

**Production and Properties of Snack Foods Developed by
Extrusion from Composite of Barley, and Tomato and
Grape Pomaces**

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Dedicated to my parents

ABSTRACT

PRODUCTION AND PROPERTIES OF SNACK FOODS DEVELOPED BY EXTRUSION FROM COMPOSITE OF BARLEY, AND TOMATO AND GRAPE POMACES

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The barley flour and barley flour-pomace blends (tomato, grape) were extruded in a 30 mm APV co-rotating twin-screw extruder. Response surface methodology using a central composite design was used to evaluate the effects of independent variables, namely die temperature, screw speed for barley flour extrudates as well as pomace level for barley-pomace extrudates on system parameters (specific mechanical energy, die pressure and die melt temperature), physical properties (sectional expansion index and bulk density), color parameters (Hunter L , a , b and ΔE), functional properties (water absorption and solubility indices), textural properties (peak force, slope and distance) and antioxidant activity and total phenolics, degree of gelatinization, *in vitro* starch digestibility and β -glucan content. Selected samples of extrudates were evaluated sensorially in terms of appearance (color and porosity), taste (bran flavor, bitterness, sweetness and tomato flavor), off-odor, texture (hardness, crispness and brittleness) and overall acceptability by semi-trained panelists. In addition, total fiber content of some extrudates was determined. The system parameters and product properties were most affected by changes in temperature and to a lesser extent by screw speed and it was observed that those properties were also dependent on the changes of pomace level for barley-pomace blend extrudates. Extrusion cooking significantly reduced both antioxidant activities and total phenolics and increased digestibility of all extrudate samples. Results showed that the content of β -glucan is higher in barley flour than in extrudates of barley flour and pomace blends.

Key words: Extrusion, Barley, Tomato pomace, Grape pomace, Response surface methodology, Independent variables

ÖZET

DOMATES VE ÜZÜM POSALARI İLE ARPADAN EKSTRÜZYONLA GELİŞTİRİLEN ÇEREZ GIDALARIN ÜRETİMİ VE ÖZELLİKLERİ

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Arpa unu, arpa unu-posa karışımları (domates, üzüm) 30 mm APV model paralel dönebilir çift vidalı ekstrüder ile ekstrüde edildi. Merkezi karma tasarımı ile yüzey tepki metodu, arpa ürünleri için bağımsız değişkenler olan kalıp sıcaklığı ve vida dönüş hızı, arpa-posa karışımlarından üretilmiş ürünler için de ek olarak posa oranının sistem parametreleri (spesifik mekanik enerji, kalıp basıncı ve kalıp erime sıcaklığı), fiziksel özellikler (enine genleşme indeksi ve yığın yoğunluk), renk parametreleri (Hunter L , a , b ve ΔE), fonksiyonel özellikler (su soğurma ve suda çözünme indeksleri), tekstürel özellikler (sertlik, gevreklik ve kırılgenlik), antioksidan aktivite, toplam fenolik bileşenleri, jelatinizasyon derecesi, nişasta sindirilebilirliği ve β -glucan miktarı üzerine olan etkilerini değerlendirmek için kullanıldı. Arpa ve arpa-posa karışımı ekstrüde ürünlerden seçilmiş örnekler, yarı eğitimli panelistler tarafından duyuşal olarak görünüş (renk ve gözenekli yapı), tat (kepek tadı, acılık, tatlılık ve domates tadı), kötü koku, tekstür (sertlik, gevreklik ve kırılgenlik) ve genel kabul edilebilirlik açısından değerlendirildi. Ayrıca, bazı ürünlerde toplam lif içeriği tayini yapıldı. Sistem parametreleri ve ürün özellikleri en çok sıcaklık ve daha az miktarda vida dönüş hızındaki değişikliklerden etkilendi ve arpa-posa karışımı ekstrüde ürün özelliklerinin aynı zamanda posa miktarına da bağılı olduğu gözlemlendi. Ekstrüzyonla pişirme, örneklerin antioksidan aktivitelerini ve toplam fenolik madde bileşenlerini önemli derecede azalttı ve sindirilebilirliği artırdı. Sonuçlar, arpa unundaki β -glucan miktarının arpa ve arpa-posa karışımlarından elde edilen ürünlerdeki β -glucan miktarına göre daha yüksek olduğunu gösterdi.

Anahtar Kelimeler: Ekstrüzyon, Arpa, Domates posası, Üzüm posası, Yüzey tepki metodu, Bağımsız değişkenler

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NOMENCLATURE

AA	Antioxidant activity
AACC	American Association of Cereal Chemists
ANOVA	Analysis of variance
AOAC	Association of Official Analytical Chemists
BD	Bulk density
BG	β -Glucan content
CV	Coefficient of variance
D_i	Distance
DG	Degree of gelatinization
DPPH	2,2-diphenyl-1-picrylhydrazyl
DSC	Differential scanning calorimetry
EC	European Commission Regulation
FDA	Food and Drug Administration
GOPOD	Glucose oxidase peroxidase reagent
HIV	Human immunodeficiency virus
LDL	Low density lipoprotein
LEI	Longitudinal expansion index
DP	Die pressure
PF	Peak force
R^2	Coefficient of determination
S	Slope
SD	<i>In vitro</i> starch digestibility
SEI	Sectional expansion index
SME	Specific mechanical energy
T	Die melt temperature
TP	Total phenolic content
WAI	Water absorption index
WSI	Water solubility index
ΔE	Total color change

CHAPTER 1

INTRODUCTION

In recent years, there is an increasing demand for conversion of fruit and vegetable by-products into useful products. The primary motivation is to minimize environmental impact of these by-products and to utilize valuable constituents that remain, such as lycopene, antioxidants and dietary fiber. An appropriate management of the wastes from fruit and vegetable processing might considerably reduce the cost of transport and utilization. Another advantage, in economic terms, is the potential reuse of pomace as a raw material for the manufacture of new products, thus making it possible to reduce the troublesome seasonal pattern from which some industries suffer (Nawirska and Kwaśniewska, 2005).

Tomato is one of the most popular vegetables and an integral part of human diet worldwide. Significant amounts are consumed in the form of processed products such as juice, paste, puree, ketchup, sauce and salsa (Del Valle et al. 2006). During tomato processing by-product, known as tomato pomace, consists of the dried and crushed skins and seeds of fruit. It has been reported that skin is an important component of pomace and is considered as sources of lycopene. A number of studies have indicated that a lycopene rich diet lowers the risk of certain chronic diseases such as cardiovascular disease and cancer, particularly prostate cancer and pancreatic cancer (Johnson, 2000; Rao and Agarwal, 2000). Fiber is the major compound of tomato by-product up to 50% dry weight basis (Del Valle et al. 2006). Thus tomato by-product can be considered as a potential source of fiber for human food formulations. By this way the use of tomato processing by-products could provide gaining valuable substances and at the same time reduce the waste disposal problem. Grape is one of the world's largest fruit crops and the press residues resulting from winemaking (that is, seeds, skins and highly pressed must) are rich in phenolics and generated in huge amounts (Kammerer et al. 2004). In the zones of grape and wine production, a great quantity of residues are generated whose storage, transformation or elimination pose problems in economic terms. For this reason, the recovery of the

antioxidant compounds present in these by-products could represent an interesting advance in the maintenance of the environmental equilibrium (Alonso et al. 2002). Numerous studies have demonstrated the antioxidant and health promoting effects of phenolic compounds present in grapes and wine, particularly in relation to cardiovascular diseases (Scalbert et al. 2005). Grape pomace therefore represents a potentially valuable source of phenolic antioxidants that may have technological applications as functional food ingredients and possible nutritional benefits.

With the growing awareness of the beneficial effects of healthy diet on the quality of life as well as on cost-effectiveness of health care, the food industry is facing the challenge of developing new products with special health enhancing characteristics. To meet this challenge, it must identify new sources of nutraceuticals and other natural and nutritional materials with the desirable functional characteristics. Although less common in food formulations than other cereals, barley has the ability to promote good consumer health through its nutritional components such as fiber (both soluble and insoluble fiber), antioxidants and B-vitamins. Considering the high nutritional quality of barley and the competitive nature of food markets for functional food, an extruded barley based snack product has potential. Dietary fiber has received increased attention recently. As consumers become more concerned about eating food with health benefits, barley, which is naturally healthy, easily available and inexpensive crop is strongly favored for increased incorporation into human diet (Czuchajowska et al. 1998). The dietary fiber content of barley contributes to its nutritional value, making it a highly desirable cereal grain today.

Barley is among the most ancient of the cereal crops. It is the fourth most important cereal in the world after wheat, rice and corn (Jadhav et al. 1998). Barley is a new and different flavor alternative for today's consumers. The nutritional profile of barley places it in a prime position for development as a functional food, a food with a health benefit. Barley flour products have the potential to appeal to health conscious consumers of all ages as a functional food, or simply as an alternative to existing products. Barley contains high levels of β -glucans, which are important contributors to dietary fiber, has significant blood cholesterol lowering effects (McIntosh et al. 1991; Martinez et al. 1992). One mechanism by which they may exert their effects is through increasing intestinal viscosity and in this way reducing

cholesterol and glucose absorption (Anderson et al. 1990), which is beneficial in the management of diabetes (Klopfenstein, 1988). There is a prime opportunity for increased commercialization of barley flour today due to increasing availability in the supply chain, additional research confirming the health benefits, consumer demand for healthy foods and the favorable reception of barley by consumers.

One viable method for utilization of fruit and vegetable by-products into useful products is extrusion processing due to its versatility, high productivity, relative low cost, energy efficiency and lack of effluents. Successful incorporation of tomato pomace into extruded products that deliver physiologically active components represents a major opportunity for food processors providing the consumer a healthy barley-based product to choose from which is currently lacking in the marketplace.

Extrusion cooking is an important and popular food processing technique classified as a high temperature/short time process to produce fiber-rich products (Gaosong and Vasanthan, 2000; Vasanthan et al. 2002). In the extruder, the food mix is thermomechanically cooked to high temperature, pressure and shear stress which are generated in the screw-barrel assembly. The cooked melt is then texturized and shaped in the die (Arhaliass et al. 2003). The thermomechanical action during extrusion brings about gelatinization of starch, denaturation of protein and inactivation of enzymes, microbes and many anti-nutritional factors; all this occurs in a shear environment, resulting in a plasticized continuous mass (Bhattacharya and Prakash, 1994).

The recovery of food wastes is rapidly expanding around the world. Considering this and valuable substances of fruit and vegetable by-products in particular antioxidants, dietary fibers, minerals and essential fatty acids which are beneficial for health, the basic goal is to put into practice processes and technologies that help conversion of by-products into useful products.

Therefore, the objectives of this research are:

1. To amplify the possibilities of utilization and recycling of tomato and grape pomaces from food chain with the combination of barley through the production of extruded snacks using extrusion process.
2. To investigate the effects of extrusion die temperature, screw speed and pomace level on the system parameters (specific mechanical energy, die

pressure and die melt temperature), physical properties (sectional expansion index and bulk density), color parameters (Hunter L , a , b and ΔE), functional properties (water absorption and solubility indices), textural properties (peak force (measure of hardness), slope (measure of crispness) and distance (measure of brittleness)), antioxidant activity, total phenolic content, degree of gelatinization, *in vitro* starch digestibility and β -glucan content.

3. To increase manufacturer's awareness and visibility of barley as a human food, motivate consumers to use more barley in their diets and to communicate the nutritional benefits of barley to consumers and health/nutrition professional by producing such snack foods.

In summary; the aim of this study is investigating the transformation and upgrading of food processing-derived plant based by-products with barley into environmentally friendly added value food products which are acceptable to consumer taste. The so-called product is considered to contain significant amount of dietary fiber, antioxidant and phenolic content in addition to the basic nutrients.

CHAPTER 2

LITERATURE REVIEW

2.1 Fruit and Vegetable By-products

The food industry produces large volumes of wastes in particular fruit and vegetable wastes resulting from production, preparation and consumption of food. Especially, fruit and vegetable processing wastes pose increasing disposal and severe loss of valuable substances e.g., dietary fiber, lycopene, antioxidants, pectin, essential fatty acids, antimicrobials, minerals etc. These substances have many health-promoting benefits. Dietary fiber in wastes acts as bulking agent, normalizing intestinal motility and preventing diverticular disease. Some types may also be important in reducing colonic cancer, in lowering serum cholesterol levels and in preventing hyperglycemia in diabetic patients (Larrea et al. 2005a; Thebaudin et al. 1997). Pectic substances of the dietary fiber have a hypocholesterolemic effect because they complex with bile acids and prevent their reabsorption in small intestines. The occurrence of prostate cancer in men and breast cancer in women as well as risk of cardiovascular disease may be reduced by lycopene intake (Kris-Etherton et al. 2002). Antioxidants in fruit and vegetable wastes reduce oxidative stress through inhibition of lipid peroxidation, a factor that is currently linked to a host of diseases such as cancer and heart disease. Fruit and vegetable wastes, for example, potato peel are rich in dietary fiber (Arora et al. 1993; Camire et al. 1997; Laufenberg et al. 2003) and tomato, grape and apple pomaces are probably most important sources of dietary fiber and antioxidants (Bobek et al. 1998; Moure et al. 2001).

Apple pomace has been shown to be good source of polyphenols which are predominantly localized in the peels. It was reported that citrus seeds and peels were found to possess high antioxidant activity (Schieber et al. 2001). Lu and Foo (1997) investigated apple pomace from juice processing as a source of fine chemicals. Polyphenols extracted from apple pomace could be successfully separated by column chromatography which resulted in the isolation of epicatechin, caffeic acid, three dihydrochalcone glycosides and five different quercetin glycosides. The total level of

the polyphenols in the pomace was found about 7.24 g/kg dry matter, of which more than half consisted of quercetin glycosides (4.46 g/kg). Their result showed that apple pomace contained a high level of polyphenols which could be commercially exploited. Research has been carried out to evaluate some functional properties of fiber concentrates from apple and citrus fruit residues in order to use them as potential fiber sources in the enrichment of foods (Figuerola et al. 2005). It was found that all the fiber concentrates had a high content of dietary fiber (between 44.2 and 89.2 g/100 g dry matter), with a high proportion of insoluble dietary fiber and the characteristics found in the concentrates suggested many potential applications such as volume replacement, thickening or texturizing in the development of foods reduced in calories and rich in dietary fiber.

In another study, Nawirska and Kwaśniewska (2005) determined the amounts of particular dietary fiber fractions in samples containing apple, black currant, chokeberry, pear, cherry and carrot pomace. The results revealed that in each pomace sample, pectins occurred in the smallest amounts and the content of lignin was very high for black currant and cherry pomace or comparatively high for pear, chokeberry, apple and carrot pomace, respectively. Comparison of the contents of hemicellulose and pectin in the investigated study of pomace showed that the highest contents of these species were in chokeberry pomace (41%) and the lowest in apple pomace (36%). It could therefore be anticipated that the chokeberry and apple pomaces which had the highest concentrations of these species, would be equally good sorbents for heavy metals (Nawirska and Kwaśniewska, 2005).

Peschel et al. (2006) studied eleven fruit and vegetable by-products and two minor crops for industrial exploitation potential by determination of their extraction yield, total phenolic content and antioxidant activity. They resulted that extracts with the highest activity, economic justification and phenolic content were obtained from apple (48.6 mg Gallic acid equivalents (GAE)/g dry extract), pear (60.7 mg GAE/g), tomato (61.0 mg GAE/g), golden rod (251 mg GAE/g) and artichoke (514.2 mg GAE/g). Apple, golden rod and artichoke byproducts has been extracted at pilot plant scale and their antioxidant activity has been confirmed by determination of their free radical scavenging activity and the inhibition of simulated linoleic acid peroxidation. The authors demonstrated the possibility of recovering high amounts of phenolics

with antioxidant properties from fruit and vegetable residuals not only for food but also cosmetic applications.

Grape pomace is the press residue remaining when grapes are processed for wine-making. The pomace consists of pressed skins, disrupted cells from the grape pulp, seeds and stems. Among fruits, grapes constitute one of the major sources of phenolic compounds and pomace is particularly rich in phenols (Meyer et al. 1998). Anthocyanins, catechins, flavonol glycosides, phenolic acids and alcohols and stilbenes are the principal phenolic constituents of grape pomace (Schieber et al. 2001). Numerous studies have demonstrated the antioxidant and health promoting effects of phenolic compounds present in grapes and wine, particularly in relation to cardiovascular diseases (Scalbert et al. 2005). The evidence is clearly mounting that grape polyphenols are absorbed by the body and increase total antioxidative capacity of blood plasma or decrease peroxidation of low density lipoprotein (LDL) (Shrikhande, 2000). Grape pomace therefore represents a potentially valuable source of phenolic antioxidants that may have technological applications as functional food ingredients and possible nutritional benefits. Grape pomace is also characterized by a high content of dietary fiber and associated polyphenols (Valiente et al. 1995) and could be used as a potential ingredient for dietary fiber-rich supplements (Martín-Carrón et al. 2000).

Grape seeds are rich sources of polyphenolics which have been shown to act as strong antioxidants and exert health promoting effects (Jayaprakasha et al. 2001). Meyer et al. (1997) studied phenolic extracts from 14 different types of fresh grapes in terms of the inhibition of human LDL oxidation in vitro. According to their results, the relative antioxidant activity increased with grape seed crushing and with longer extraction times. This seed extraction provided greater concentration of flavan-3-ol flavonoid compounds of antioxidation potential in inhibiting LDL oxidation. Natural flavonoids can donate hydrogen to and/or react with superoxide anions, hydroxyl radicals and lipid peroxy radicals, all of which can cause lipid peroxidation in vitro, leading to LDL oxidation implicated in the development of atherosclerosis (Shrikhande, 2000). In a study of antioxidant activity of grape pomace, antioxidant-rich fractions have been extracted from grape pomace using ethyl acetate, methanol and water (Chidambara Murthy et al. 2002). As the methanol extract showed high antioxidant activity also, it might be directly correlated to the

high phenolic content of the methanol extract of grape pomace. The data obtained in their study revealed that the grape pomace extracts are free radical scavengers and primary antioxidants, which react with free radicals. The ability of the grape pomace extract to quench hydroxyl radicals seems to directly relate to the prevention of propagation of the process of lipid peroxidation and the extract seems to be a good scavenger of active oxygen species, thus reducing the rate of chain reaction (Chidambara Murthy et al. 2002).

