in our previous study [Gumusay et al., 2019]. At higher screw speed, residence time was shorter, cooking of extrudates decreased, thus luminosity increased and BI decreased. Volumetric heating occurs during microwave expansion. Volumetric heating induce high center temperature in the pellet [van der Sman and Bows, 2017] and most of this heating energy may be used to expansion process and temperature

gradient may be insufficient to burn of pellets after expansion. In this study expansion increased with increase of screw speed. Thus luminosity increased, but BI and ΔE decreased at more expanded snacks due to absence burning of process.

Increasing of feed moisture content resulted in decrease of luminosity and increase of BI values. Bastos-Cardoso et al. [2007] reported that luminosity decreased if feed moisture content is higher than 25.5% [Bastos-Cardoso et al., 2007]. This result about luminosity was in agreement with our study. Ernoult et al. [2002] shown that burning starts from the center during microwave heating as expansion did [Ernoult et al., 2002]. Increasing feed moisture content resulted in low expansion of half product in this study. Burning process may be more intense in poor-expanded snacks during microwave heating and so BI increased and luminosity decreased with increase of feed moisture content.

According to Figure 5.3d, XG ratio has quadratic effect on BI. BI increased with increase of XG ratio to 0.5% and BI decreased if XG ratio is above 0.5%. Similar but reverse effect of XG addition level on the expansion was observed in this study (Figure 5.3b). Expansion change with XG addition affected the BI due to burning process as described above. ΔE increased with increase of XG ratio. Delgado-Nieblas [2012] reported that ΔE increased with addition of squash flour containing high amount of fiber [Delgado-Nieblas, 2012]. XG also has a fiber structure and structural modifications of fiber components during microwave heating might have been caused to higher burning of half products [Hashimoto and Grossman, 2003].