Tomato is one of the most popular vegetables, used as a salad, in food preparations and as juice, soup, puree, ketchup or paste. During tomato processing a by-product, known as tomato pomace, is generated. This by-product represents, at most, 4% of the fruit weight (Del Valle et al. 2006). Tomato pomace consists of the dried and crushed skins and seeds of the fruit (Tadeu-Pontes et al. 1996). Del Valle et al. (2006) evaluated the chemical composition of tomato pomace collected at different steps during tomato processing for paste in order to assess the quality of this by-product. It was concluded that average value of tomato pomace composition (on a dry weight basis) was 59.03% fiber, 25.73% total sugars, 19.27% protein, 7.55% pectins, 5.85% total fat and 3.92% mineral content, with no great differences between samples collected at different steps during tomato processing. Tomato pomace could be used as a potential source of fiber, protein or fat (most of which is polyunsaturated).

Research has been done to determine the fatty acid composition and physicochemical characteristics of the oils extracted from industrial tomato seed wastes from hot and cold break treatments (Cantarelli et al. 1993). Results indicated that the oil yield of tomato seeds was 19.0 and 14.5% for cold and hot break treatments. The total saturated and unsaturated fatty acid composition was 29.4 and 70.6% for cold break seed oil and 31.3 and 68.6% for hot break seed oil. It was found that in both treatments palmitic acid was the major saturated fatty acid, followed by stearic acid. Linoleic acid was the major unsaturated fatty acid followed by oleic acid. Both oleic and linoleic acids added up to over 60% of total fatty acids, being higher in cold break seed oils. The authors concluded that both treatments produced high nutritional oil quality. The skin, another important component of pomace, was utilized for extracting the red pigment using organic solvents (Tonucci et al. 1995). Lycopene is an excellent natural food color and also serves as a micronutrient with important

health benefits (Kaur et al. 2005). Baysal et al. (2000) clearly stated that a large quantity of carotenoids is lost as waste in tomato processing. Supercritical CO₂ extraction of lycopene and β -carotene from tomato paste waste resulted in recoveries of up to 50% when ethanol was added by 5% (Baysal et al. 2000). Lycopene is a major carotenoid in human serum tissues and in the diet. It is unique among the carotenoids in that it has one major food source-tomatoes. Epidemiologic studies suggest that a diet rich in lycopene is related to a decreased risk of certain diseases, particularly cancers of digestive tract, prostate and pancreas as well as cardiovascular disease and HIV infection. The chemoprotective effect of lycopene is thought to be due to its role as antioxidant (Johnson, 2000).

Toor and Savage (2005) determined the major antioxidants and antioxidant activity in different fractions (skin, seeds and pulp) of three tomato cultivars in New Zealand. It was found that the skin fraction of all cultivars had higher levels of total phenolics, total flavonoids, lycopene, ascorbic acid and antioxidant activity compared to their pulp and seed fractions. The skin and seeds of all cultivars on average contributed 53% to the total phenolics, 52% to the total flavonoids, 48% to the total lycopene, 43% to the total ascorbic acid and 52% to the total antioxidant activity present in tomatoes. Rao and Agarwal (2000) reviewed the role of antioxidant lycopene in cancer and heart disease. Epidemiological and a small number of animal and experimental studies have provided evidence in support for its protective role in heart disease and cancer. Dietary intakes of tomatoes and tomato products containing lycopene have been shown to be associated with decreased risk of chronic diseases such as cancer and cardiovascular diseases.

In the past, fruit and vegetable wastes often have been dumped or used without treatment for animal feed or as fertilizers. In the last years, there is an increasing demand for their conversion into useful product for preventing pollution of the environment as well as for economic motives and utilization of valuable constituents from food processing wastes. Several researchers have been working on the development of multifunctional ingredients from fruit and vegetable residues and its application in different food products, e.g., pie fillings, crackers, cookies, bread. For example; apple pomace has been suggested in bakery products, pie filling and

cookies as a potential food ingredient (Wang and Thomas, 1989; Carson et al. 1994) and used as a source of dietary fiber in wheat bread (Masoodi and Chauhan, 1998).

In a study of Wang and Thomas (1989), apple pomace was utilized as a source of sugar and dietary fiber by incorporating dried apple pomace directly into bakery products. Total dietary fiber of freeze-dried apple pomace and drum-dried apple pomace was found to be 35.29 and 33.24%, respectively. Sensory evaluation demonstrated that the experimental muffins with 50% (w/w) of the plain wheat bran substituted by powdered apple pomace were significantly more desirable than the control bran muffins. The authors concluded that the high dietary fiber and fruit sugar content of the apple pomace could provide consumers with an alternative source of fiber and give bakers an option for reducing the amount of sugar and other fiber added to their products. In addition, this utilization of apple pomace could provide a savings to the juice processor through both the sale of pomace and the elimination of disposal costs.

Carson et al. (1994) have determined some compositional and physical properties of dried apple pomace and evaluated the acceptability of pie filling and oatmeal cookie containing pomace as an ingredient. Average dietary fiber was found to be 36.8%. Food products prepared from dried apple pomace was rated as liked moderately. In another study, apple pomace was incorporated into wheat flour in ratios of 2, 5, 8 and 11 % and blends were evaluated for their bread making quality. It was concluded that apple pomace could be incorporated in breads up to 5% without changing the quality of bread drastically (Masoodi and Chauhan, 1998).

Orange pulp has been extruded using a Brabender laboratory single screw extruder to modify the properties of the fiber components. The extrusion process decreased insoluble dietary fiber in 39.06% and soluble dietary fiber was increased by 80%. It was found that the barrel temperature was the most important variable followed by the interactions between barrel temperature and screw speed, which affected the insoluble and soluble dietary fiber contents (Larrea et al. 2005a). Orange pulp has been extruded to modify the functional and structural properties of fiber and used in cookies. The fiber content of extruded orange pulp has been found to be 74.87g/100g total alimentary fiber, 54.81g/100g insoluble alimentary fiber and 20.06g/100g soluble alimentary fiber. Biscuits of good technological quality and with a good level

of acceptance have been obtained by means of replacing up to 15 g in 100 g of the wheat flour with extruded orange pulp. It was reported that the energy value of the biscuits decreased with increased extruded orange pulp content, with reductions of 2.95, 6.62 and 10.67g/100g in total calories as compared to the control (Larrea et al. 2005b).

In another study, Camire et al. (1997) studied the effects of extrusion on dietary fiber of potato peel obtained from the abrasion peeling method used by chip manufacturers and the steam peeling procedure used for production of dehydrated potatoes and to identify differences in dietary fiber composition between these types of peels. They concluded that extrusion cooking reduced starch content and increased total dietary fiber in steam peels. Total dietary fiber in abrasion peels was not affected by extrusion. Extrusion increased soluble nonstarch polysaccharides in both types of peels. More glucose was recovered from insoluble fiber of extruded steam peels than from abrasion peels, suggesting that resistant starch might have been formed (Camire et al. 1997). Press residue from black currant juice production has been used for the development of a nutritious breakfast cereal using an extrusion process at high temperatures. The milled residual fraction with crushed seeds contained significant amounts of essential fatty acids, minerals and dietary fiber. They incorporated successfully the maximal useful amount of this ingredient, about 30%, in a mix containing 30% oat flour and oat bran, 30% potato starch, 7.5% sugar, 1.5% malt extract and 1% salt. It was reported that a product with more than 40% black currant press residue was hard and did not expand well (Tahvonen et al. 1998). Mendonça et al. (2000) investigated possibilities of corn bran utilization through the production of expanded snacks with high fiber using extrusion process. They recommended that the best combination of variables to produce snack with good sensory acceptability and high fiber content was 250 g/kg corn bran, 160 g/kg moisture, 190 °C temperature of extrusion and 4g/kg glycerol monostearate.

2.2 Barley

Barley (*Hordeum vulgare L.*) is among the most ancient of the cereal crops. Archeological studies have revealed that barley was cultivated by about 8000 B.C. in Iran. However, other evidence shows that barley in essentially the form that exists today was used at least 17,000 years ago in the Nile River Valley of Egypt. The

original area of cultivation has been reported to be in the Fertile Crescent of the Middle East, in present day Lebanon, Iran, Iraq and Turkey. This is also the most likely area of barley origin (Nilan and Ullrich, 1993).

Barley is a widely adapted crop and grows under a wider range of environmental conditions than any other cereal. It appears to require fewer heat units than other cereal cereals to reach physiological maturity and can therefore be grown successfully at higher latitudes and altitudes than other cereal crops. Barley is relatively tolerant to drought and to alkaline and salt conditions, but grows best in temperate regions of the world where growing seasons are long, cool and moderately dry (MacGregor, 1993).

Barley is the most common cereal in Turkey. Today, we think of barley as an animal feed or for malt, but there is good archaeological evidence for its role as human food. Allied with evidence from classical texts for the importance of barley as a human food, it is likely that ancient barley remains represent human food just as much as ancient wheat. Barley is sporadically noted as a food in Turkey in the present, but it is unclear when it ceased to be an important food for humans.

2.2.1 Kernel structure

The cereal grain is comprised of the caryopsis and the enclosing hull or husk formed from the lemma and palea. The caryopsis consists of the pericarp, integuments, aleurone layer, endosperm and germ or embryo (Figure 2.1) (MacGregor, 1993). The pericarp is developed from ovary walls and acts as a protective cover over the entire kernel. Integuments are outer cell layers and differentiate into the seed coat. The endosperm is a starchy mass embedded in a protein matrix and is a source of nutrients for the developing embryo. The aleurone is the outermost layer of the endosperm cells that contains protein bodies and enzymes connected with endosperm digestion. The embryo is located at the attachment end of the caryopsis on its dorsal side. The barley kernels are spindle-shaped, thicker in the center, and tapering toward each end. Some of the kernel characteristics are useful for distinguishing one cultivar from another, particularly when characterization has to be done from the threshed grain. In hulled barley, the lemma and palea (husk or hull) remain attached to the caryopsis when the grain is threshed, whereas in hull-less barley the caryopsis threshes free (Jadhav et al. 1998).

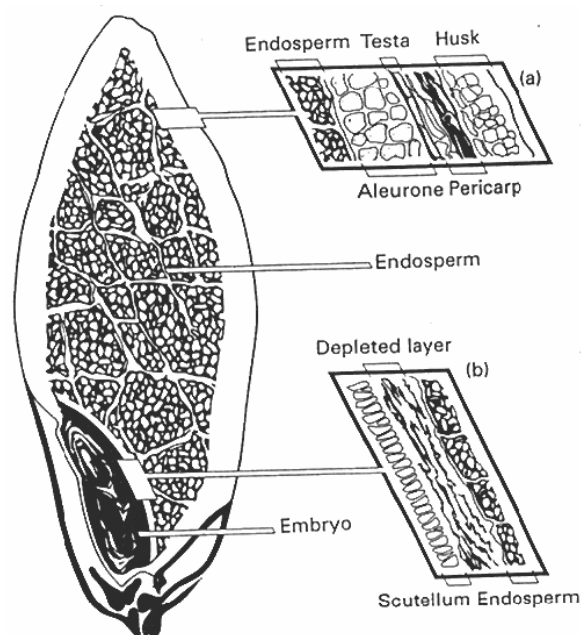


Figure 2.1 A longitudinal section of barley kernel a) Outer layers of kernel; b) junction between the embryo and endosperm

2.2.2 Composition

Grain composition and quality are influenced by prevailing environmental conditions, which include such variables as temperature, day length, water supply and the availability of soil minerals (Jadhav et al. 1998). Barley contains starch, protein, non-starch polysaccharides and lipids as its major components (Table 2.1) (MacGregor, 1993). Non-starch polysaccharides include cellulose, β -glucan and hemicellulose. Majority of the hemicelluloses in barley is arabinoxylans. The minor components are sugars, vitamins and minerals.

Table 2.1 Barley composition

Component	Content (% dry weight)
Starch	60-64
Arabinoxylans	4.4-7.8
β -Glucan	3.6-6.1
Cellulose	1.4-5.0
Simple carbohydrates (glucose, fructose, sucrose and maltose)	0.41-2.9
Oligosaccharides (raffinose, fructosans)	0.16-1.8
Proteins	8-15
Lipids	2-3
Minerals	2-3

2.2.2.1 Starch

The major constituent of barley kernels is starch, which is present in the endosperm in the form of discrete granules (Figure 2.1) and represents, on average, 60-64% of the weight of the kernel (Table 2.1). In general, barley starch contains 75 % amylopectin and 25% amylose. However, starch from waxy barley cultivars contains 95-100% amylopectin and starch from high amylose barley cultivars may contain more than 40% amylose (MacGregor, 1993). Barley starch consists of a mixture of large, lenticular granules 15-25 μm in diameter and smaller, irregularly shaped granules $<10 \mu\text{m}$ in diameter. In the mature kernel, the starch is present exclusively in the endosperm but is not distributed uniformly. The last cells in which starch synthesis occurs during kernel development are those around the endosperm periphery. Therefore, these cells, especially those in the subaleurone region, are filled, preferentially, with protein and not starch. In comparison, more centrally located cells are filled primarily with starch and contain lower levels of protein (MacGregor and Fincher, 1993). Barley starch granules are birefringent and exhibit the characteristic “maltese cross” pattern when viewed under polarized light (Goering et al. 1973). Reported gelatinization temperatures for many bulk samples of normal starch lie in the range 56-62°C, but these values can be affected by both barley cultivar and growing conditions (Tester et al. 1991). High-amylose and waxy barley starches gelatinize at high temperatures and over a wider range of temperature than the corresponding large granules. Waxy barley starches, which contain low levels of both amylose and lipids swell to a greater extent than normal starches (Georing et al. 1973). However, high-amylose barley starches do not swell as much as normal starches (Morrison et al. 1986). It appears that the amylopectin fraction is responsible for swelling power of a given starch (Jadhav et al. 1998). Structural carbohydrates contained in barley grains-earlier considered as a waste material- may find use in industrial processing of barley for various end uses. Structural carbohydrates also regulate the digestibility of non-structural carbohydrates in barley and aid in filtration process. The hydrolyzing behavior and functional properties of starch, governed by shape and size of the granule, swelling and gelatinization pattern, amylose and amylopectin contents, molecular weight, setting and aggregation profile are equally important. This may extend the utilization potential of these barley starches in value added products (Yadav et al. 2000).

2.2.2.2 Dietary fiber

The non-starch polysaccharides and lignin are collectively designated total dietary fiber and are generally considered to be plant cell wall components. The cell wall structural polysaccharides of barley endosperm consist primarily of β -glucan, arabinoxylan and cellulose (Newman and Newman, 1991). The dominating fibers components in barley are the β -glucans and the arabinoxylans, located mainly in the cell walls of the endosperm and the aleurone layer. Most of the β -glucan is located in endosperm cell walls, but aleurone cell walls are also rich in β -glucan (Lehtonen and Aikasalo, 1987). The cell walls of barley endosperm contain about 75% β -glucan and 20% arabinoxylan, whereas the aleurone cell walls contain 26% β -glucan and 71% arabinoxylan. The minor components are cellulose, glucomannan and (1 \rightarrow 3)- β -glucans (Jadhav et al. 1998).

Total β -glucan contents of most barley grain range from 2 to 11% by weight of the grain, but they usually fall between 4 and 7% (Jadhav et al. 1998). β -Glucans form long cylindrical molecules containing up to about 250,000 glucose units. Structurally, it is (1 \rightarrow 3) (1 \rightarrow 4)- β -D-glucan with cellulose-like portions linked through (1 \rightarrow 3) glycosidic bonds, which makes it more soluble. The β bond is not digestible by enzymes in human gastrointestinal tract, resulting in the classification of β -glucan as a soluble dietary fiber (Burkus and Temelli, 2005). The β -glucans are linear molecules with around 30% β -(1 \rightarrow 3) and 70% β -(1 \rightarrow 4) linkages randomly dispersed and are associated with firmly linked peptide sequences in the barley endosperm cell wall (Fleming and Kawakami, 1977).

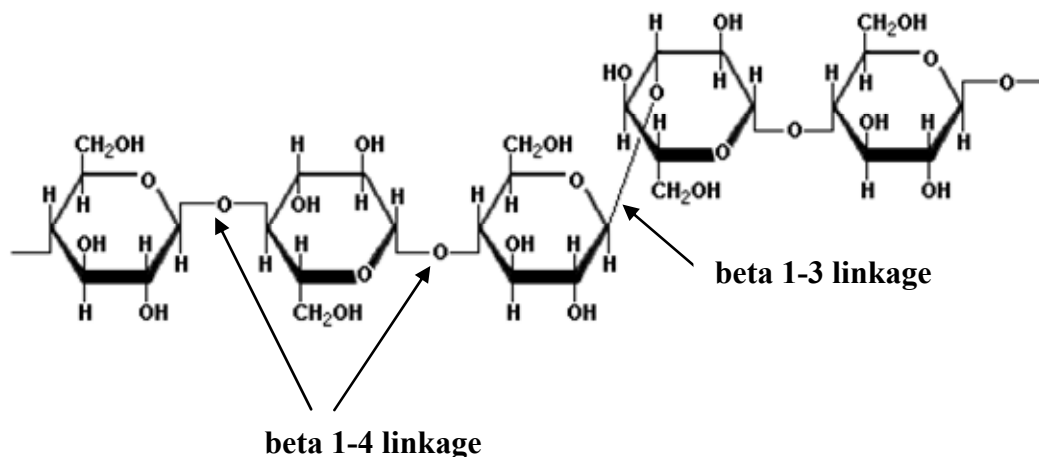


Figure 2.2 Structure of β -glucan

Arabinoxylans content in barley grain range from 4.4-7.8%. Arabinoxylans are polymers of xylose and arabinose and are present mainly in the cell walls of endosperm and aleurone in soluble and insoluble forms. They absorb many times their own weight of water and form viscous solutions in water (MacGregor, 1993). These polysaccharides consist predominantly of the pentoses, arabinose and xylose and are therefore often referred to as pentosans (MacGregor and Fincher, 1993). In human diets, pentosans are one of the components of the dietary fiber afforded by barley grain or its products (Jadhav et al. 1998). The effects of arabinoxylans on end-use quality of barley have not bran afforded the same attention that has been directed to the (1→3), (1→4)-β-glucans, presumably because arabinoxylans are less abundant in the starchy endosperm. Arabinoxylans may constitute only about 1.5% by weight of barley endosperm, but their ability to form highly viscous solutions can have a large impact on the technological utilization of barley. The arabinoxylans are more abundant in the outer grain layers; although their extraction from the highly lignified maternal tissues may not be easily affected with water (MacGregor and Fincher, 1993).

Minor cell wall components of barley are cellulose, glucomannan and (1→3)-β-glucan (Bacic and Stone, 1981). Cellulose is a (1→4)-β-glucan containing up to several thousand β-gluco-pyranosyl residues linked through (1→4) β-linkages to form very long, linear chains. In the walls of barley aleurone and starchy endosperm cells, cellulose microfibrils, which constitute only 2% of the walls, are apparently embedded in a matrix of (1→3), (1→4)-β-glucans and arabinoxylan (MacGregor and Fincher, 1993).

Only small amounts of simple sugars, glucose and fructose are present in the kernel; the major sugars are sucrose and raffinose, which are present mainly in the embryo. Varying amounts of fructosans, which are fructosyl polymers of sucrose, are also present in the mature kernel (MacGregor, 1993).

2.2.2.3 Protein

Barley typically ranges in protein content from 8 to 15% (Table 2.1). Barley contains a large number of proteins which can be separated into four major groups on the basis of solubility. Albumins and globulins (15-30% of barley protein) are soluble in

water and salt solutions, respectively; hordeins (35-50% of barley protein), the major storage proteins in barley, are soluble in aqueous alcohol solutions; glutelins (15-20% of barley protein), a mixture of storage and structural proteins, are soluble in alkali. Although aleurone cells are rich in protein, most of the barley protein is in the endosperm, especially in the subaleurone region (MacGregor, 1993).

2.2.2.4 Lipid

Barley generally contains 2-3% lipid. About 70% of barley lipid is present in the endosperm, 20% is in the embryo and the remainder is in the outer layers of kernel (MacGregor, 1993). Tocols are biologically active components found in barley and extracted with its oil. They are tocopherols and tocotrienols. Each type of tocol consists of four isomers: α , β , γ and δ . Biological effects of tocols are believed to result from their antioxidant action, specifically the inhibition of lipid peroxidation in biological membranes (Jadhav et al. 1998). Vitamin E content in barley has been determined in the range of 16.2-23.8 mg/kg (Prýma et al. 2007). Peterson and Qureshi (1993) showed that the barley samples were substantially higher in total tocols, averaging 58 mg/kg, with a range of 42-80 mg/kg in their study. It was reported that barley had substantial quantities of β - and γ -tocotrienols. The authors stated that barley, with a higher concentration of total tocols and a greater proportion of tocotrienol to tocopherol has a particularly good potential for food use. Zieliński et al. (2001) found a significant amount of tocotrienols in whole-grain of barley of 15.6 mg/kg.

2.2.2.5 Minerals and vitamins

Mineral elements exist in small concentrations in all cereal grains and are essential to the health. Minerals such as magnesium, sulphur, sodium, potassium, zinc and calcium are concentrated in the outer layers of kernel. Phosphorus is present, mainly, as phytic acid in the aleurone (MacGregor, 1993). In a separate study, the highest content of trace elements of barley has been noted for 25.11, 13.60 and 2.53 $\mu\text{g/g}$ on dry weight basis for zinc, manganese and copper, respectively (Zieliński et al. 2001). Barley is also source of B-complex vitamins, especially thiamine, pyridoxine, riboflavin and pantothenic acid (Newman and Newman, 1991).

2.2.3 The role of barley in nutrition and health

The current interest in barley is the ability to promote good consumer health through its nutritional components such as fiber, antioxidants, B-vitamins. β -Glucan has been shown to have effects in lowering blood cholesterol level and controlling blood sugar, probably mainly due to its high viscosity property as a soluble fiber to bind cholesterol and bile acids and facilitate their elimination from the body (Anderson et al. 1990; McIntosh et al. 1991; Martinez et al. 1992). Anderson et al. (1990) proposed following mechanisms, which are modification of bile acid absorption and metabolism, interference with lipid absorption and metabolism, production of short-chain fatty acids from fiber fermentation in the colon and alteration in concentrations of or sensitivity to insulin and other hormones, on how dietary fiber could lower serum cholesterol.

The hypocholesterolemic property of barley has been demonstrated in both animal models and human clinical studies. Wang et al. (1992) studied on 96 male broiler chicks fed a corn-soybean meal diet, a barley diet with β -glucanase and diet without β -glucanase. They reported that barley β -glucan caused high viscosity in chick's small intestine and thereby reduced absorption of lipids, protein and possibly other dietary nutrients, thus caused reduction in plasma cholesterol and final body weights of chicks. In another study, 21 mildly hypercholesterolemic men aged 30-59 consumed comparable barley and wheat foods for each of 4 weeks. Results showed that barley foods containing β -glucan and prepared from dietary-fiber-rich fractions of milled barley are capable of lowering plasma cholesterol in hypercholesterolemic men, relative to similar wheat foods (McIntosh et al. 1991). This conclusion agreed with that of Martinez et al. (1992), who carried out a study with 144 male broiler chicks were fed with barley and wheat diets supplemented with five fat sources. Results indicated that the high soluble fiber content of hull-less barley exerts a hypocholesterolemic effect in chicks regardless of dietary fat source, possibly mediated through lowered fat absorption. Kalra and Jood (2000) also concluded that total and soluble β -glucan were strong predictors of the cholesterol-lowering in serum and liver of rats. The viscous property of soluble β -glucan might result in reduced absorption or reabsorption of lipids. In another human study, 11 healthy men were fed low fiber and high fiber meal contained pasta (Bourdon et al. 1999). The

low fiber meal contained pasta made with wheat flour. The high fiber meals contained pasta prepared by replacing 40% of the wheat with two types of barley flour: barley naturally high in β -glucan and the other flour enriched in β -glucan during processing. Results showed that carbohydrate was absorbed slower from the high fiber meal containing β -glucan with a slower response of insulin. Consumption of the barley containing meals appeared to stimulate reverse cholesterol transport, which might contribute to the cholesterol lowering ability of barley.

α -Tocotrienol extracted from barley flour was identified as a hypocholesterolemic component for chicks. This component was shown to inhibit hydroxymethylglutaryl-CoA reductase activity in the liver, thus reducing *in vivo* cholesterol synthesis (Qureshi et al. 1986). They reported that broiler chicks fed a diet with 10 ppm α -tocotrienol for 21 days significantly decreased the concentration of serum total and low density lipoprotein cholesterol. In a separate study, 12 male broiler chicks were fed corn-based diets with either 10% barley oil, 10 % corn oil or 10% margarine for ten days. Results showed that oil extracted from waxy barley contained 558 mg/kg α -tocotrienol and 253 mg/kg polyunsaturated fatty acids. Commercial corn oil, Mazola, contained 23 mg/kg α -tocotrienol and 295 g/kg polyunsaturated fatty acids. Margarine of soy oil contained no tocotrienols and 81 g/kg polyunsaturated fatty acids. A feeding trial with male broiler chicks fed corn based diets with barley oil, corn oil or margarine showed that barley oil suppressed the elevation of plasma total and low density cholesterol concentrations compared to margarine and suppressed plasma low density cholesterol but not high density cholesterol concentrations compared to the corn oil. Both α -tocotrienol and polyunsaturated fatty acids might be responsible for the suppressive effect of barley oil on plasma cholesterol concentrations in these chicks (Wang et al. 1993a).

2.3 Extrusion Cooking

Extrusion is a continuous cooking and shaping (forming) process designed to give unique physical and chemical functionality to food materials. It is a popular unit operation for producing a variety of food products from numerous ingredients requiring a wide range of processing conditions. This technology has also many advantages such as the versatility, high productivity, low cost, unique product shapes

and high product quality (Falcone and Phillips, 1988; Gaosong and Vasanthan, 2000).

Extrusion cooking is a specialized form of processing, which is unique in food and feed processing because of the conditions that are used to transform the raw materials. It is a relatively low moisture process compared with conventional baking or dough processing. Normal moisture levels used are in the range of 10-40% on a wet weight basis. Despite these low moistures the mass of raw materials is transformed into a fluid and subjected to a number of operations to mix and transform the native ingredients into new functional forms. A second feature that distinguishes extrusion cooking from other food processes is the use of very high temperatures, usually in the range 100-180°C. The aqueous dough systems are superheated and the water vapor is contained within the extruder at high pressure. The use of high temperatures reduces the processing time and allows a full transformation of raw material to its functional form in periods as little as 30-120 s. Almost all extrusion cooking processes are operated continuously with raw materials fed into processing units. The products may be created by extrusion from dies to form the required product structure in direct extrusion, or to form the half-products in the second generation snack pellets (Guy, 2001).

Extrusion cooking allows simultaneous mixing, cooking and texturization of complex recipes at high dried-mass concentrations and viscosities. The shearing effect of screws combined with thermal energy generated by viscous dissipation quickly cooks the mixture, so that the rheological properties of raw materials are significantly modified due to physical and chemical changes of the biopolymers (Wang et al. 1990). When extrusion-cooked melts exit the die, they suddenly go from high pressure to atmospheric pressure. This pressure drop causes an extensive flash-off of internal moisture and the vapor pressure, which is nucleated to form bubbles in the molten extrudate, allows the expansion of melt (Arhaliass et al. 2003).

The application of extrusion technology is one of the most economic processes, being used increasingly in the food industries for the development of new products such as snacks, baby foods, breakfast cereals, pasta products, texturized protein food stuffs and modified starch from cereals. During extrusion cooking, the raw materials undergo many chemical and structural transformations, such as starch gelatinization,

protein denaturation, complex formation between amylose and lipids, and degradation reactions of vitamins, pigments, etc. Therefore, chemical and structural transformations in foods during extrusion cooking determine the quality of extruded products (Bhattacharya and Prakash, 1994; Yeh and Jaw, 1998; Ilo et al. 1999).

The type of ingredients (e.g. starch and protein), feed moisture content and process variables (e.g. barrel temperature, screw speed and feed rate) influence physical characteristics of extrudates. Major ingredient of starch provides most of the texture and structure of expanded products made from cereals (Bhattacharya and Hanna, 1987). Della Valle et al. (1989) stated that extrusion cooking of starch led to a degradation of amylose and amylopectin by chain splitting and macromolecular chain splitting was due to shear. In the presence of heat water affects the hydrogen bonds between the polysaccharide chains in the granule structure and the starch granules undergo gelatinization and disintegration (Iwe et al. 1999).

2.3.1 Extruders

A food extruder is a device that expedites the shaping and restructuring process for food ingredients. Extruders can be used to cook, form, mix, texturize and shape food products under conditions that favor quality retention, high productivity and low cost. The food extruder may be of single-screw or twin-screw design. The extrusion system, whether a single-screw or corotating twin-screw configuration, must accomplish a number of phenomenon in a very short time under controlled, continuous, steady state conditions. These phenomena include tempering, feeding, mixing, cooking, cooling and shaping. The pressure, temperature, moisture and resulting viscosity of the extrudate are affected by the system configuration and processing conditions (Riaz, 2000). The major difference between a single-screw extruder and a twin-screw extruder lies in the mechanism of transportation. A single-screw extruder has a screw rotating in a closely fitting barrel. It is easy to see that if the material adheres to the screw and slips at the barrel wall there will be no output. To achieve maximal output, the material must slip as freely as possible at the screw surface and adhere as much as possible to the barrel wall. In twin-screw extruders, two parallel screws are placed in a figure-eight sectioned barrel. The primary objective of this geometry is to overcome problems of slip at the wall. A single-screw extruder consists of three separate regions: a solids transport zone, in which

the material is transported in the form of a solid and in which the material is preheated up to the melting temperature; a melt zone, in which melting or transition to plasticated mass occurs; and a pump zone, in which the pressure needed to push the material through the die opening is built up (Janssen, 1989). In twin-screw extruders, the screw can be configured to enhance conveying, kneading, shearing, pressure development and filling of the screw. Twin-screw extruders have an enhanced ability to convey food materials down the screw's length. Screw fill is independent of the conveying action but is related to the resistance to flow at the die. The greater the resistance, the more product will back up and fill the preceding screw section. In single-screw extruders, the action of the screw on the food material is coupled with screw design, screw fill and temperature profiles. The shear rate within the flow volume of a twin-screw extruder is more uniform than in the channel of a single screw extruder. Consequently, the processing of individual food particles is more consistent, leading to greater homogeneity of structure and texture within the product (Harper, 1986).

Riaz (2000) reported following advantages of twin-screw extruders over single-screw extruders.

- They handle viscous, oily, sticky, or very wet materials and some other products which will slip in a single-screw extruder. (It is possible to add up to 25% fat in a twin-screw extruder.)
- They have positive pumping action and reduced pulsation at the die.
- There is less wear in smaller parts of the machine than in the single-screw extruder.
- They feature a non-pulsating feed.
- A wide range of particle size (from fine powder to grains) may be used, whereas single screw is limited to a specific range of particle size.
- Cleanup is very easy because of the self-wiping characteristics.
- The barrel head can be divided into two different steams.
- They provide for easier process scale-up from pilot plant to large scale production.
- Their process is more forgiving to inexperienced operators.

Twin-screw extruders can be classified on the basis of direction of screw rotation in the following two categories.

- Co-rotating twin-screw extruders
- Counter-rotating twin-screw extruders

These categories can be further subdivided on the basis of position of the screws in relation to one another into the following: intermeshing and non-intermeshing (Figure 2.3) (Riaz, 2000).

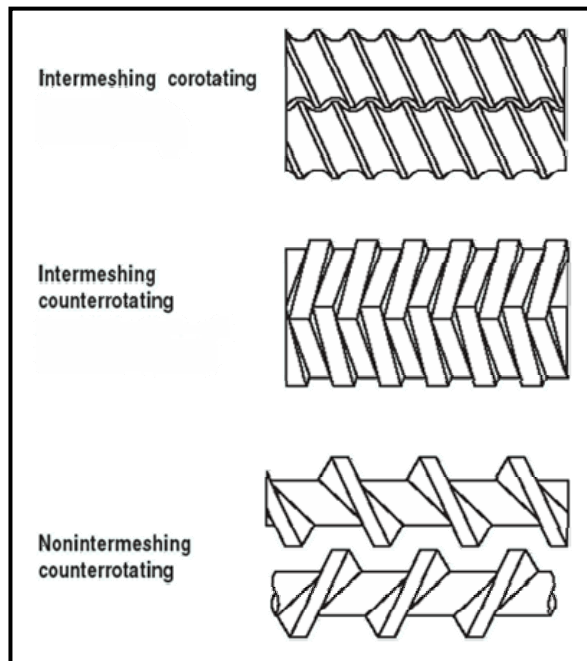


Figure 2.3 Schematic diagram of fully intermeshing co- and counter-rotating and non-intermeshing twin-screw

The twin-screw extruder consists of several subcomponents. These subcomponents and their functions include the following:

Holding/live bin: A live bin for mixed raw material capable of discharging raw premixed dry ingredients continuously and uniformly. It provides a buffer of raw material at the inlet so the extruder can operate continuously and without interruption (Huber, 2000). Typically, the height of raw material in the bin is maintained within defined limits by high and low sensors which activate a conveyor supplying the bin. The bin is designed to prevent bridging of its contents and blocking the feed screw leading to the preconditioner (Riaz, 2001). In a bin, a live or agitated bottom often is used to achieve a more uniform mass flow of material into the process flow. The live bottom prevents bridging above the bin's delivery screw or screws. A single-screw or

twin-screw conveyor can be used to deliver material from the bin into the process. For a more consistent flow of material, a certain feedstock level or range may need to be maintained in the live bottom bin. This is especially true for volumetric feeding systems and, to some extent, gravimetric or loss-in-weight feeding systems for a given formulation are needed for product consistency (Brent, 1999).

Variable speed metering/feeding device: A feeding device is used to feed the raw, dry mixed ingredients uniformly and in an uninterrupted manner at the desired flow rate. Speed of the feed screw to the conditioner or extruder must be variable to ensure a continuous uniform supply of raw material, which, in turn, leads to consistent and uniform operation of the extruder (Riaz, 2001). Two basic types of dry feeders are used to feed extruders: volumetric and gravimetric. The former assumes that the density of the feed materials does not change over time so that a constant volume of feed results in a constant mass flow rate. Further, volumetric feeders are open loop by nature, so they cannot be controlled in a feedback mode to assure a stable rate. Because of the simplicity of the volumetric feeder, it is less expensive than gravimetric variety, but it often lacks the ability to deliver the required repeatability on products other than free-flowing granular materials having very uniform particle size (Harper, 1989a). Volumetric systems may have a narrower range of levels for operation and, of course, cannot offer direct mass flow rate information for process monitoring and control. Variations in the feedstock rate may be observed at the die as a pressure change, especially on a single-screw extruder operating with a starved or incompletely filled extruder screw (Brent, 1999). Because of the tight requirements on feeder repeatability used in extrusion applications, most large-scale operations use some sort of gravimetric feeding device. The loss-in-weight feeder is gaining in popularity on large extrusion systems. This is primarily due to its simplicity, durability, ease of calibration and reduced sensitivity to flow disturbances from the feed hopper. The loss-in-weight feeder consists of a screw feeder and supply hopper on a scale. The speed of the screw is automatically adjusted to achieve the required rate of weight loss from the entire assembly. Periodically, the hopper must be refilled and the feeder goes into open-loop control mode for the short duration involved, returning to automatic mode when refilling is complete. Loss-in-weight feeders using vibrating screws and hopper bottoms have been very successful in delivering repeatable feed rates for materials that are not free-flowing or that

bridge relatively easily (Harper, 1989a). Loss-in-weight systems allow the proportioning of dry recipe ingredients and other liquid flows or additives utilized in a final product to be controlled within $\pm 0.5\%$ accuracy (Huber, 2000).

Preconditioner or preconditioning cylinder: Preconditioning cylinder in which liquids and/or steam, and/or other vapors may be uniformly combined with the premeasured dry (or pretempered) ingredient mix. The feeder screw can feed directly into the extruder inlet throat or into a preconditioning cylinder, which is used to preblend steam and water with the dry recipe. Preconditioning may be defined as a prerequisite processing step of putting a substance in the proper or desired condition. Preconditioning is a very important part of the extrusion process. The dried recipe combined with the steam and water is retained in the preconditioning cylinder long enough for each particle to achieve temperature and moisture equilibration. Mixing, hydration, cooking, pH modifications and addition of vapors, flavors, lipids, colors and meat slurries may all take place in a properly designed preconditioning process. The single most important aspect of the preconditioning system is the added mixing and retention time which is imperative for all reactions, chemical or physical (Huber, 2000).

Extruder assembly: The extruder assembly or barrel is composed of a jacketed head, a rotating extruder shaft which carries screws and shearlocks, stationary barrel housing, a die and product cut-off knife (Riaz, 2001). Jacketing the extruder heads is important to permit modification of the temperature along the length of the barrel. The heads can be heated by steam, hot water, or thermal oil. They can be cooled using water or other cooling media. The extruder bore may be of uniform diameter from inlet to discharge; it can be tapered, decreasing in bore diameter from inlet to discharge, or it can be of uniform diameter with the final segment of the barrel being tapered or decreasing in diameter. The food extruder, whether co-rotating twin screw or single-screw, must exert several actions in a very short time under controlled, continuous, steady state operating conditions. These actions include singularly or collectively any or all of the following: heating, cooling, conveying, feeding, compressing, reacting, mixing, homogenizing, melting, cooking, texturizing and shaping (Huber, 2000).