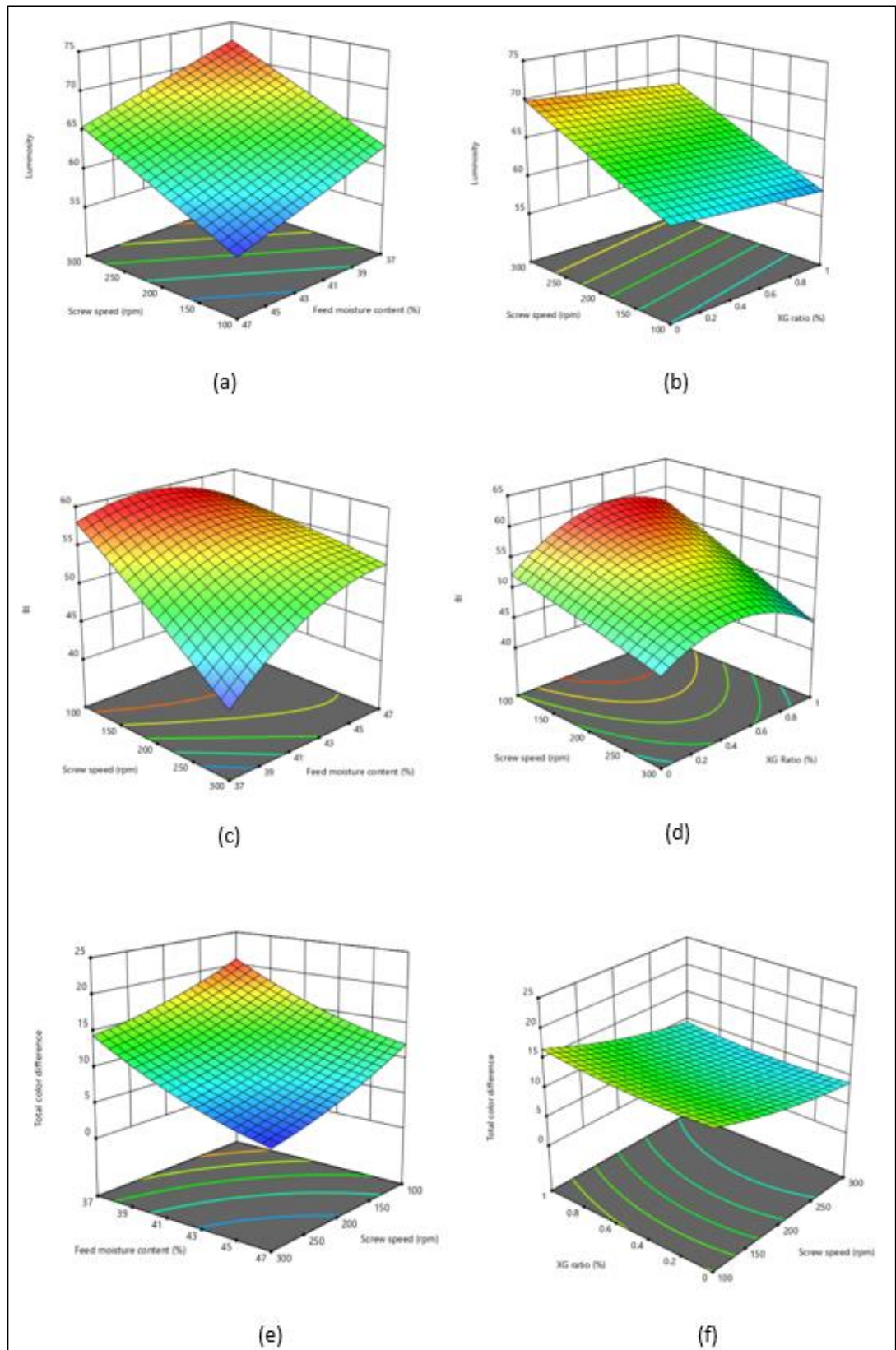


Figure 5.3: Three dimensional surface diagrams illustrating the effects of screw speed, feed moisture and XG ratio on color properties; luminosity (a-b), browning index (c-d) and total color difference (e-f).

5.2.5. Optimization and Model Validation

Desirability function approach was used to optimize the process conditions for extrusion by numerical optimization. The optimum solution was attained at the screw speed of 300 rpm, feed moisture content of 47% and XG ratio of 0.34%. Desirability value was found 0.782. The corresponding results for optimized extrusion process were determined as EI 2.13, luminosity 65.78, BI 52.24, ΔE 4.75, apparent viscosity 41.24 Pa s and apparent amylose content 33.09%. Extrusion process was performed at optimum conditions. The experiments were performed triplicate and results were given as average value with standard deviation (Table 5.3). There was no significant difference between experimental results and predicted values ($p > 0.05$). This shows that the adequacy of the developed models was confirmed for identifying the relationship between the process variables and responses.

Table 5.3: Predicted values and experimental results microwave expanded half products at optimum extrusion conditions.

Response	Predicted values	Experimental results
Apparent viscosity (Pa s)	41.24	41.80 ± 1.74
Apparent amylose content (%)	33.09	32.37 ± 1.73
Expansion Index	2.13	2.09 ± 0.30
Luminosity	65.78	65.51 ± 0.78
BI	52.24	52.52 ± 0.68
ΔE	4.75	4.88 ± 0.37

6. CONCLUSIONS

This thesis revealed that screw speed, feed moisture and XG addition level influence on functional and physical properties of corn based half products and quality characteristics of expanded extrudates by frying and microwave heating. RSM has been a useful tool for analysis of the simultaneous interactive effects of independent variables on half and expanded product characteristics, for a better understanding of the corn snack production process.

The low DG and specific volume values for half products were obtained from most extrusion conditions and this was required to produce flat, thin and bubble-free half products. Lightness and yellowness of half products were increased with increases in screw speed, feed moisture and XG level. Increased screw speed decreased WAI and a_w values and increased WSI values significantly. The developed models with the BBD found to be statistically valid. The optimized combinations of extrusion conditions were found 300 rpm screw speed, 47% feed moisture and 0.44% XG level, with a desirability value of 0.91.

Increasing screw speed and moisture content during extrusion resulted in expansion, rough surface and holes after frying of extrudates. Higher expansion provided with high feed moisture decreased hardness of fried samples but degradation of starch with increased screw speed enhanced hardness. Increases of hardness with incorporation of XG can be due to fiber and hydrophilic structure. Fried products, produced from half products prepared at high feed moisture and screw speed, had higher oil absorption. Increases of oil absorption should be result of enhanced expansion and holes on surface. Increasing screw speed, moisture content and xanthan gum level affected lightness and redness of fried samples. Optimum conditions were determined by desirability function were found 246 rpm screw speed, 47% feed moisture content and 1% XG ratio, with a desirability value of 0.76.

Screw speed had a positive effect on expansion however feed moisture content had negative effect for microwave expanded extrudates. Critic XG addition level was found as 0.5% for EI. When XG addition level was above the 0.5%, expansion of microwave expanded extrudates decreased. Luminosity of microwave expanded extrudates increased but BI and ΔE decreased with increase of screw speed. Feed moisture content had a negative effect on luminosity and a positive effect on BI values

of microwave expanded samples. ΔE increased and luminosity decreased with increase of XG ratio. Apparent viscosity and amylose content of expanded microwave corn snacks increased with increase of screw speed and feed moisture level. XG addition resulted in decrease of apparent viscosity and amylose content of microwave expanded samples. Optimum conditions determined by desirability function were found 300 rpm screw speed, 47% feed moisture content and 0.34% XG ratio, with a desirability value of 0.78.

The results obtained in this thesis may be employed as baseline information for further scale-up of third generation snack production with several additives and under different production conditions.

REFERENCES

- Adamu A., Jin Z. Y., (2002), "Effect of chemical agents on physical and rheological properties of starch-guar gum extrudates", *International Journal of Food Properties*, 5 (2), 261-275.
- Aguilar-Palazuelos E., de J. Zazueta-Morales J., Martínez-Bustos F., (2006), "Preparation of high-quality protein-based extruded pellets expanded by microwave oven", *Cereal Chemistry*, 83 (4), 363-369.
- Ainsworth P., Ibanoglu Ş., Plunkett A., Ibanoglu E., Stojceska V., (2007), "Effect of brewers spent grain addition and screw speed on the selected physical and nutritional properties of an extruded snack", *Journal of Food Engineering*, 81 (4), 702-709.
- Alam M. S., Pathania S., Sharma A., (2016), "Optimization of the extrusion process for development of high fibre soybean-rice ready-to-eat snacks using carrot pomace and cauliflower trimmings", *LWT-Food Science and Technology*, 74, 135-144.
- Altan A., McCarthy K. L., Maskan M., (2008), "Evaluation of snack foods from barley–tomato pomace blends by extrusion processing", *Journal of Food Engineering*, 84 (2), 231-242.
- Annapure U. S., Singhal R. S., Kulkarni P. R., (1999), "Screening of hydrocolloids for reduction in oil uptake of a model deep fat fried product", *Lipid/Fett*, 101 (6), 217-221.
- AOAC., (2000), "Official methods of analysis of the Association of Official Analytical Chemists International", 17th Ed. Gaithersburg, MD: AOAC.
- Arambula V. G., Mauricio S. R. A., Figueroa C. J. D., Gonzalez-Hernandez J., Ordorica, F. C. A., (1999), "Corn masa and tortillas from extruded instant corn flour containing hydrocolloids and lime", *Journal of Food Science*, 64 (1), 120-124.
- Ari Akin P., Miller R. A., (2017), "Starch–hydrocolloid interaction in chemically leavened gluten-free sorghum bread", *Cereal Chemistry*, 94 (5), 897-902.
- Bajaj I., Singhal R., (2007), "Gellan gum for reducing oil uptake in sev, a legume based product during deep-fat frying", *Food Chemistry*, 104 (4), 1472-1477.
- Bastos-Cardoso I., de J. Zazueta-Morales J., Martínez-Bustos F., Kil-Chang Y., (2007), "Development and characterization of extruded pellets of whole potato (*Solanum tuberosum* L.) flour expanded by microwave heating", *Cereal Chemistry*, 84 (2), 137-144.
- Bhatnagar S., Hanna M. A., (1995), "Physical, mechanical, and thermal properties of starch-based plastic foams", *Transactions of the ASAE*, 38 (2), 567-571.