Die: Moving past the discharge end of the screw in the direction of the overall desired flow, the transformed mass of material enters the die assembly. The die assembly can be as simple as a single orifice that influences the final shape of the emerging extrudate. For greater flexibility in process design and reduced cost, a die insert can be changed with another insert to create a new extrudate shape rather than replacing the entire die assembly. Multiple orifice dies can have a number of die inserts and often are used on larger extruders for greater mass throughput (Brent, 1999). Tapered die holes will reduce back pressure requirements, create a smoother product surface and cause less mechanical damage to the extruded ingredients. Teflon coating of die inserts will also create a smoother surfaced product which has greater resistance to penetration upon biting. A die insert having an abrupt cross-sectional change short land length will cause greater mechanical damage to food ingredients and lead to a finer cell structure and softer, pithier texture. This type of insert is used to produce round extruded pieces. During the abrupt change in the dough and, when released, creates the rounded shape. Dies having a high shear rate have a potentially greater effect on product texture. High shear rates at the die cause greater shear induced damage and reduce molecular size, creating softer textured products with smaller pores, increased solubility and less mechanical strength (Harper, 1986). In terms of the extrusion process, which includes the extruder barrel, extruder screw(s) and die assembly, the die offers the last resistance to flow in the process. A common objective of many extrusion cooking operations is the creation of a pumpable mass just before the die. Raw materials enter into the extruder, are conveyed through the barrel, and are transformed eventually into a melted mass somewhere near the die. For starchy materials, the transformed mass can have a translucent appearance, often denoting a high degree of cook. For some extrusion cooking and forming operations, a melted mass can recover from the constrained region of the die upon exit and enlarge. The extrudate can emerge with no cellularity discernible to the naked eye. This phenomenon is referred to as die swell. Expanded products can result from forces attributable to die swell plus the evolution of a gaseous phase upon discharge from the die. In the latter case, the transformed extrudate takes on a shape that no longer represents the die cross-section and often exhibits a visible cellularity. Differences in the cellularity and thus the size, shape, cell wall thickness and distribution, play a role in defining final product quality (Brent, 1999).

Cutting Device: With the aid of a cutting device at or near the die face, distinct extrudate pieces can be sliced from a continuous strand of material streaming from the die or each die insert. The mass throughput, velocity distribution, cutter speed, cuts per revolution or cycle and the longitudinal expansion of expanded products combine to define the length of the extrudate pieces (Brent, 1999). During the transformation of feedstock material into a pumpable mass, material and flow properties develop and change at various, but specific, locations along the length of the rapidly rotating screw. The locations are determined usually by design and achieved by incorporating restrictive devices such as shear or kneading blocks in the screw configuration. The shear at any one location is compounded along the screw and dies configurations. Assuming no slippage of the material at the barrel wall, the resultant high rates of shear in the screw, for a given length of filled screw, may not be desirable for all processes. Excess fragmentation of large molecules such as starch and protein in the feedstock can result from the high shear forces, elevated temperatures and pressures involved in this type of extrusion. Resistance to flow often is associated with friction and an elevation in material temperature. Thus, viscous dissipation of the mechanical energy from the rotating screws accounts for some of the temperature rise in the material, thereby reducing the viscous behavior or altering the rheological properties of the melt (Brent, 1999).

The actions and sequence of occurrences in the barrel require the extrusion chambers of the co-rotating twin-screw and single-screw extruders to be subdivided into processing zones. The feeding zone, the kneading zone and final cooking zone (Figure 2.4) are the most commonly referenced (Huber, 2000). The feeding zone generally has deep channels which receive the feed. The preconditioned or dry material entering this zone is conveyed to the kneading zone. Water may be injected at this point to help develop dough and improve heat transfer in the extruder barrel. The density of material increases because of water and steam addition as the material is conveyed into the kneading zone. Screw pitch in this zone decreases and the flight angle also decreases to facilitate mixing and higher degrees of barrel fill.

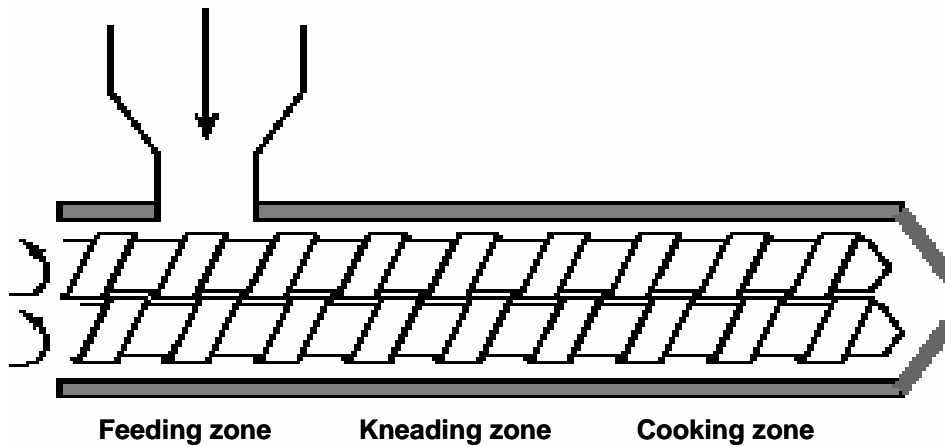


Figure 2.4 Extruder processing zones

This zone applies compression, mild shear and thermal energy to the feedstock and the extrudate begins to lose some of its granular definition and pressure develops in the barrel (Riaz, 2001). The mechanism of shear begins to play a dominant role because of the barrel fill condition. Barrel pressure is modest in the early part of the kneading zone. This permits, when desired, the injection of steam at pressures of 5-10 atmosphere. When used, this steam carries thermal energy as well as moisture into the extrudate. The discrete particles of material begin to agglomerate because of the increasing temperature resulting from conduction, direct steam injection and energy dissipation resulting from friction. As the extrudate moves through the kneading zone, it begins to form a more integral flowing dough mass and it will typically reach its maximum compaction. The shear is usually moderate and the extrudate temperature continues to increase. Amorphousing and/or texturizing occur in final cooking zone (Huber, 2000). Screw flights in this zone are typically shallow and have a short pitch. The function of this zone is to compress and pump the material in the form of a plasticized mass to the die. Temperature and pressure typically increase very rapidly in this region because of the extruder screw configuration. Shear is highest in this zone and product temperature reaches its maximum and is held for less than five seconds before the product is forced through the die. The product expands as a result of moisture vaporization as it exits through the die into a region of low pressure. The extruded material can then be cut into desired lengths by the knife attachments (Riaz, 2001).

2.3.1.1 Co-rotating twin-screw extruders

Co-rotating extruders are considered to be one of the more useful general extrusion systems available because of their flexibility, good mixing characteristics, decreased wear and high shear rates and throughput (Fichtali and van de Voort, 1989). Co-rotating machines have the screws turning in the same direction. Such machines have been the most popular for food processing because of their higher capacity. The screws on co-rotating twin-screw extruders have various sections which perform different functions in the extrusion process. Screw sections can have either rounded or square channels and still be classified as fully intermeshing and self-wiping. Self-wiping screws prevent buildup of ingredients along the shank of the screw, known as screw wrap, which can cause an interruption of conveying action and surging. Because the spacing between the parallel shafts is fixed, the flight depth is constant along the screw length and equal to the extent to which the outside radii of the screws overlap. Initial sections of the screw profile on a co-rotating twin-screw extruder are designed to convey granular ingredients into the machine (Harper, 1992). Beyond the direction of rotation or degree of intermeshing, screws can have various pitches, flight geometries and channel depths which can further affect the operating characteristic of a twin screw extruder. In general, screws come in sections that have specific functions, such as feeding, melting, mixing, pumping or shearing (Fichtali and van de Voort, 1989). They have pitch angles in the range of 15° which achieves an effective conveying angle in excess of 30° , or about three times the effective conveying angle of single-screw extruders with similar pitch. The conveying capability of twin-screw extruders allows them to handle sticky and/or otherwise difficult to convey feed ingredients. The conveying action is from one screw to the next, increasing intermixing of the materials in the channel. Unlike the single-screw extruder, much of the screw channel is not filled (Harper, 1992).

In co-rotating fully intermeshing twin-screw extruder, the flight of one screw engages or penetrates the channels of the other screw. Positive pumping action, efficient mixing and self-cleaning characteristics are offered. These features distinguish them from single screw and non-intermeshing screw extruders (Riaz, 2000). The material itself keeps the screws centered in the barrel, allowing small clearances to be used between the screws and the barrel and between the screws, which gives them a self-cleaning action. The self-cleaning action of the screws is

practical importance, as it prevents material from sticking to the screw, which could lead to the scorching of heat sensitive material. In addition, the self-cleaning action leads to a more homogeneous velocity profile, which in turn results in a narrowing of the residence time distribution. Increasing throughput and decreasing screw speed can narrow the velocity distribution considerably, allowing more uniform shear stresses to be obtained with a consequent improvement in material homogeneity (Fichtali and van de Voort, 1989).

To increase mechanical energy dissipation and enhance mixing, kneading disks are employed. Working is increased when the number of lobes on the disk increases. The relative position of the lobes on the shaft can be arranged so that their tips form a helical pattern which also imparts a conveying action. Increased mixing, heat transfer and viscous dissipation of mechanical energy input occurs in sections of the twin-screw extruder which are completely filled. To increase filled sections, some sort of restriction is placed in the screw configuration. Wear usually occurs at the restriction and preceding screw elements requiring these to be replaced most frequently to maintain the operational characteristics and efficiency of the extruder. The mixing capabilities of twin-screw extruders make them better suited as heat exchangers for viscous materials. Contact with the barrel wall is essential for efficient heat transfer requiring the heat exchange section to be completely filled with the food ingredients. Since the mixing elements in the heat transfer section and substantial mechanical energy, the twin-screw machines serve as better heaters than coolers. The pressure profile down the length of the barrel on twin-screw extruders can be varied to allow venting with partial cooling of ingredients and/or the incorporation of additional components. Reverse pitch elements cause the kneading disk section to be filled and to dissipate substantial energy. Following the reverse pitch section, the pressure decreases abruptly so venting of volatiles and flashing of moisture occurs. Subsequent sections convey, pressurize and force the dough through the die opening. The last turns of the screws are devoted to developing the necessary pressure within the dough to force it through the die. Ideally, the dough is thoroughly mixed and pressurized so that it will flow uniformly through the openings on the die (Harper, 1992).

2.3.1.2 Counter-rotating twin-screw extruders

In the counter-rotating position the extruder screw rotates in the opposite direction. These types of extruders are not widely used in the food industry although they are excellent conveyors. They are good in processing relatively non-viscous materials requiring low speeds and long residence times (Riaz, 2000). Intermeshing counter-rotating twin-screw extruders with small gaps essentially form closed channels, which minimize leakage flow between channels and reduce mixing and residence time distribution. Under these circumstances, conditions close to plug flow occur; however, localized pressure builds up at the top of the chambers where the screws converge, leading to high shear rates and stresses. These stresses can cause a wearing of the system by pressing the screws against the barrel. Since wear increases proportionally with screw speed, counter-rotating extruders are generally operated at lower speeds, resulting in lower throughputs for a given void volume. As a consequence of the general transport mechanism, a large percentage of material inside the channels is not exposed to sufficient uniform shear for good dispersion.

The benefits of this design are good pumping action, high output efficiency and narrow residence time distributions. Conversely, its limitations are poor mixing, lower throughputs, low product homogeneity and highly localized stresses that may lead to a shorter barrel life. To improve mixing and minimize the effects of high local pressures, larger clearance between the screws is an option often considered but at the expense of pumping action due to increased backflow, making the machine more sensitive to variations in head pressure. Counter-rotating extruders are used when high positive conveying action, low overall shear rates and narrow residence time distributions are required as in the case of thermally sensitive materials. They are especially useful for running low viscosity materials, slurries or rapidly solubilizing sugars and gums (Fichtali and van de Voort, 1989).

2.3.2 Parameters affecting extrusion cooking

Extrusion process parameters include raw materials, feed rate, moisture content, screw speed, barrel temperature profile, screw/die configuration. Changes in the operating parameters will cause changes in dependent process variables such as die product temperature, die pressure and viscosity as well as product quality attributes (Camire et al. 1997) through changes in specific mechanical energy and thermal

energy inputs during the product residence in the extruder (Harper, 1989b; Thymi et al. 2005). In extrusion cooking, biopolymers, mainly starches and proteins, are plasticized with water and subjected to mechanical and thermal energy treatment to achieve the desired texturization for food type end products and specific functional properties for modified starches and/or proteins (Chessari and Sellaheewa, 2001). Chessari and Sellaheewa (2001) outlined the interactions of raw material properties, process variables and product characteristics as shown in Figure 2.5.

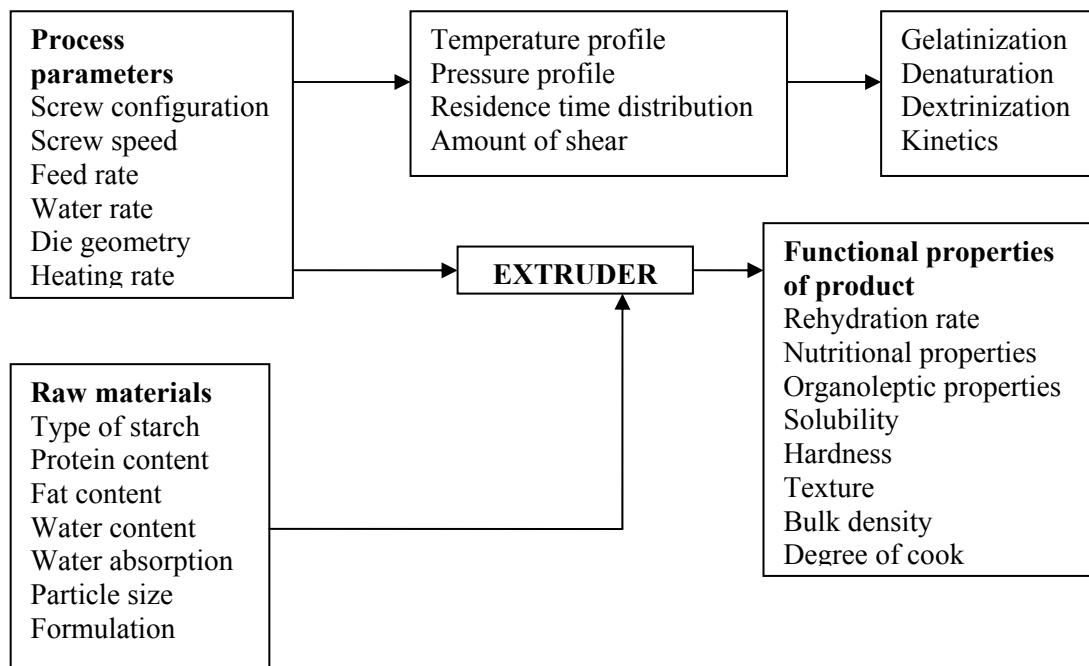


Figure 2.5 Interaction of raw material properties, process variables and product characteristics

2.3.2.1 Raw material

Extruded foods and feeds are made from a wide and diverse range of raw materials. These ingredients are similar in their general nature to the ingredients used in all other types of foods and feeds. They contain materials with different functional roles in the formation and stabilization of the extruded products and provide color, flavor and nutritional qualities found in different product types. The transformation of raw materials during processing is one of the most important factors that distinguish one food process and food type from another. For a particular product type a selection of ingredients is processed through a set processing regime. For extrusion cooking this

involves heating to high temperatures, the application of mechanical mixing and shearing, before finally extruding to form a structure (Guy, 2001).

Feedstock for an extrusion process line can include particulate solids, powdery materials, liquids and slurries. Use of quality of raw materials, proper mixing and particle size reduction practices can be advantageous to achieve process and, ultimately, product uniformity. Knowledge of the proximate composition of the raw materials often is necessary to meet nutritional composition requirements as well as make informed decisions for process adjustments. Unknown differences or alterations in the functionality of raw materials prior to extrusion can result in undesirable processing behavior and/or finished product quality. Screening or particle size reduction ensures a consistent distribution of particle sizes to the process. As with most processes, better control of the input materials allows for more successful control of the output. Moreover, accumulation of raw materials in any way of the process flow operations can lead to blockage and downtime in the extrusion process line. Typically, feedstock material contains most of the dry ingredients and possibly some minor liquid ingredients, which have been properly blended prior to placement in a feed hopper or bin (Brent, 1999).

2.3.2.2 Feed rate

Feed rates are normally kept low enough that the extruder operates under “starved-fed” conditions. Controlling the feed rate in a starve-fed extruder will affect product texture. Lowering the feed rate will reduce the fill of extruder. With only a partially filled screw, pressure flow can be proportionally more significant, causing greater circulation in the channel and potentially more mechanical damage to the food molecules (Harper, 1986). Consistency in the finished product does not depend solely on the input streams but also on the rate at which those streams are metered into the process flow. Inconsistency in the rate at which a stream is added to the process can have a dramatic effect on the product. For example, gross variation in an input stream can cause large and unacceptable changes in bulk density of the finished product (Brent, 1999).

2.3.2.3 Moisture content

Moisture is a critical catalyst in extrusion cooking processes. Moisture in the form of steam, injected into a preconditioning device and into the extruder barrel, brings with

it additional energy for cooking. This increases capacity and reduces the requirement for large drive motors. Moisture is necessary for starch gelatinization and protein denaturization. The mechanical energy required for processing decreases as moisture is increased (within limits). Moisture, in the forms of steam and water, added to a preconditioning device softens the particles of cereal grain, thus, reducing their abrasiveness. This reduces extruder component wear and, in turn, operating costs (Huber, 2000).

Moisture content during extrusion provides the driving force for expansion and also contributes to the rheological properties of the melt, which in turn affect expansion. Moisture is the main plasticizer of the cereal flours, which enables them to undergo a glass transition during the extrusion process and thus facilitates the deformation of the matrix and its expansion (Moraru and Kokini, 2003). Moisture in the barrel is the water added in relation to the feed rate that changes the raw material properties. Changes in water added cause change in specific mechanical energy, pressure and strain applied to the extrudates resulting in product differences. Water obviously plays a critical role in the expansion process. It has been found to strongly affect the degree of gelatinization as a reactant in complex interactions with other components (Holay and Harper, 1982). Expansion is a function of the amount of shear force during extrusion. Low moisture content caused high shear resulting in higher expansion (Davidson et al. 1984). Chinnaswamy and Hanna (1988a) reported that low moisture content of starch might restrict the material flow inside the extruder barrel, increasing the shear rate and residence time, which would perhaps increase the degree of starch gelatinization and, thus, expansion. However, when the moisture content of starch is too low (below 14% db), it may create very high shear rates, longer residence time, and, thus, might increase product temperature. Such conditions are known to cause starch degradation and dextrinization, which would perhaps reduce the expansion (Chinnaswamy and Hanna, 1988a).