Bhattacharya M., Hanna M. A., (1987), “Kinetics of starch gelatinization during extrusion cooking”, *Journal of Food Science*, 52, 764-766.

Birch G. G., Priestley R. J., (1973), “Degree of gelatinisation of cooked rice”, *Starch-Stärke*, 25 (3), 98-100.

Borries-Medrano E., Jaime-Fonseca M. R., Aguilar-Mendez M. A., (2016), “Starch–guar gum extrudates: Microstructure, physicochemical properties and in-vitro digestion”, *Food Chemistry*, 194, 891-899.

Box G. E., Behnken D. W., (1960), “Some new three level designs for the study of quantitative variables”, *Technometrics*, 2 (4), 455-475.

Brown M., Fallahi P., Muthukumarappan K., Singha P., Sindelar S., (2015), “A comparative study of the effects of non-starch polysaccharide gums on physical properties of single-screw extruded aquafeed”, *Journal of Food Processing & Technology*, 6 (6), 457.

Butler M., (2016), “Xanthan Gum Applications and Research Studies”, Nova Science Publishers Inc.

Cai W., Diosady L. L., (1993), “Model for gelatinization of wheat starch in a twin-screw extruder”, *Journal of Food Science*, 58, 872-875.

Camacho-Hernández I. L., Zazueta-Morales J. J., Gallegos-Infante J. A., Aguilar-Palazuelos E., Rocha-Guzmán N. E., Navarro-Cortez R. O., Jacobo-Valenzuela N., Gómez-Aldapa C. A., (2014), “Effect of extrusion conditions on physicochemical characteristics and anthocyanin content of blue corn third-generation snacks”, *CyTA-Journal of Food*, 12 (4), 320-330.

Carvalho C. W., Takeiti C. Y., Onwulata C. I., Pordesimo L. O., (2010), “Relative effect of particle size on the physical properties of corn meal extrudates: Effect of particle size on the extrusion of corn meal”, *Journal of Food Engineering*, 98 (1), 103-109.

Chang Y. H., Ng P. K., (2011), “Effects of extrusion process variables on quality properties of wheat-ginseng extrudates”, *International Journal of Food Properties*, 14 (4), 914-925.

Chiu H. W., Peng J. C., Tsai S. J., Tsay J. R., Lui W. B., (2013), “Process optimization by response surface methodology and characteristics investigation of corn extrudate fortified with yam (*Dioscorea alata* L.)”, *Food and Bioprocess Technology*, 6 (6), 1494-1504.

Colonna P., Tayeb J., Mercier C., (1989), “Extrusion Cooking of Starch and Starchy Products”, In: C. Mercier, C.P. Linko, J.M. Harper, Editors, “Extrusion Cooking”, American Association of Cereal Chemists Inc.

Dar A. H., Sharma H. K., Kumar N., (2014), “Effect of Frying Time and Temperature on the Functional Properties of Carrot Pomace, Pulse Powder and Rice Flour–Based Extrudates”, *International Journal of Food Engineering*, 10 (1), 139-147.

Delgado-Nieblas C., Aguilar-Palazuelos E., Gallegos-Infante A., Rocha-Guzmán N., Zazueta-Morales J., Caro-Corrales J., (2012), “Characterization and optimization of extrusion cooking for the manufacture of third-generation snacks with winter squash (*Cucurbita moschata* D.) flour”, *Cereal Chemistry*, 89 (1), 65-72.

Ding Q. B., Ainsworth P., Plunkett A., Tucker G., Marson H., (2006), “The effect of extrusion conditions on the functional and physical properties of wheat-based expanded snacks”, *Journal of Food Engineering*, 73 (2), 142-148.

Dueik V., Sobukola O., Bouchon P., (2014), “Development of low-fat gluten and starch fried matrices with high fiber content”, *LWT-Food Science and Technology*, 59 (1), 6-11.

Ernoul V., Moraru C. I., Kokini J. L., (2002), “Influence of fat on expansion of glassy amylopectin extrudates by microwave heating”, *Cereal Chemistry*, 79 (2), 265-273.

Fallahi P., Muthukumarappan K., Rosentrater K. A., (2016), “Functional and Structural Properties of Corn, Potato, and Cassava Starches as Affected by a Single-Screw Extruder”, *International Journal of Food Properties*, 19 (4), 768-788.

Fallahi P., Rosentrater K. A., Muthukumarappan K., Tulbek M., (2013), “Effects of steam, moisture, and screw speed on physical properties of DDGS-based extrudates”, *Cereal Chemistry*, 90 (3), 186-197.

Farkas B. E., Singh R. P., Rumsey T. R., (1996), “Modeling heat and mass transfer in immersion frying. I, model development”, *Journal of Food Engineering*, 29 (2), 211-226.

Gimeno E., Moraru C. I., Kokini J. L., (2004), “Effect of xanthan gum and CMC on the structure and texture of corn flour pellets expanded by microwave heating”, *Cereal Chemistry*, 81 (1), 100-107.

Gomez M. H., Aguilera J. M., (1983), “Changes in the starch fraction during extrusion-cooking of corn”, *Journal of Food Science*, 48 (2), 378-381.

Gonzalez R. J., De Greef D. M., Torres R. L., Borrás F. S., Robutti J., (2004), “Effects of endosperm hardness and extrusion temperature on properties of products obtained with grits from two commercial maize cultivars”, *LWT-Food Science and Technology*, 37 (2), 193-198.

Grenus K. M., Hsieh F., Huff H. E., (1993), “Extrusion and extrudate properties of rice flour”, *Journal of Food Engineering*, 18 (3), 229-245.

Gulati P., Weier S. A., Santra D., Subbiah J., Rose D. J., (2016), “Effects of feed moisture and extruder screw speed and temperature on physical characteristics and

antioxidant activity of extruded proso millet (*Panicum miliaceum*) flour”, *International Journal of Food Science and Technology*, 51 (1), 114-122.

Gumusay O. A., Seker M., Sadikoglu H., (2019), “Response surface methodology for evaluation of the effects of screw speed, feed moisture and xanthan gum level on functional and physical properties of corn half products”, *LWT-Food Science and Technology*, 111, 622-631.

Hanna M. A., Chinnaswamy R., Gray D. R., Miladinov V. D., (1997), “Extrudates of starch-xanthan gum mixtures as affected by chemical agents and irradiation”, *Journal of Food Science*, 62 (4), 816-820.

Harper J. M., (1994), “The Technology of Extrusion Cooking”, Blackie Academic & Professional.

Hashimoto J. M., Grossmann M. V. E., (2003), “Effects of extrusion conditions on quality of cassava bran/cassava starch extrudates”, *International Journal of Food Science and Technology*, 38 (5), 511-517.

Hoover R., Ratnayake W. S., (2005), “Determination of Total Amylose Content of Starch”. In: R. E. Wrolstad, T. E. Acree, E. A. Decker, Editors, “Handbook of Food Analytical Chemistry: Water, Proteins, Enzymes, Lipids, and Carbohydrates”, John Wiley & Sons.

Htoon A., Shrestha A. K., Flanagan B. M., Lopez-Rubio A., Bird A. R., Gilbert E. P., Gidley M. J., (2009), “Effects of processing high amylose maize starches under controlled conditions on structural organisation and amylase digestibility”, *Carbohydrate Polymers*, 75 (2), 236-245.

Kawas M. L., Moreira R. G., (2001), “Effect of degree of starch gelatinization on quality attributes of fried tortilla chips”, *Journal of Food Science*, 66 (2), 300-306.