Excess water is not available in extrusion and the starch granules do not swell and rupture, as in classical gelatinization, but are instead mechanically disrupted by high shear forces and drastic pressure changes resulting in disappearance of native starch crystallinity, plastification, expansion of the food structure, reduced paste viscosity, loss of water holding capacity, increased reconstitutability of the extrudate, softer product texture and changes in color (Kokini et al. 1992a; Onwulata et al. 1998).

2.3.2.4 Screw speed

Screw speed controls the fill during starved feeding. Extrusion screws which turn rapidly and have shallow flights and long lengths that increase residence time will cause the greatest damage or disruption of the molecules' native state (Harper, 1986). It is reported that an important variable in controlling extrusion operation parameters and product properties is the speed of the extruder screws. Screw speed and other extrusion processing parameters (e.g., barrel temperature, moisture content) determine not only the expansion ratio but also the extrudate internal structure, both of which influence the textural properties of the final product (Mezreb et al. 2003). It was found that the higher screw speed induced a decrease of sectional expansion along with an increase in longitudinal expansion, yet corn based products proved to be more sensitive to sectional expansion than wheat based products. Desrumaux et al. (1998) also reported that screw speed strongly affected extrusion response variables and extrudate characteristics. When screw speed increased, die pressure and apparent viscosity decreased, longitudinal expansion index (LEI) increased, while sectional expansion index (SEI) and volumetric expansion index decreased. It has been reported that screw speed has generally a positive effect on extrudate expansion due to the increase in shear, and thus decrease in melt viscosity induced by high screw speeds (Kokini et al. 1992b).

Della Valle et al. (1987) observed no clear influence of screw speed on product temperature and die pressure. Jin et al. (1994) reported that an increase in the screw speed raised the product temperature and specific energy but reduced die pressure. Ryu and Ng (2001) also found that lower screw speed generated greater die pressure than higher screw speed, but the reverse was observed for specific mechanical energy (SME) input. SME is the amount of mechanical energy dissipated as heat inside the material, expressed per unit mass of material. The SME input is a good quantitative descriptor in extrusion processes, since it allows the direct comparison of different combinations of extrusion conditions such as screw speed, feeding rate and torque. The amount of mechanical energy delivered to the extruded material determines the extent of macromolecular transformations and interactions that take place; that is, starch conversion and, consequently, the rheological properties of the melt. Increased SME leads to lower viscosity, which promotes mobility and thus may lead to an increase in the rate of bubble growth (Moraru and Kokini, 2003).

SME is strongly dependent on the process conditions such as screw speed, barrel temperature, moisture content, feed composition and screw configuration. All of these affect the viscosity of the material and the flow field inside the screw channels. SME increases with increasing viscosity, increasing screw speed and decreasing mass flow rate. Both the extrusion operation and the screw performance can be well characterized by the SME value (Godavarti and Karwe, 1997).

2.3.2.5 Barrel temperature profile

Extrusion temperature plays an important role in changing the rheological properties of the extruded melts, which in turn affect the expansion volume (Moraru and Kokini, 2003). Preset melt temperature affects melt viscosity. Preset low temperature creates a more viscous flow, which creates more shear and higher melt temperature measured at the extruder die. Preset high temperature results in a less viscous flow and reduced melt temperature at the die (Onwulata et al. 2000).

At high temperatures the pressure of saturated vapors exceeds the melt pressure towards the die exit, favoring bubble growth inside the die in the direction of flow and thus longitudinal expansion. At low temperatures, bubble growth starts at the die outlet since the pressure of saturated vapors is lower than the melt pressure in the die (Della Valle et al. 1997). An increase in barrel temperature decreases the melt viscosity. The reduced viscosity effect would favor the bubble growth during extrusion. Moreover, the degree of superheating of water in the extruder would increase at higher temperatures, also leading to greater expansion (Ding et al., 2005).

2.3.2.6 Screw/die configuration

Screw configuration, speed and the addition of reverse screw elements, increase shear and control energy distribution responses such as melt temperature, torque and pressure; and the melt temperature then determines the degree of puffing (Sokhey et al., 1994). The extruder die plays an important role in the extrusion process. The extruder die and the forming jig determine the dimensions of the semi-finished products, but the important points to be taken into consideration are the rheological and thermodynamic processes in the die and in the forming, as well as in the stretching processes present between die and forming, which have decisive effects on the quality of the semi-finished products (Sokhey et al. 1997). Die effects can be

considered independently from the type of extruder used to form the dough. Die shape influences finished piece shape and texture. Tapered die holes will reduce back-pressure requirements, create a smoother product surface, and cause less mechanical damage to the extruded ingredients. A die insert having an abrupt cross-sectional change and short land length will cause greater mechanical damage to food ingredients and lead to finer cell structure and softer, pithier texture (Harper, 1986). A die with small diameter and shorter length would be used for greater radial expansion and minimizing energy consumption (Sokhey et al. 1997).

It is shown that screw configuration is a potent variable in determining product properties. The variation in water solubility with specific mechanical energy input conforms to Meuser's model. In this model, the water solubility index is found to be simply related to the specific mechanical energy input and the temperature of the dough at the dies of the extruder. The screw configuration controls the specific mechanical energy input range. Screw configurations of low conveying efficiency give rise to low-viscosity melts which expand into low bulk density products (Kirby et al. 1988).

2.3.3 Evaluation of extrudates

Physical, functional, textural and sensory properties are important parameters for evaluating the quality of produced extrudates.

2.3.3.1 Physical properties

a) Expansion index

Expansion is an important characteristic of extruded products being developed as snack and ready-to-eat products by food industries (Baladran-Quintana et al. 1998). Expansion index describes the degree of puffing undergone by the sample as it exits the extruder. Expansion phenomena are dependent on the viscous and elastic properties of melted dough (Asare et al. 2004). The expansion and texture formation of extrudates is complex even for products based on a single component. Viscoelastic properties of the melt, mechanism of bubble nucleation and growth, as well as plastisizing properties of water in the transition from fluid (melt) to viscoelastic and subsequently to glassy state, are all important for expansion and final texture of extrudates. Multicomponent and multiphase structure of the melt would modify each

of these processes, ultimately affecting expansion and texture of products (Zasyarkin and Lee, 1998).

Increased feed moisture content during extrusion may reduce the elasticity of the dough through plasticization of the melt (Ding et al. 2006) and decrease the dough temperature because moisture would reduce friction between the dough and the screw/barrel, and have a negative impact on the starch gelatinization, thereby reducing the product's expansion (Asare et al. 2004). Expansion is a function of the amount of shear force during extrusion. Low moisture content caused high shear resulting in higher expansion (Davidson et al. 1984).

The extrudate expansion follows two different directions that are sectional (radial) and longitudinal expansion. The measurement of sectional and longitudinal expansions indices allows a better description of the cell structure of expanded extrudates (Alvarez-Martinez et al. 1988). Moisture plays a key role in the mechanism responsible for radial expansion. Considering the elastic properties of the amylopectin network as being responsible for diametral expansion is helpful in explaining such a role for moisture. Increased water content in the melt would soften the amylopectin molecular structure and reduce its elastic characteristics to decrease diametral expansion (Alvarez-Martinez et al. 1988).

b) Bulk density

Bulk density is a very important product quality attribute from the viewpoint of commercial production of extruded products because most extruded products are filled by weight and not by volume. Therefore, if the bulk density varies during production, either the pack will not be full or it will overflow. As both these scenarios have serious production implications, in addition to the moisture content, bulk density is a quality attribute that is measured regularly for quality assurance purposes. In addition to controlling the correct volume of product in the pack, if the bulk density is controlled properly, generally the product texture will be within the required quality limits. This is because there is a relationship between the bulk density and the texture because both these parameters are controlled by the degree of expansion. The die design also has an effect on the bulk density (Chessari and Sellahewa, 2001).

Product bulk density is directly related to the extent of extrudate expansion and is a very important parameter in the production of expanded and formed food products (Köksel et al. 2003). At high moisture levels, the bulk density is also high. This is because the extrusion cooking is not enough to cause vaporization of the moisture, leading to retention of moisture and hence the reduced puffing of the product. As a result a denser product is obtained (Asare et al. 2004; Baik et al. 2004).

An increase in the barrel temperature increases the degree of superheating of water in the extruder encouraging bubble formation and also a decrease in melt viscosity leading to reduced density (Ding et al. 2006). Bulk density and expansion are inversely related and a high bulk density is associated with a low expansion (Rayas-Duarte et al. 1998; Suknark et al. 1998) because a more compact material is obtained after milling a less expanded product (Onyango et al. 2004a).

c) Color

Color is an important visual quality of food products. There are many reactions that take place during extrusion cooking that affect color. Among them, the most common are nonenzymatic browning reactions (Maillard reaction, sugar caramelisation, etc.) and pigment degradation (Ilo and Berghofer, 1999). The processing conditions used in extrusion cooking (high temperature and low water content) are known to favor the Maillard condensation of amino groups with reducing sugars which leads to the formation of color compounds and a reduction in available lysine (Berset, 1989). Nonenzymatic browning is associated with a flavor development. If browning is too intensive, undesirable colors and flavors may appear. Also, the changes of color in extrusion cooking may be a visual indicator to assess the process intensity concerning chemical changes or nutritional loss in food (Ilo and Berghofer, 1999).

Extrusion cooking changes the nature of many food constituents, including starches and proteins, by altering their physical, chemical and nutritional properties (Camire, 2000). The resulting changes in conformation, together with the partial degradation of starch and protein, can result in increases in the availability of reactive groups which can go on to take part in reactions, including the Maillard reaction (Sgaramella and Ames, 1993). With high temperature, high pressure, and low moisture content of

the feed, extrusion cooking often results in a colored product even though the residence time is low (Lei et al. 2007).

Several authors have studied color development during extrusion cooking. Sgaramella and Ames (1993) studied on the development of color, formed as a result of the Maillard reaction, in starch-glucose-lysine mixtures of varying moisture content and processed by extrusion cooking. They concluded that extrusion cooking starch alone always resulted in a white or off-white extrudate. On the other hand, all the extrudates prepared from the starch-glucose-lysine mixtures showed the development of definite color (ranging from pale cream to golden brown), which varied in intensity according to the die temperature, moisture content and amounts of glucose and lysine used. In general, increases in die temperature and amounts of glucose and lysine and decreases in moisture content, each resulted in increased color development. Apruzzese et al. (2000) demonstrated the feasibility of using a fiber optic equipped visible near infrared spectrometer to monitor both color and composition in an extruder during the extrusion of yellow corn flour. Series of experiments were conducted with and without adding any coloring in order to study changes in color and composition during the extrusion process. Their results showed that difference in food dye concentrations can be detected in-line. The results also showed that at higher screw speeds lightness increases, indicating that the sample is lighter. Increased temperature at constant screw speed provided a darker product. The spectrum shows a maximum around 700 nm typical of the red and orange color, indicating the browning of the sample. Lei et al. (2007) developed image analysis method to determine the color change of rice-glucose-lysine blend during extrusion. They concluded that the image analysis method developed provides an objective and efficient approach for assessing the color development of extrudates.

2.3.3.2 Functional properties

a) Water absorption index

Water absorption index (WAI) measures the volume occupied mainly by the starch after swelling in excess water, which maintains the integrity of starch in aqueous dispersion (Mason and Hosoney, 1986). WAI can be used as an index of gelatinization (Sacchetti et al. 2004; Ding et al. 2005; Ding et al. 2006). Water absorption has been generally attributed to the dispersion of starch in excess water,

and the dispersion is increased by the degree of starch damage due to gelatinization and extrusion-induced fragmentation, that is, molecular weight reduction of amylose and amylopectin molecules (Rayas-Duarte et al. 1998).

b) Water solubility index

The water solubility index (WSI) expresses the percentage of dry matter recovered after the supernatant is evaporated from the water absorption determination (Anderson et al. 1969). WSI is related to the quantity of soluble molecules which is related to dextrinization. The water solubility of starch increases with expansion (Colonna et al. 1989). WSI, often used as an indicator of degradation of molecular components (Kirby et al. 1988), measures the degree of starch conversion during extrusion which is the amount of soluble polysaccharide released from the starch component after extrusion (Ding et al. 2005). Mezreb et al. (2003) stated that the water solubility index is indeed related to the degree of starch transformation. The increase of screw speed induced a sharp increase of specific mechanical energy. The high mechanical shear degraded macromolecules, so the molecular weight of starch granules decreased. Consequently, the water solubility index increased because starch granules were then more soluble in water.

It has been reported that raising the barrel temperature would increase the amount of gelatinized starch that could increase the amount of soluble starch (Ding et al. 2005), resulting in an increase in water solubility index (Yuliani et al. 2006). Increasing barrel temperature in the presence of shear would also induce starch fragmentation leading to an increase in solubility of extruded starch (Colonna et al. 1989; Yuliani et al. 2006).

2.3.3.3 Textural properties of extrudates

Texture is one of the most important sensory attributes of extruded products. The rapid flashing of water forms the characteristic texture of extruded products when the starch melt comes out of the extruder die. Water changes from liquid to vapor as the pressure is suddenly reduced from a high pressure in the extruder to atmospheric pressure. As bubbles of water vapor come out of the starch melt, the product stretches and the matrix sets because of evaporative cooling. Air bubbles get trapped in this matrix and the characteristic puffed structure is formed. The texture of

extruded products is dependent on the cell size distribution and cell wall thickness. The rheology of the melt has a significant effect on the expansion mechanism and therefore on the final product texture. The rheological properties of the melt are controlled by the formulation, temperature profile, barrel moisture content, screw speed and screw profile (which affect shear forces in the extruder) and the die design. The interaction of carbohydrates, proteins and lipids has an effect on the melt rheology and product texture (Chessari and Sellahewa, 2001).

Texture refers to the human sensation of food derived from its rheological behavior during mastication and swallowing. The successful development of food products requires both comprehensive understanding of texture as perceived by the consumer and appropriate measurement methods. The hardness and crispness can be used to evaluate textural properties of extrudates. The hardness and crispness of expanded extrudate is a perception of the human being and is associated with the expansion and cell structure of product (Ding et al. 2005; Ding et al. 2006). Maximum peak force obtained from texture analyzer gives the hardness of product. The peak force represents the resistance of extrudate to initial penetration whereas the area under the curve is represented as crispness of product (Ding et al. 2005, Ding et al. 2006). Rayas-Duarte et al. (1998) reported that high breaking strength values are generally related to large cells with thicker cell walls, creating a crunchy texture. Low breaking strength values are usually related to a large number of small cells per unit area with thinner cell walls, resulting in a crispy texture.

The effect of extrusion conditions on hardness of extrudates has been studied by many authors. It was found that feed moisture and barrel temperature have the most significant effect on extrudate hardness. An increase in feed moisture caused an increase in the hardness of the extrudates. This result agrees well with a decrease in expansion observed. However, an increase in temperature resulted in a decrease in hardness (Liu et al. 2000; Ding et al. 2005; Ding et al. 2006). In some studies breaking strength was used to evaluate final texture of product (Ilo et al. 1999; Onwulata et al. 2001a). Reduced breaking strength indicates reduced hardness, an improvement in extrudate quality (Onwulata et al. 2001a). An increase in feed moisture also causes a reduction in barrel temperature thereby creating a lower vapor pressure in the melt and resulting in less flashing of moisture and a reduced sectional expansion index (Badrie and Mellows, 1991). The trapped steam instead condenses

in the extrudate on cooling and is responsible for the higher extrudate moisture content. Extrudates with higher moisture contents were compacter and harder after cooling than those with lower moisture contents. This textural difference is because the partially expanded structure of the extrudate processed at high moisture content collapses and solidifies more rapidly during cooling and drying and forms a hard gritty texture (Onyango et al. 2004a).

2.3.3.4 Sensory evaluation

When processed in a cooker-extruder, food materials are subjected to high temperatures and pressures in combination with shearing stresses. As a result the material undergoes physical and chemical modifications such as gelatinization and breakdown of starch, denaturation of proteins and interactions between their products. These changes influence the appearance, aroma, flavor and texture of the extruded products (Chen et al. 1991). Chen et al. (1991) studied the effects of extrusion parameters on the sensory properties of corn meal extrudates. They used the descriptive sensory analysis to characterize appearance, aroma, flavor, and texture of extrudates. It was concluded that temperature was the most significant factor affecting airiness, toasted corn aroma and flavor, denseness, crispiness, chewiness and hardness of extrudates.

Trained descriptive panels are an inexpensive way to taste competitor products and determine if changes are occurring with their formulations or processing conditions. As soon as changes occur, panelists can alert product scientists, allowing more time to respond to market shifts. Trained descriptive panels are used to describe the complete sensory profile of target products and progress in developing experimental products. The language developed in this process is also used in follow up consumer panels. Product differences that can be related to differences in analytical tests can be determined and correlations developed with human perception, thus validating analytical methods and reducing the costs of human panels. Hedonic scales are extensively used in consumer sensory analysis. They focus on pleasant and unpleasant features of the food tested, measure like-dislike and are used for overall, texture and flavor acceptability. Hedonic scale is easily used with non-trained panelists (Jacoby and King, 2001).

Both descriptive sensory analysis and hedonic scale have been used to evaluate sensory properties of extruded food produced with different raw materials. Camire et al. (2002) studied to evaluate the stability and acceptability of blueberry and grape anthocyanins in extruded cereals. They used a 9-point hedonic scale for overall acceptability and acceptability of color, sweetness, hardness and flavor. In their result the addition of fruit juice concentrate significantly affected overall acceptability and acceptability of color, sweetness and flavor but not hardness acceptability. Rampersad et al. (2003) investigated the effects of adding pigeonpea flour to cassava flour on the physico-chemical and sensory quality of enrobed flavored extrudates. In this research, trained panelists rated the texture of unflavored cassava/pigeonpea extrudates to determine the most suitable textured product in the first stage. In the second stage, attributes of color, odor, flavor, texture and overall acceptability of enrobed flavored products have been evaluated by hedonic testing using consumer panelists. Authors found that the effect of pigeon pea addition to cassava flour had significant influence on sensory texture of extrudates. All enrobed flavored extrudates were liked moderately to very much in overall acceptability, with chocolate extrudates having the highest scores for color and flavor. In another research, Liu et al. (2000) studied the effect of screw speed, moisture content and different percentages of oat flour on the physical and sensory properties of oat-corn puffs and to correlate the instrumental and sensory data. Descriptive sensory analysis was used to evaluate flavor, texture, aftertaste and appearance of oat-corn puff products. It was concluded that decreasing moisture content and increasing screw speed resulted in increased product temperature, which was highly correlated with attributes of a more expanded product such as lightness, crispness, shininess and an open cell structure. With a higher screw speed and a higher product temperature, corn-related flavors were more likely to develop. They observed high correlations between physical and sensory properties. Ahmed (1999) studied the effect of incorporating increasing percentage of flaxseed flour on the chemical, physical, microstructure and sensory quality of corn based snack. Hedonic scale was used for the product's sensory attributes of appearance, aroma, taste, texture and color. Sensory evaluation showed that total score gradually decreased by increasing proportion of added flaxseed but still acceptable for the panelists. Veronica et al. (2006) fortified a maize-based snack with partially defatted soybean and analyzed for physical, chemical and sensory characteristics. The extruded samples were subjected

to two different sensory evaluations. A 9-point hedonic scale was used to determine the preference in color, flavor, taste and overall acceptability, while a 5-point “just-about-right” scale was used to assess sensory crispness and puffiness of the products. The results showed that incorporating partially defatted soybean in a maize-based snack had a positive effect on chemical properties but had a negative effect on the physical and sensory characteristics.

2.3.4 Summary of recent extrusion studies

Extrusion cooking is a popular food processing technique, especially for the production of fiber rich products such as snack foods, breakfast cereals. Snack foods have become component of the eating habits of the majority of the world’s population. Basically, they are prepared from natural ingredients or components according to pre-designed plans to yield products with specified functional properties. These products are shaped and expanded at the extrusion die and generally require no further processing except for some minimal drying. Most products are extruded through round holes where the speed of the cutter determines whether it is a ball, rod, or curl. The recent applications of extrusion cooking in literature have been summarized as follows:

Liu et al. (2000) studied the effect of screw speed, moisture content and four different percentages of oat flour on the extrudate physical and sensory properties. They concluded that an expanded product that can be used as a cereal or snack food could be made with oat flour through blending with corn flour. It was found that both oat flour percentage and feed moisture affected its physical and sensory properties while the effect of screw speed was not significant. Sebio and Chang (2000) investigated extrusion of yam flour and effects of process parameters on physicochemical properties of the extrudates. They observed increasing expansion and decreasing density and hardness with increasing barrel temperature and decreasing feed moisture content. The other study related with extrusion is incorporation of whey products in extruded corn, potato or rice snacks. Whey products including sweet whey solids or whey protein concentrate were added with known concentration to corn meal, rice or potato flour to improve the nutrient density of snack products under conditions included low shear, high shear and combination of high shear/low moisture. It was indicated that an acceptable snack

that incorporates whey proteins such as whey protein concentrate or sweet whey solids could be produced through the extrudates might be significantly expanded (Onwulata et al. 2001a). Ryu and Ng (2001) determined the effect of processing variables, such as water injection rate, barrel temperature and screw speed on the expansion and mechanical properties of wheat flour and whole cornmeal extrudates. They resulted that the cross-sectional expansion index of wheat flour and cornmeal extrudates increased with a decrease in water injection rate and/or an increase in barrel temperature. A drop in the elastic modulus and breaking strength in bending with the decrease in water injection rate was also observed by authors. They concluded that the texture of extruded products could be controlled by changing the SME input as well as process variables.

Köksel et al. (2003) investigated processing of durum wheat in a co-rotating twin-screw extruder with different levels of moisture content of the feed, screw speed and feed rate to develop bulgur-like product. They reported that overall acceptable quality characteristics of produced a new bulgur-like product were comparable to those of commercial bulgur. Therefore, extrusion seems to be promising for the production of dry, relatively inexpensive bulgur-like products with acceptable sensory properties. Hashimoto and Grossmann (2003) used cassava bran as a fiber source for cassava starch extrudates and determined the effect of extrusion conditions on some physicochemical properties of extrudates. They indicated that cassava starch blended with different levels of cassava bran could be used as raw material for extruded snack production, if the appropriate operational conditions were applied. Sacchetti et al. (2004) investigated effects of changing of extrusion temperature and feed composition on the functional, physical and sensory properties of chestnut and rice flour-based snack-like products. They found that chestnut flour was suitable for the extrusion-cooking process adopted if properly mixed with rice flour, with 30% chestnut flour processed at 120°C produced a snack-like with limited density and browning that was judged good by a sensory panel. Asare et al. (2004) studied the product characteristics of extruded rice-cowpea-groundnut blends. They found that well-expanded rice-legume blend extrudates of less bulk density and lower moisture content could be produced at low feed moisture content (<20%). In addition to this, the optimal process variables for the production of puffed snack with enhanced product quality characteristics were determined as low feed moisture of 14-20.09%

and maximum additions of 20% cowpea and 10% groundnut. The effect of extrusion conditions on the physicochemical properties and sensory characteristics of rice-based expanded snacks were studied by Ding et al. (2005). They found that feed rate, feed moisture and barrel temperature had significant effect on various extrudate properties, with feed moisture having the greatest influence on the properties of the extrudate.

Thymi et al. (2005) investigated the effect of extrusion conditions (temperature, feed moisture content, residence time and rotation speed) on the structural properties of extruded corn starch. It was reported that extruded product apparent density, porosity and expansion ratio were found to be dependent on feed moisture content, residence time and temperature while they were not affected by screw speed. In another research, the effect of pectin alone or in combination with wheat fiber on the physical and structural properties of extruded cornstarch, under specific moisture content, barrel temperature and screw speed combinations were studied using a laboratory single screw extruder (Yanniotis et al. 2007). Macroscopic and microscopic examination showed that extrudates containing pectin did not show any apparent difference in the size and the number of the cells compared to extrudates from corn starch without pectin or fiber, while in extrudates with wheat fiber the cell size was smaller and the number of cells higher. Pectin reduced radial expansion and hardness of extruded cornstarch and increased porosity. Fibers interfere with bubble expansion and reduced expansion, cell size and porosity. Fibers also reduced moisture content and increased hardness. Camire et al. (2007) examined the effects of dehydrated fruit powders as colorants and antioxidants in extruded white cornmeal breakfast cereals. They concluded that although anthocyanins from fruit powders survived extrusion and retained some antioxidant activity, the levels used in their study were too low. Higher levels of fruit powder would increase production costs, but the expense might be offset by the more attractive and functional cereals that result.

CHAPTER 3

MATERIALS AND METHODS

3.1 Materials

3.1.1 Raw materials

Barley flour, from hull-less whole barley grain, was obtained from Bob's Red Mill Natural Foods (Milwaukie, OR, USA). The particle size distribution of the barley flour was 12.1% (on mesh 40; 0.420 mm); 42.9% (on mesh 60; 0.250 mm); 38.9% (on mesh 80; 0.177 mm); 5.5% (on mesh 100; 0.149 mm); 0.4% (on mesh 120; 0.125 mm) and 0.2% (mesh 120). Barley flour was stored at 4°C until used.

Tomato pomace, tomato-processing by-product, was obtained from the ConAgra Foods tomato processing plant located in Oakdale (California, USA). The pomace, obtained from the paste line, had a moisture content of 46.4% (wb). It was dried at 50°C overnight in a forced-air drier (Model # R-4, Commercial Dehydrator System, Inc., Eugene, OR, USA). The dried tomato pomace was coarsely ground and passed on sieve with mesh size of 20. Then, the sieved tomato pomace was finely ground and stored in polyethylene bags at -20°C for further usage. The moisture content of dried tomato pomace was 2.43±0.2% (wb).

Thompson seedless grape pomace was provided by Department of Viticulture and Enology, UC Davis pilot plant. Grapes were conveyed into stemmer and crusher by screw conveyor. They were pressed immediately after crushing. The resulting juice was separated from pomace and processed for wine making. Grape pomace was frozen as sheet in a blast freezer (Model FB27-5-5 ST S, Conrad, Michigan, USA) at -63.9°C for freeze drying. The pomace was freeze-dried (Model 50-SRC-5, Virtis, Gardiner, NY, USA) at 8 Pa pressure for 72 h. The condenser temperature was approximately -40°C. Freeze-dried grape pomace was finely ground and stored in polyethylene bags at -20°C for further usage. The moisture content of dried grape pomace was between 3.9 and 6.3% (wb).

3.1.2 Chemicals

Folin-Ciocalteu reagent (Merck KGaA, Darmstadt, Germany) and ferulic acid (Merck Schuchardt OHG, Hohenbrunn, Germany) were supplied from Merck. 2,2-diphenyl-1-picrylhydrazyl (DPPH) and porcine pancreatic α -amylase (No A-6255) were obtained from Sigma (Sigma-Aldrich, Inc., St. Louis, MO, USA).

3.2 Sample Preparation

3.2.1 Barley flour

The samples were conditioned to $21.97\pm 0.48\%$ (wb) moisture by spraying with a calculated amount of water and mixing continuously at medium speed in a mixer (Mod. F-30T, Blakeslee, Chicago, ILL, USA). The level of moisture content was chosen according to preliminary tests and to ensure smooth operation of the laboratory-scale extruder. The samples were put in buckets and stored at 4°C overnight. The feed material was then allowed 3 h to equilibrate at room temperature prior to extrusion. This preconditioning procedure was employed to ensure uniform mixing and hydration and to minimize variability in the state of the feed material. Moisture content of samples was determined by halogen moisture analyzer (Model HR83 and HR83P, Mettler-Toledo GmbH, Greifensee, Switzerland) at 105°C .

3.2.2 Barley flour-tomato pomace and barley flour-grape pomace blends

Blends were prepared by mixing barley flour and tomato pomace and grape pomace in the ratios of 100:0, 98:2, 94:6, 90:10 and 87.3:12.7 on a dry-to-dry weight basis. The blended samples were conditioned to $21.84\pm 0.26\%$ (wb) and $21.66\pm 0.49\%$ (wb) moisture for barley flour-tomato pomace and barley flour-grape pomace by spraying with a calculated amount of water and mixing continuously at medium speed in a mixer (Model F-30T, Blakeslee, Chicago, ILL, USA). The samples were put in buckets and stored at 4°C overnight. The feed material was then allowed 3 h to equilibrate at room temperature prior to extrusion. This preconditioning procedure was employed to ensure uniform mixing and hydration and to minimize variability in the state of the feed material. Moisture content of samples was determined by halogen moisture analyzer (Model HR83 and HR83P, Mettler-Toledo GmbH, Greifensee, Switzerland) at 105°C .

3.3 Extrusion Cooking

A laboratory-scale co-rotating twin-screw extruder (Figure 3.1a) (MPC/V-30, APV, Staffordshire, England) with a System9000 torque rheometer (Haake Buchler, Paramus, NJ) that provided computer control and data acquisition was used. The slit die (Haake Buchler, Paramus, NJ, USA) had dimensions of 1.47 mm x 20 mm x 150 mm. The barrel diameter and its length to diameter ratio (L/D) were 30 mm and 13:1, respectively. The MPC/V-30 had a clamshell barrel consisting of three independent zones and transition section temperatures controlled by electrical heating and compressed air cooling. A computerized control and data acquisition system were used to control five set temperatures and rotor speed and to record five melt temperatures, pressure at the slit die and torque data. Data acquisition rate was every six seconds. The barrel zone temperatures were set constant at 30, 60, 100; temperature of the transition section between the barrel and the die was set at 130 °C throughout the experiments. The die temperature was changed according to the experimental design. The actual extruder screw speed was 2.5 times the rotor speed. The screws were composed of screw elements and lope-shaped paddles which could be assembled on the hexagon-shaped shafts to give different screw configurations. The screw configuration used is shown in Figure 3.1. The screw configuration had three pieces of 1.5 D twin lead feed screws, two 1 D twin lead feed screws, nine kneading elements oriented at 30° feed forward, one 1 D single lead feed screw followed by nine kneading elements oriented at 30° feed forward and 1 D discharge screw.

Barley flour and pomace blends were fed into extruder with a K-tron Type T-20 twin-screw volumetric feeder (K-Tron Corp., Pitman, NJ, USA) at a rate of 2.11 ± 0.042 kg/h. Extrudate was collected when the operation condition was at steady state identified by torque value that varied less than 5%. The samples were dried at 52 °C overnight in a forced-air drier (Model # R-4, Commercial Dehydrator System, Inc., Eugene, OR, USA). The final dried samples contained a maximum of 4.7%, 5.5% and 5.7% (wb) moisture for barley flour, barley flour-tomato pomace and barley flour-grape pomace extrudates, respectively. Dried samples were stored in polyethylene bags at room temperature and used for further analysis.

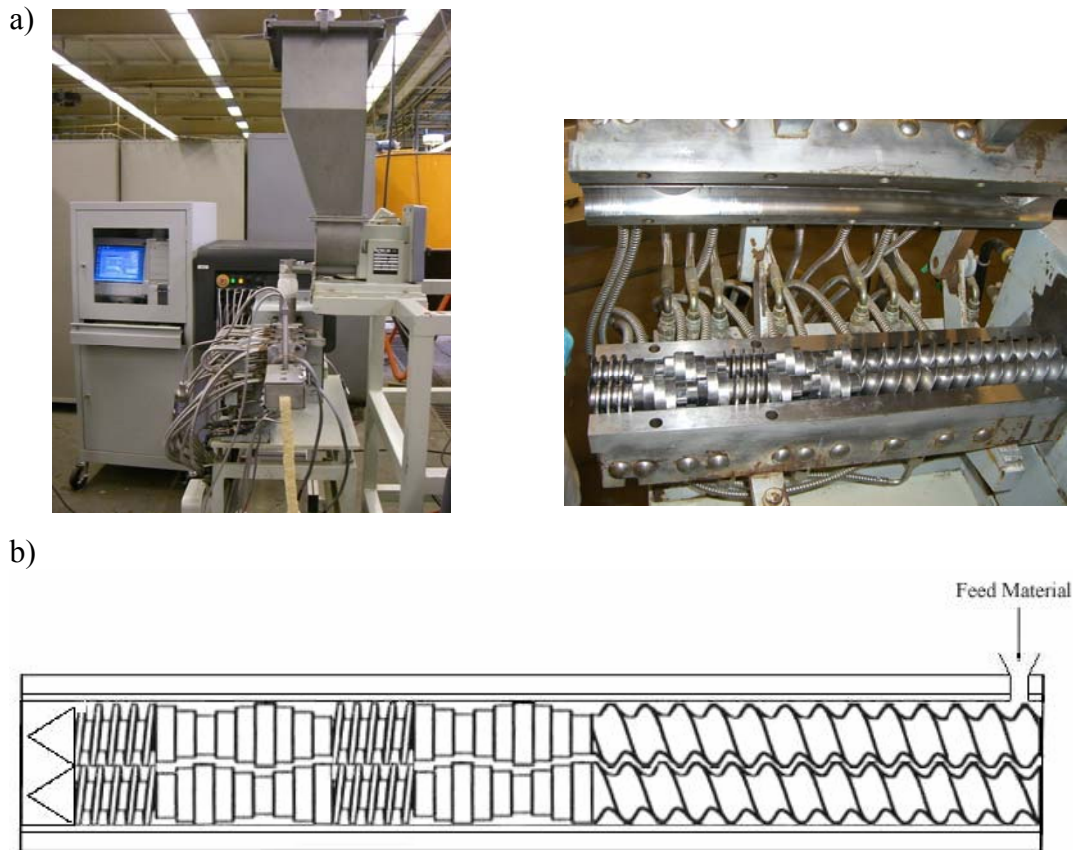


Figure 3.1 Photographs of extruder and screws (a) and schematic representation of screw configuration (b)

3.4 Experimental Design

The central composite design for two independent variables was performed for barley flour and three independent variables for barley flour-tomato pomace and barley flour-grape pomace. The independent variables considered were die temperature (X_1) and screw speed (X_2) for barley flour but die temperature (X_1), screw speed (X_2) and pomace level (X_3) for barley flour-tomato pomace and barley flour-grape pomace. The independent variables and variation levels are shown in Tables 3.1 and 3.2. Experimental design yielded a negative pomace level (-0.7%) for the coded value of -1.682. But it was taken as zero since negative pomace level is physically unsound. The levels of each variable were established according to literature data and preliminary trials. The outline of experimental design with the coded and actual levels is presented in Tables 3.3 and 3.4. Dependent variables were specific mechanical energy (SME), die pressure (DP), die melt temperature as system parameters and sectional expansion index (SEI), bulk density, water absorption and solubility indices (WAI and WSI), texture, color, antioxidant activity (AA), total phenolic content (TP), degree of starch gelatinization (DG), starch digestibility (SD)

and β -glucan content (BG) as product responses. Response surface methodology was applied for experimental data using a commercial statistical package, Design-Expert version 6.0.6 (Statease Inc., Minneapolis, MN, USA) for the generation of response surface plots.

Table 3.1 Process variables used in the central composite design for two independent variables for barley flour

	Variable level codes					
	Code	-1.414	-1	0	1	1.414
Die temperature($^{\circ}$ C)	X ₁	136	140	150	160	164
Screw speed (rpm)	X ₂	140	150	175	200	210

The same software was used for statistical analysis of experimental data. The results were analyzed by a multiple linear regression method which describes the effects of variables in the first order, a two-factor interaction (2FI) and second order polynomial models. The selected models were fitted to experimental data and regression coefficients obtained. Statistical significance of the terms in the regression equation was examined by ANOVA for each response. The significance of all the terms in the polynomial equations was judged by determining the probability level. When a model has been selected, an analysis of variance was calculated to assess how well the model represents the data. To evaluate the goodness of the models, coefficient of determination (R^2), F -values, the derived P values and coefficient of variance (CV) were determined. The lack-of-fit term was also used to judge adequacy of model fit. A Pearson's correlation matrix on product responses and system parameters and also between sensory attributes and textural parameters were carried out using SPSS 11.0 (SPSS Inc., Chicago, IL, USA) in order to determine correlation coefficients between parameters. Duncan's multiple range test was performed to determine differences between treatments by using SPSS.