Kebede L., Worku S., Bultosa G., Yetneberek S., (2010), “Effect of extrusion operating conditions on the physical and sensory properties of tef (*Eragrostis tef* [Zucc.] Trotter) flour extrudates”, *Ethiopian Journal of Applied Science and Technology*, 1 (1), 27-38.

Koop T., Luo B., Tsias A., Peter T., (2000), “Water activity as the determinant for homogeneous ice nucleation in aqueous solutions”, *Nature*, 406, 611-614.

Lawton B. T., Henderson G. A., Derlatka E. J., (1972), “The effects of extruder variables on the gelatinisation of corn starch”, *The Canadian Journal of Chemical Engineering*, 50 (2), 168-172.

Lazou A., Krokida M., (2010), “Functional properties of corn and corn-lentil extrudates”, *Food Research International*, 43 (2), 609-616.

Lee E. Y., Lim K. I., Lim J. K., Lim S. T., (2000), “Effects of gelatinization and moisture content of extruded starch pellets on morphology and physical properties of microwave-expanded products”, *Cereal Chemistry*, 77 (6), 769-773.

- Liu C., Zhang Y., Liu W., Wan J., Wang W., Wu L., Zuo N., Zhou Y., Yin Z., (2011), "Preparation, physicochemical and texture properties of texturized rice produce by improved extrusion cooking technology", *Journal of Cereal Science*, 54 (3), 473-480.
- Liu Y., Hsieh F., Heymann H., Huff H. E., (2000), "Effect of process conditions on the physical and sensory properties of extruded oat-corn puff", *Journal of Food Science*, 65 (7), 1253-1259.
- Lowe J. A., Kershaw S. J., (1995), "Water activity-moisture content relationship as a predictive indicator for control of spoilage in commercial pet diet components", *Animal Feed Science and Technology*, 56 (3-4), 187-194.
- Lue S., Huff H. E., (1991), "Extrusion cooking of corn meal and sugar beet fiber: effects on expansion properties, starch gelatinization, and dietary fiber content", *Cereal Chemistry*, 68 (3), 227-34.
- Maga J. A., Fapojuwo O. O., (1988), "The effect of various hydrocolloids on some physical properties of extruded corn grits", *International Journal of Food Science and Technology*, 23 (1), 49-56.
- Maga J. A., Kim C. H., Wolf C. L., (1991), "The effect of gellan gum addition on corn grits extrusion", *Food Hydrocolloids*, 5 (5), 435-441.
- Maitre A., Rathod R. P., Annapure U. S., (2017), "Effect of hydrocolloids and process parameters on the extrusion behavior of pearl millet grits", *International Food Research Journal*, 24 (4), 1538-1544.
- McPherson A. E., Bailey T.B., Jane J., (2000), "Extrusion of cross-linked hydroxypropylated corn starches I. pasting properties", *Cereal Chemistry*, 77 (3), 320-325.
- Mezreb K., Goullieux A., Ralainirina R., Queneudec M., (2003), "Application of image analysis to measure screw speed influence on physical properties of corn and wheat extrudates", *Journal of Food Engineering*, 57 (2), 145-152.
- Moraru C. I., Kokini J. L., (2003), "Nucleation and expansion during extrusion and microwave heating of cereal foods", *Comprehensive Reviews in Food Science and Food Safety*, 2 (4), 147-165.
- Mudgett R. E., (1982), "Electrical-properties of foods in microwave processing", *Food Technology*, 36, 109-115.
- Natabirwa H., Nakimbugwe D., Lungaho M., Muyonga J. H., (2018), "Optimization of Roba1 extrusion conditions and bean extrudate properties using response surface methodology and multi-response desirability function" *LWT-Food Science and Technology*, 96, 411-418.

Noorakmar A. W., Cheow C. S., Norizzah A. R., Zahid A. M., Ruzaina I., (2012), “Effect of orange sweet potato (*Ipomoea batatas*) flour on the physical properties of fried extruded fish crackers”, *International Food Research Journal*, 19 (2), 657.