Table 3.2 Process variables used in the central composite design for three independent variables for barley flour-tomato pomace and barley flour-grape pomace

	Variable level codes					
	Code	-1.682	-1	0	1	1.682
Die temperature($^{\circ}$ C)	X ₁	133	140	150	160	167
Screw speed (rpm)	X ₂	133	150	175	200	217
Pomace level (%)	X ₃	0	2	6	10	12.7

Table 3.3 Experimental design for extrusion experiment with coded and actual variable levels for barley flour

Run	Coded levels		Actual levels	
	X ₁	X ₂	Die temperature (°C)	Screw speed (rpm)
1	-1	-1	140	150
2	1	-1	160	150
3	-1	1	140	200
4	1	1	160	200
5	-1.414	0	136	175
6	1.414	0	164	175
7	0	-1.414	150	140
8	0	1.414	150	210
9	0	0	150	175
10	0	0	150	175
11	0	0	150	175
12	0	0	150	175
13	0	0	150	175

Table 3.4 Experimental design for extrusion experiment with coded and actual variable levels for barley flour-tomato pomace and barley flour-grape pomace

Run	Coded levels			Actual levels		
	X ₁	X ₂	X ₃	Die temperature (°C)	Screw speed (rpm)	Pomace level (%)
1	-1	-1	-1	140	150	2
2	1	-1	-1	160	150	2
3	-1	-1	1	140	200	2
4	1	1	-1	160	200	2
5	-1	-1	1	140	150	10
6	1	-1	1	160	150	10
7	-1	1	1	140	200	10
8	1	1	1	160	200	10
9	-1.682	0	0	133	175	6
10	1.682	0	0	167	175	6
11	-1	-1.682	0	150	133	6
12	-1	1.682	0	150	217	6
13	0	0	-1.682	150	175	0
14	0	0	1.682	150	175	12.7
15	0	0	0	150	175	6
16	0	0	0	150	175	6
17	0	0	0	150	175	6
18	0	0	0	160	175	6
19	0	0	0	150	175	6
20	0	0	0	150	175	6

Moreover, numerical optimization was carried out for independent variables of extrusion cooking of barley flour and barley flour-pomace blends. For this purpose, desirability function of response surface methodology was used. Desirability functions are one of the useful approaches to optimization of multiple responses. The desirability approach consists of the following steps: (1) Conduct experiments and fit response models (y_i) for all m responses. (2) Define individual desirability functions for each response (d_i). (3) Maximise the overall desirability with respect to the controllable factors (Corzo and Gomez, 2004). The general approach is to first convert each response y_i into an individual desirability function d_i that varies over the range $0 \leq d_i \leq 1$ where if response y_i is at its target value, then $d_i = 1$, and if it is outside an acceptable region, $d_i = 0$. Then the design variables were chosen to maximize the overall desirability as

$$D_o = (d_1 d_2 \dots d_m)^{1/m} \quad (3.1)$$

where m is the number of responses and D_o is the overall desirability. Design-Expert uses direct search methods to maximize the desirability function D_o (Myers and Montgomery, 2002).

3.5 Chemical Analysis

The proximate analyses of raw materials and total dietary fiber content of sensorily acceptable barley-tomato pomace and barley-grape pomace extrudates were performed by Silliker, Inc. (Modesto, Calif., USA). Ash, moisture, protein, starch and total dietary fiber were analyzed according to approved methods AOAC 940.26A, AOAC 925.40 (vacuum oven), AOAC 991.20E (Kjeldahl, protein factor: 6.25), EC L123/72 (Ewers starch) and AOAC 991.43, respectively (AOAC, 1995). Fat analysis was done by acid hydrolysis and the carbohydrate content was calculated by difference.

3.6 System Parameters

Specific mechanical energy, the mechanical energy input per unit mass of the extrudate, was calculated by dividing the net power input to the screw by the extrudate flow rate. SME input was calculated by the following equation (Sokhey and Chinnaswamy, 1992; Fan et al. 1996; Chang et al. 1999):

$$\text{SME (Wh.kg}^{-1}\text{)} = \frac{\text{screw speed (rad.s}^{-1}\text{)} \times \text{net torque (N.m)}}{\text{mass flow rate (kg.h}^{-1}\text{)}} \quad (3.2)$$

Torque was recorded every 6 s for at least 12 min and SME was calculated and averaged for each processing condition. Die pressure was measured using a Dynisco pressure transducer (PT-412, Dynisco, Franklin, MA, USA). Readings were recorded every 6 s for at least 12 min and average values were expressed as kPa. Die melt temperature was also measured by thermocouple and monitored for every 6 s by the computerized data acquisition system.

3.7 Physical Analysis for Extruded Products

3.7.1 Expansion

Expansion of extrudates was evaluated as sectional expansion. The width and thickness of 15 pieces of extrudate taken at random were measured with a digital caliper and the average calculated. The sectional expansion index (SEI) was calculated using the equation proposed by Alvarez-Martinez et al. (1988):

$$\text{SEI} = \frac{S_e}{S_d} = \frac{W_e \times h_e}{W_d \times h_d} \quad (3.3)$$

where S_e and S_d are the cross sectional areas of the extrudate and the die; W_e and h_e are the width and thickness of the extrudate and W_d and h_d are the width and thickness of the die respectively.

3.7.2 Bulk density

Bulk density (BD) was determined by measuring the volume of extrudate by glass bead displacement (Hwang and Hayakawa, 1980; Sokhey et al. 1997). Glass beads with a diameter range of 1.00 to 1.18 mm were used as displacement medium. Bulk densities of the extrudates were calculated as:

$$\rho_b = \frac{W_{ex}}{W_{gb}} \times \rho_{gb} \quad (3.4)$$

where ρ_b is the bulk density using glass bead displacement method (g/cm^3), W_{ex} is the extrudate mass (g), W_{gb} is the mass of glass beads displaced (g) and ρ_{gb} is the density of the glass beads (g/cm^3). The values were average of four measurements.

3.7.3 Water absorption and solubility indices

The water absorption index (WAI) is the weight of gel obtained per gram of dry ground sample. The WAI of extrudates was determined according to the AACC method 56-20 (AACC, 1995). The ground extrudate was suspended in water at room temperature. After standing for 10 min, gently stirred during this period, samples were centrifuged for 15 min at 1000g (Allegra™ 6 Centrifuge, Beckman Coulter Inc., Palo Alto, CA, USA). The supernatant was decanted into a tared aluminum pan. The WAI was calculated as the weight of sediment obtained after removal of the supernatant per unit weight of original solids as dry basis. The water solubility index (WSI) is the percentage of dry matter recovered after the supernatant is evaporated from the water absorption determination. The supernatant was dried in a vacuum oven at 84.4 °C and 20-24 mmHg gauge pressure for 24 h and weighed. The WSI was the weight of dry solids in the supernatant expressed as a percentage of the original weight of sample on dry basis (Jin et al. 1995). WAI and WSI determinations were replicated four times.

3.7.4 Texture

The peak force (PF) as an indication of hardness was measured with a TA-XT2i Texture Analyzer (Texture Technologies Corp., Scarsdale, NY, USA) using three point bend test with a sharp-bladed probe (55 mm wide, 40 mm high, 9 mm thick). The test speed was 2 mm/s and the distance between two supports was 22 mm. The curve was recorded and analyzed by Texture Exponent 32 software program (version 3.0). The slope (S) (N/mm) and distance (D_i) (mm) at which a product breaks were measured from force-distance curve and evaluated as crispness and brittleness respectively (Jackson et al. 1996; Texture Technologies (a)). Ten measurements were performed on each sample.

3.7.5 Color

HunterLab LabScan XE (Hunter Associates Laboratory, Inc., Reston, Virginia, USA) was used to determine color values of barley flour and ground extruded samples in terms of the Hunter *L*, *a* and *b* as measures of lightness, redness and yellowness, respectively. The measuring head was equipped with 51 mm diameter viewing port and used the system of diffuse illumination with 10° viewing geometry. The illuminant was D65. The colorimeter was calibrated against a standard white tile

($L=91.43$, $a=-0.74$, $b=-0.25$). The extrudates were ground in a laboratory grinder and passed through a 60 mesh sieve prior to color analysis. For each sample, four measurements were taken and averaged. The total color change (ΔE) was calculated as:

$$\Delta E = \sqrt{(L - L_o)^2 + (b - b_o)^2 + (a - a_o)^2} \quad (3.5)$$

where the subscript 'o' indicates initial color values of the raw material and L , a and b indicate color values of extrudates.

3.8 Sensory Analysis

Six samples that have acceptable textural properties (low hardness, high crispness and high brittleness) were selected from 6 experiments out of 13 for barley extrudates while five extrudate samples from 5 experiments out of 20 were selected to be based on textural property and different pomace level for both barley-tomato pomace and barley-grape pomace extrudates for sensory evaluation in terms of appearance, taste, off-odor, texture and overall acceptability. A semi-trained panel of thirty-five students and from faculty from the Food Engineering Department at the University of Gaziantep (Turkey) evaluated the extruded snacks for appearance (color and porosity), texture (hardness, crispness and brittleness), and overall acceptability on a 7-point hedonic scale (from 1=extremely dislike to 7=extremely like), while taste (bran flavor, bitterness, sweetness and tomato flavor) and off-odor were rated on a different 7-point scale (from 1=non to 7=very high). The samples approximately 5 g were presented in white cups labeled with random letters. The extrudates offered to the panelists at the same moisture content. Evaluation was conducted in individual air-conditioned booths at room temperature (23°C). The flavor attributes were evaluated under red lighting, while incandescent light was used for appearance and texture attributes. Panelists rinsed their mouths with water after tasting each sample. All sensory attributes were presented before sensory analysis. The sheet that contains definitions of sensory attributes was also placed in individual booths. Panelists evaluated hardness as amount of force required to break sample on first chew with molars. Crispness was measured as degree to which breaking noise is heard. Brittleness was measured as capacity of a sample to break up into numerous pieces during the first bite. Porosity was visually evaluated as number of pores per unit area by panelists.

3.9 Chemical Analysis for Extruded Products

3.9.1 Antioxidant activity

The antioxidant activity (AA) of raw materials and extrudates was measured by using method of Brand-Williams et al. (1995) as modified by Beta et al. (2005). It involves use of the free radical 2,2-diphenyl-1-picrylhydrazyl (DPPH), where antioxidants are allowed to react with the stable radical in a methanol solution. The extrudates were ground and passed through screen of 250 μm . 1 g of samples was extracted with methanol (10 mL) at room temperature for 2 h on a laboratory stirrer. After centrifugation at 1000g for 10 min, the supernatant was used for determination of antioxidant activity. An extract of 0.1 mL was reacted with 3.9 mL of a 6×10^{-5} mol/L of DPPH solution (2.4 mg of DPPH in 100 mL of methanol). The mixture was shaken immediately after adding DPPH and allowed to stand at room temperature in a dark and the decrease in absorbance was measured at 515 nm after 30 min using a Lambda 25 UV-visible spectrophotometer (Perkin Elmer, Shelton, CT, USA). The control was prepared without any extract and methanol was used as the blank. Antioxidant activity was expressed as the percent inhibition of the DPPH radical by the samples and was calculated using the following formula:

$$\% \text{ Inhibition} = \left(1 - \left[\frac{A_t}{A_o} \right] \right) \times 100 \quad (3.6)$$

where A_o is the absorbance of the control at $t=0$ min and A_t is the absorbance of sample at $t=30$ min. All determinations were performed in duplicate and results averaged.

3.9.2 Total phenolic content

Total phenolic content (TP) was determined in raw materials and extrudates by the Folin-Ciocalteu method (Singleton and Rossi, 1965) using the modification of Gao et al. (2002). The extrudates were ground and passed through screen of 250 μm . Samples (200 mg) were extracted with 4 mL of acidified (3 M HCl) methanol (HCl/methanol/water, 0.2:80:10, v/v) at room temperature for 2 h on a laboratory stirrer. The resulting mixture was centrifuged at 1000g for 10 min. The supernatant was used for the determination of total phenolics. 0.2 mL of each extract was mixed with 1.5 mL of freshly diluted 10-fold Folin-Ciocalteu reagent. After the mixture was allowed to equilibrate for 5 min, 1.5 mL of Na_2CO_3 (60 g/L) was added. The

absorbance of the mixture after incubation at room temperature for 90 min was measured at 725 nm using a Lambda 25 UV-visible spectrophotometer (Perkin Elmer, Shelton, CT, USA). The results were expressed as ferulic acid equivalents (mg ferulic acid/g sample (db)) using a calibration curve with ferulic acid. The calibration curve range was 50-500 $\mu\text{g/mL}$ ($R^2=0.9984$). All samples were analyzed in duplicates.

3.9.3 Starch digestibility

In vitro starch digestibility (SD) of raw materials and extrudates was determined according to the method of Onyango et al. (2004b). 5 mg of sample of extrudate was dissolved in 1 mL of 0.2 M phosphate buffer (pH 6.9). A stock solution of α -amylase was prepared by mixing 20 mg of porcine pancreatic α -amylase with 50 mL of same buffer. 0.5 mL of α -amylase solution was added to the sample suspension and incubated at 37°C for 2 h on an incubator with shaking (Model INNOVA 40R, New Brunswick Scientific, Edison, NJ, USA). After incubation period, 1 mL of 3-5 dinitrosalicylic acid was quickly added and the mixture was heated for 5 min in a boiling water bath to inactivate α -amylase. Samples were withdrawn and cooled. Then the solution was made up to 25 mL with distilled water and filtered prior to measurement of absorbance at 510 nm using a Lambda 25 UV-visible spectrophotometer (Perkin Elmer, Shelton, CT, USA). A blank for each sample was prepared by the same manner. But in this case the samples were incubated first and 3-5 dinitrosalicylic acid was added before addition of the enzyme solution. Maltose monohydrate was used as the standard and a standard curve was prepared using solutions containing known concentrations of maltose monohydrate (10-160 $\mu\text{g/mL}$). *In vitro* starch digestibility of samples was expressed as mg maltose/g sample (db). All the experiments were conducted at least in duplicate.

3.9.4 Determination of β -glucan content

The mixed linkage β -glucan assay kit (Megazyme International Ireland Ltd., Wicklow, Ireland) was used for determination of β -glucan content (BG) in barley flour and extrudates. The extrudates were ground and passed through a 420 μm screen prior to analysis. Extrudate samples (200 mg) and barley flour (80 mg) were put in glass centrifuge tubes and extrudate samples were pre-extracted with aqueous ethanol (50%, v/v) to remove free sugars and to reduce the levels of fats and oils.

Samples were wetted with 0.2 mL of aqueous ethanol to aid dispersion. Sodium phosphate buffer (4 mL, 20 mM, pH 6.5) was added and stirred on a vortex mixer. On mixing, the tubes were placed in a boiling water bath and incubated for 60 sec. The mixture was stirred vigorously on a vortex mixer and incubated at 100°C for a further 2 min and stirred again. The tubes were incubated at 50°C and allowed to equilibrate for 5 min. 0.2 mL of lichenase (10 U) was added to the tube and stirred. The tubes were sealed with parafilm and incubated for 1 h at 50°C with regular vigorous stirring on a vortex mixer. After incubation, 5 mL of sodium acetate buffer (200 mM, pH 4.0) was added to the tube containing barley flour and mixed vigorously. The amount of sodium acetate buffer added was reduced to 2 mL for extrudate samples. Then the tubes were allowed to equilibrate at room temperature for 5 min and centrifuged at 1000g for 10 min. 0.1 mL of aliquots were dispensed accurately into the bottom of three test tubes using micro-pipettor. β -Glucosidase (0.1 mL, 0.2 U) in 50 mM sodium acetate buffer (pH 4) was added to two of these tubes (the reaction). To the third tube, 50 mM acetate buffer (0.1 mL, pH 4.0) was added to prepare the reaction blank. All tubes were incubated at 50°C for 10 min. 3 mL of GOPOD reagent (glucose determination reagent) was added to each tube and incubated at 50°C for a further 20 min. The tubes were removed and measure the absorbance at 510 nm using a Lambda 25 UV-visible spectrophotometer (Perkin Elmer, Shelton, CT, USA). Absorbance of D-glucose standard was read at 510 nm against the reagent blank. The glucose standard comprises 0.1 mL sodium acetate buffer, 0.1 mL D-glucose standard and 3 mL of GOPOD reagent. The reaction blank contains 0.1 mL of distilled water instead of D-glucose standard. All the tests were performed in duplicate and the average values were reported. β -Glucan (%) was calculated on a dry weight basis with following equation:

$$\beta\text{-Glucan (\%)} = \Delta A \times F \times 94 \text{ (or 64)} \times \frac{1}{1000} \times \frac{100}{W} \times \frac{162}{180} \quad (3.7)$$

where ΔA = Absorbance after β -glucosidase treatment (reaction) minus reaction blank absorbance

F = A factor for the conversion of absorbance values to μg of glucose

= 100 (μg of D-glucose)/absorbance of 100 μg of D-glucose

94 = Volume correction factor for barley flour

64 = Volume correction factor for extrudates

1/1000 = Conversion from μg to mg

W = The calculated dry weight of the sample analyzed in mg

162/180 = A factor to convert from free D-glucose, as determined, to anhydro-D-glucose, as occurs in β -glucan.

3.9.5 Degree of starch gelatinization (Amylose-iodine complex formation)

The degree of starch gelatinization (DG) in extrudates was determined according to the method of Wootton and Chaudhry (1980) as modified by Ibanoglu et al. (1996), which is based on the formation of a blue iodine complex by amylose released during gelatinization. A ground sample (2.0 g, db) was dispersed in 100 mL of distilled water by mixing on a laboratory stirrer for 2 min at room temperature. The resulting suspension was then centrifuged at 1500g for 10 min and 1 mL iodine solution (4% KI, 1% I_2) was added to 1 mL of the aliquot of supernatant to form a blue complex with the dissolved amylose present in the sample and made up to 10 mL with distilled water. The absorbance (A_1) was then measured at 600 nm using a spectrophotometer (Lambda 25 UV/VIS Spectrometer, Perkin Elmer, Shelton, CT, USA) against a reference solution containing all reagents except extrudate sample. 2.0 g of unextruded barley flour-tomato pomace blends was heated and brought to boiling in water with gentle stirring, held for 5 min boiling and made up to 100 mL with distilled water. Starch in boiled unextruded blends was accepted as 100% gelatinized. The 100% gelatinized starch was treated with iodine and its absorbance (A_2) was measured as described previously. The extent of starch gelatinization in extruded samples was calculated as follows (Ibanoglu et al. 1996):

$$\text{Degree of gelatinization (\%)} = \frac{A_1}{A_2} \times 100 \quad (3.8)$$

3.10 Measurements of Starch Gelatinization by Differential Scanning Calorimetry

Gelatinization of barley flour, barley flour-pomace blends (100:0, 98:2, 94:6, 90:10 and 87.3:12.7 flour to pomace ratio on a dry basis) and selected extrudates were analyzed by using a differential scanning calorimeter (DSC-6, Perkin-Elmer, Netherlands). Selection of extrudates was based on the extreme and moderate conditions of extrusion of barley flour and barley flour-pomace blends. The

extrudates were ground and sieved through a 250 μm screen. Ground samples of about 10 mg (db) were weighed on aluminum pans and 25 μL of distilled water was added using a micro-syringe. The sample pans were sealed and allowed to equilibrate overnight in a refrigerator. Samples were heated at the rate of 5°C/min from 5 to 150°C with nitrogen flushing (40 cm^3/min). A sealed empty pan was used as a reference. Gelatinization onset temperature (T_o), peak temperature (T_p), conclusion temperature (T_c) and the enthalpy (ΔH) of the endotherm for gelatinization and the peak melting temperature of the amylose-lipid complex (T_{pcx}) and the enthalpy of melting of the amylose-lipid complex (ΔH_{cx}) were measured and calculated by using Pyris software (version 7.0).