O’shea N., Arendt E., Gallagher E., (2014), “Enhancing an extruded puffed snack by optimising die head temperature, screw speed and apple pomace inclusion”, *Food and Bioprocess Technology*, 7 (6), 1767-1782.

Oginni O. C., Sobukola O. P., Henshaw F. O., Afolabi W. A. O., Munoz L., (2015), “Effect of starch gelatinization and vacuum frying conditions on structure development and associated quality attributes of cassava-gluten based snack”, *Food Structure*, 3, 12-20.

Onwulata C. I., Smith P. W., Konstance R. P., Holsinger V. H., (2001), “Incorporation of whey products in extruded corn, potato or rice snacks” *Food Research International*, 34 (8), 679-687.

Osman M. G., Sahai D., Jackson D. S., (2000), “Oil absorption characteristics of a multigrain extrudate during frying: Effect of extrusion temperature and screw speed”, *Cereal Chemistry*, 77 (2), 101-104.

Perez A. A., Drago S. R., Carrara C. R., De Greef D. M., Torres R. L., Gonzalez R. J., (2008), “Extrusion cooking of a maize/soybean mixture: Factors affecting expanded product characteristics and flour dispersion viscosity”, *Journal of Food Engineering*, 87 (3), 333-340.

Phillips G.O., Williams, P.A., (1995), “Interaction of Hydrocolloids in Food Systems”, In A.G. Gaonkar, Editor, “Ingredient Interactions: Effects on Food Quality”, Marcel Dekker.

Pinthus E. J., Saguy I. S., (1994), “Initial interfacial tension and oil uptake by deep-fat fried foods”, *Journal of Food Science*, 59, 805–807.

Primo-Martín C., Van Deventer H., (2011), “Deep-fat fried battered snacks prepared using super heated steam (SHS): Crispness and low oil content”, *Food Research International*, 44 (1), 442-448.

Rashid S., Rakha A., Anjum F. M., Ahmed W., Sohail M., (2015), “Effects of extrusion cooking on the dietary fibre content and water solubility index of wheat bran extrudates”, *International Journal of Food Science and Technology*, 50 (7), 1533-1537.

Ravindran G., Carr A., Hardacre A., (2011), “A comparative study of the effects of three galactomannans on the functionality of extruded pea–rice blends”, *Food Chemistry*, 124 (4), 1620-1626.

Seker M., (2004), “Distribution of the residence time in a single-screw extruder with differing numbers of mixing elements”, *International Journal of Food Science and Technology*, 39 (10), 1053-1060.

Seker M., (2005), "Residence time distributions of starch with high moisture content in a single-screw extruder", *Journal of Food Engineering*, 67 (3), 317-324.

Seker M., (2011), "Extrusion of snacks, breakfast cereals, and confectioneries". In M. Maskan, A. Altan, Editors, "Advances in Food Extrusion Technology", CRC Press.

Seker M., Hanna M. A., (2005), "Cross-linking starch at various moisture contents by phosphate substitution in an extruder", *Carbohydrate Polymers*, 59 (4), 541-544.

Seker M., Sadikoglu H., Hanna M. A., (2004), "Properties of cross-linked starch produced in a single screw extruder with and without a mixing element", *Journal of Food Process Engineering*, 27 (1), 47-63.

Singh J., Singh N., (2004), "Effect of process variables and sodium alginate on extrusion behavior of nixtamalized corn grit", *International Journal of Food Properties*, 7 (2), 329-340.

Sobukola O. P., Babajide J. M., Ogunsade O., (2013), "Effect of brewers spent grain addition and extrusion parameters on some properties of extruded yam starch-based pasta", *Journal of Food Processing and Preservation*, 37 (5), 734-743.

Stojceska V., Ainsworth P., Plunkett A., Ibanoglu S., (2008), "The recycling of brewer's processing by-product into ready-to-eat snacks using extrusion technology", *Journal of Cereal Science*, 47 (3), 469-479.

Stojceska V., Ainsworth P., Plunkett A., İbanoğlu Ş., (2009), "The effect of extrusion cooking using different water feed rates on the quality of ready-to-eat snacks made from food by-products", *Food Chemistry*, 114 (1), 226-232.

Suhaila M., Norhasyimah A. H., (1994), "Effects of various food components on the expansion, oil absorption and crispiness of fried rice dough", *Pertanika Journal of Tropical Agricultural Science*, 17 (1), 7-12.

Suknark K., Phillip R. D., Huang Y. W., (1999), "Tapioca-fish and tapioca-peanut snacks by twin-screw extrusion and deep-fat frying", *Journal of Food Science*, 64 (2), 303-308.

Sun Y., Muthukumarappan K., (2002), "Changes in functionality of soy-based extrudates during single-screw extrusion processing", *International Journal of Food Properties*, 5 (2), 379-389.

Teba C. D. S, Silva E. M. M. D., Chávez D. W. H., Carvalho C. W. P. D., Ascheri J. L. R., (2017), "Effects of whey protein concentrate, feed moisture and temperature on the physicochemical characteristics of a rice-based extruded flour", *Food Chemistry*, 228, 287-296.

Thakur S., Saxena D. C., (2000), "Formulation of extruded snack food (gum based cereal-pulse blend): optimization of ingredients levels using response surface methodology", *LWT-Food Science and Technology*, 33 (5), 354-361.

Tovar-Jiménez X., Aguilar-Palazuelos E., Gómez-Aldapa C., Caro-Corrales J. J., (2016), "Microstructure of a third generation snack manufactured by extrusion from potato starch and orange vesicle flour", *Journal of Food Processing and Technology*, 7 (3).

Tovar-Jiménez X., Caro-Corrales J., Gómez-Aldapa C. A., Zazueta-Morales J., Limón-Valenzuela V., Castro-Rosas J., Hernández-Ávila J., Aguilar-Palazuelos E., (2015), "Third generation snacks manufactured from orange by-products: physicochemical and nutritional characterization", *Journal of Food Science and Technology*, 52 (10), 6607-6614.

Van der Sman R. G. M., Bows J. R., (2017), "Critical factors in microwave expansion of starchy snacks", *Journal of Food Engineering*, 211, 69-84.

Wang J., Jin, Z., Yuan, X., (2007), "Preparation of resistant starch from starch–guar gum extrudates and their properties", *Food Chemistry*, 101 (1), 20-25.

Xie F., Halley P. J., Avérous L., (2012), "Rheology to understand and optimize processability, structures and properties of starch polymeric materials", *Progress in Polymer Science*, 37 (4), 595-623.

Xu E., Pan X., Wu Z., Long J., Li J., Xu X., Jin Z., Jiao A., (2016), "Response surface methodology for evaluation and optimization of process parameter and antioxidant capacity of rice flour modified by enzymatic extrusion", *Food Chemistry*, 212, 146-154.

Yagci S., Gogus F., (2008), "Response surface methodology for evaluation of physical and functional properties of extruded snack foods developed from food-by-products", *Journal of Food Engineering*, 86 (1), 122-132.

Yu L., Ramaswamy H. S., Boye, J., (2012), "Twin-screw extrusion of corn flour and soy protein isolate (SPI) blends: a response surface analysis", *Food and Bioprocess Technology*, 5 (2), 485-497.

Zhuang H., Feng T., Xie Z., Toure A., Xu X., Jin, Z., Su, Q., (2010), "Effect of Mesona Blumes gum on physicochemical and sensory characteristics of rice extrudates", *International Journal of Food Science and Technology*, 45 (11), 2415-2424.

BIOGRAPHY

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APPENDICES

Appendix A: Publications

Gumusay O. A., Seker M., Sadikoglu H., (2019), “Response surface methodology for evaluation of the effects of screw speed, feed moisture and xanthan gum level on functional and physical properties of corn half products”, LWT-Food Science and Technology, 111, 622-631.