3.11 Birefringence Measurements

A polarized light microscope (Model BX 51/52-P, Olympus, Japan) equipped with a 100-W halogen light source connected to a camera (Pixera, Model PVC100) and a computer was used to examine birefringence on barley flour and extrudate samples. The extrudates were ground and passed through a 125 μm screen. Before observation, samples were covered by a droplet of glycerol/water (20:20) solution and cover glass on microscope slide and observed at a magnification of 20X under a polarized light microscope. Two suspensions were prepared and analyzed for each sample. Pictures of ten different locations on the slide were taken randomly both under normal and polarized light. Granules or parts of granules with part of the Maltese cross still present were evaluated as ungelatinized granules during analysis.

CHAPTER 4

RESULTS AND DISCUSSION

This chapter covers effect of extrusion process variables on the system parameters and physical, chemical, textural and sensorial properties of barley, barley-tomato pomace and barley-grape pomace extrudates in the light of results obtained. The chemical composition of the raw materials (db) is given in Table 4.1. The carbohydrate content was over 82% for barley, tomato pomace and grape pomace but the distribution between starch, fiber and sugars varied considerably. The carbohydrate of barley was primarily starch (80% of total carbohydrate) and fiber while the carbohydrate of tomato pomace was fiber and sugars; the carbohydrate of grape pomace was mostly sugars. Fat content of tomato pomace was highest due to seed content when compared to barley and grape pomace. Protein content varied between 6.9 and 10.3% for raw materials.

Table 4.1 Chemical composition of raw materials (% db)

Raw material	Ash	Carbohydrate	Fat	Protein	Starch	Fiber
Barley flour	1.7	85.7	2.3	10.3	68.4	16.2
Tomato pomace	2.8	82.7	4.8	9.7	0.8	52.8
Grape pomace	4.1	86.6	2.4	6.9	0.0	12.3

4.1 System Parameters

4.1.1 Specific mechanical energy

Multiple linear regression equations of first and second orders polynomial model were generated relating specific mechanical energy as system parameters to coded levels of variables for barley and barley-pomace extrudates. The regression models allowed the prediction of the effects of independent variables on SME (Table 4.2). ANOVA was conducted to assess the significant effects of the independent variables on the responses and the responses significantly affected by the varying processing conditions. The significance of coefficients of fitted linear and quadratic models was evaluated by using the *F*-test, *p*-value, lack-of-fit and coefficient of variation.

Regression analysis indicated that the fitted models had coefficients of determination (R^2) of 0.882, 0.914 and 0.646 for barley, barley-tomato pomace and barley-grape pomace extrudates in the experimental data (Table 4.2). The lack-of-fit was not significant ($P>0.05$) for SME of barley and barley-tomato pomace extrudates whereas it was significant ($P<0.05$) for barley-grape pomace extrudates (Table A.1). The coefficients of variation for predicted models of SME were found to be 5.93, 8.51 and 12.21%, respectively for barley, barley-tomato pomace and barley-grape pomace extrudates. SME was significant on linear term of screw speed (X_2) ($P<0.05$) and quadratic term of temperature (X_1) ($P<0.05$) for barley extrudates. The positive coefficient of the first order term of screw speed (Table 4.2) indicated that SME increased with increase in this variable while negative coefficient of quadratic term of temperature suggested that excessive increase of this variable resulted in decrease of SME. Temperature had significant negative linear effect ($P<0.05$) with positive linear effect of screw speed for both barley-pomace extrudates. However, pomace level (X_3) had a significant positive linear effect on SME for only barley-tomato pomace extrudates followed by a negative quadratic effect of temperature (X_1^2) and screw speed (X_2^2) at $P<0.05$. The interaction of temperature and pomace level (X_1X_3) had a significant negative effect whereas the interaction of screw speed and pomace level (X_2X_3) had a significant positive effect ($P<0.05$) on SME of barley-tomato pomace extrudates.

Table 4.2 The regression models for system parameters using independent variables of temperature (X_1), screw speed (X_2) and pomace level (X_3) for barley and barley-pomace extrudates

Response	Model	R^2
SME _B	$208.85 + 26.0X_2 - 14.88X_1^2$	0.882
SME _T	$242.79 - 28.32X_1 + 30.53X_2 + 16.14X_3 - 14.06X_1^2$ $-14.67X_2^2 - 20.24X_1X_3 + 16.67X_2X_3$	0.914
SME _G	$217.36 - 32.79X_1 + 20.79X_2$	0.646
DP _B	$3749.41 - 385.61X_1 - 509.61X_1^2$	0.739
DP _T	$3806.71 - 1446.37X_1 - 1065.77X_1X_3$	0.874
DP _G	$3416.14 - 457.84X_1 - 375.56X_1^2$	0.739

B: barley flour, T: tomato pomace, G: grape pomace

SME indicates the mechanical energy input to the feed and is an indicator of material transformation (Lue et al. 1994). The values for SME in extrusion cooking of barley flour varied from 158 to 248 Wh/kg. Figure 4.1 shows the response surface plot of SME versus screw speed and temperature for barley extrudates. The SME increased

with increase in screw speed for barley extrudates. The increase of SME with screw speed is evident from Eq. (3.2) which shows that SME is proportional to the screw speed. The effect of screw speed on extrusion characteristics is usually complex. Conflicting results were reported as to the effect of screw speed on specific mechanical energy. Hsieh et al. (1989), Chang et al. (1999), Ryu and Ng (2001), Baik et al. (2004) and Doğan and Karwe (2003) observed that an increase in screw speed caused an increase in specific mechanical energy which was also true for the present work as the latter were proportional to screw speed, whereas Bhattacharya and Hanna (1987) observed that specific energy remained constant when screw speed increased. Lee et al. (1999) also observed that an increase in screw speed either increased or decreased SME input. The power consumption or specific energy usually increases when screw speed or dough mass viscosity increases (Jin et al. 1994). Therefore, the specific energy might increase or decrease with increasing screw speed, depending on the relative contribution of the screw speed and dough mass viscosity (Hsieh et al. 1991). Jin et al. (1994) also reported that specific energy increased with increasing screw speed because the effect of screw speed dominated the effect of mass viscosity. Baik et al. (2004) found that increasing the screw speed causes an increase in SME input in extrusion of regular and waxy barley flour attributed to the increase in shear rate when the screw speed was raised.

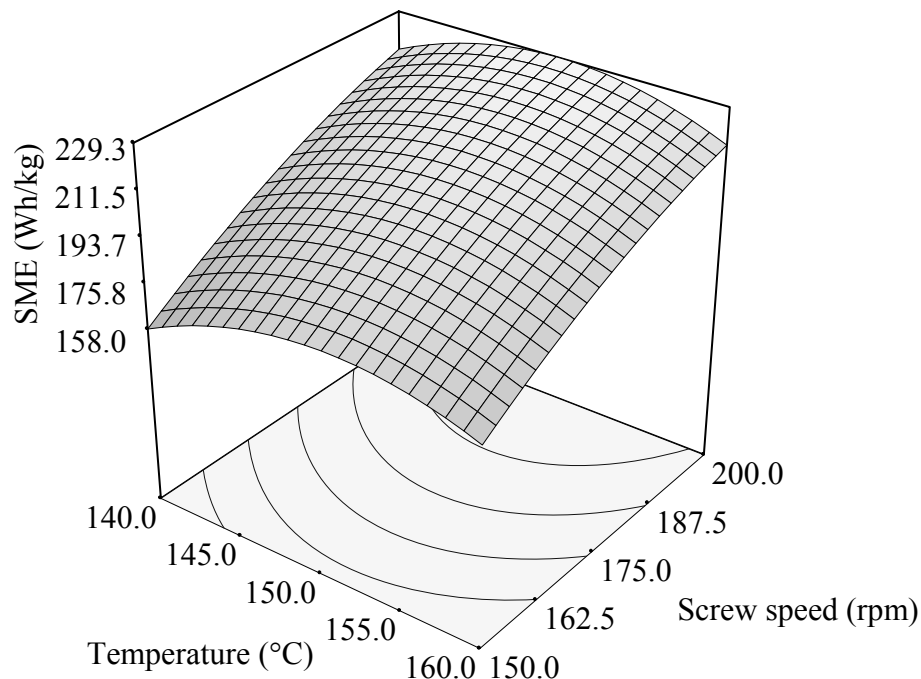


Figure 4.1 Response surface plot for the effect of screw speed and temperature on specific mechanical energy of barley extrudates

The linear effect of temperature was not significant ($P>0.05$) but the quadratic effect of temperature was significant ($P<0.05$) for SME of barley extrudates. An increase in temperature beyond 150°C caused decrease in SME (Figure 4.1). This can be explained by further increase in temperature causes a decrease in viscosity and hence a decrease in SME input (Chang et al. 1999).

The calculated SME during extrusion of barley flour-tomato pomace blends ranged from 144-341 Wh/kg while it was in the range of 120-305 Wh/kg for barley flour-grape pomace blends. The effects of screw speed and temperature on SME of barley-pomace extrudates are given in Figures 4.2 and 4.3. SME decreased with increasing temperature and decreasing screw speed. The trend of the effect of screw speed on SME for both barley-tomato pomace and barley-grape pomace extrudates was similar to that of barley extrudates. The decrease in SME with increasing temperature is in agreement with the findings of other authors in extrusion of sugar beet fiber with corn meal, cornmeal, wheat flour and whole cornmeal, quinoa flour and waxy barley (Hsieh et al. 1991; Chang et al. 1999; Ryu and Ng, 2001; Doğan and Karwe, 2003; Koxsel et al. 2004). Increases in barrel temperature cause increases in product temperature which led to the lower viscosities (Hsieh et al. 1989) of the product inside the extruder and resulting in the introduction of lower mechanical energy (Bhattacharya and Hanna, 1987; Chang et al. 1999; Hsieh et al. 1991).

The measured die melt temperature in extrusion cooking of barley flour-tomato pomace and barley flour-grape pomace blends ranged from 130-150°C and 132-158°C while 132-151°C for barley extrudates. Die melt temperature was negatively correlated ($R= -0.533$, $P<0.05$; $R= -0.637$, $P<0.01$) with SME for both barley-tomato pomace and barley-grape pomace extrudates but no correlation was observed for barley extrudates (Tables A.2, A.3 and A.4). It was obvious that increases in die temperature caused increases in product temperature. One might expect that as the product temperature in the melting zone increased, the viscosity of the dough would decrease which, in turn, would reduce torque and SME (Hsieh et al. 1991). Ryu and Ng (2001) reported that melt temperature in the die exit affected SME input and decreased with the increase in melt temperature for both wheat flour and whole cornmeal.

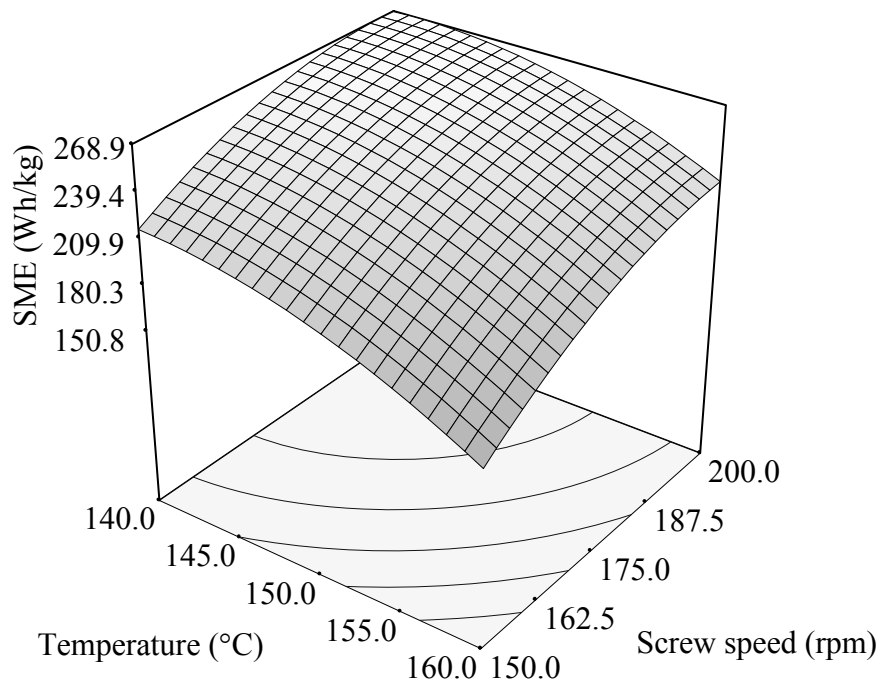


Figure 4.2 Response surface plot for the effect of screw speed and temperature on specific mechanical energy of barley-tomato pomace extrudates at a pomace level of 6%

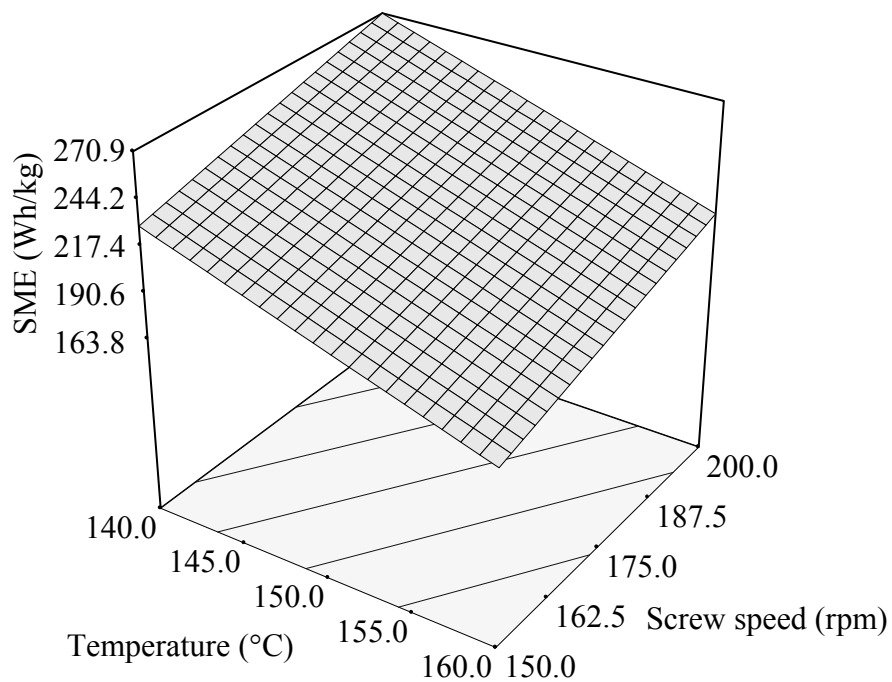


Figure 4.3 Response surface plot for the effect of screw speed and temperature on specific mechanical energy of barley-grape pomace extrudates at a pomace level of 6%

The interaction effects of tomato pomace level with temperature and screw speed on SME are shown in Figures 4.4 and 4.5. Increasing tomato pomace level in the blends increased significantly ($P < 0.05$) SME input in extrusion cooking of barley-tomato pomace blends. However, grape pomace level did not influence significantly ($P > 0.05$) SME of barley-grape pomace extrudates (Table 4.2). This difference between the tomato pomace and grape pomace might be attributed to the content of fiber. The total dietary fiber content of tomato pomace was higher than that of grape pomace (Table 4.1). An increase in SME could be explained that adding tomato pomace to barley flour gives a more viscous melt requiring a higher torque and causes an increase in SME input. The observed effect of tomato pomace on SME was similar to that reported by Hsieh et al. (1991) in extrusion of sugar beet fiber and corn meal. They reported that less water was available for starches in corn meal in the presence of sugar beet fiber. Because the viscosity of the starch-water system increases with decreasing water content, torque and specific energy increased with increasing sugar beet fiber. In a separate study, Lue et al. (1994) found a positive correlation between SME and sugar beet fiber particle size and it was reported that finer fiber tended to increase the SME of extrusion cooking of sugar beet fiber with corn meal. Jin et al. (1994) studied extrusion cooking of corn meal with soy fiber, salt and sugar. They reported that increasing the fiber content increased the viscosity of the dough mass and increased the friction between the feed material and the barrel surface and thus, a greater extruder torque was required to turn the extruder shaft. The authors also stated that an increase in specific energy could also be explained by the increased dough mass viscosity. On the other hand, Hsieh et al. (1989) found that adding wheat fiber or oat fiber to corn meals had no appreciable effects on percent torque and specific mechanical energy.

There was a large increase in SME with decreasing temperature at high tomato pomace level (10%) but at a low pomace level there was a small increase in SME (Figure 4.4). This was probably due to increasing viscosity from both low temperature and high pomace level as explained before. At low tomato pomace level, the increase in screw speed resulted in slight increase in SME, while at high pomace level, the increase in screw speed resulted in a large increase in SME (Figure 4.5).

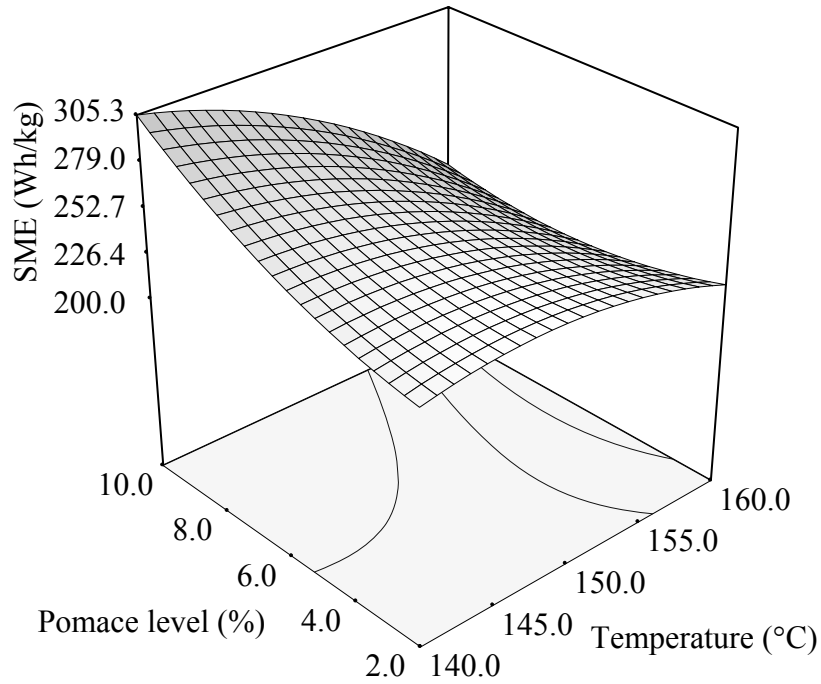


Figure 4.4 Response surface plot for the effect of temperature and pomace level on specific mechanical energy of barley-tomato pomace extrudates at a screw speed of 175 rpm

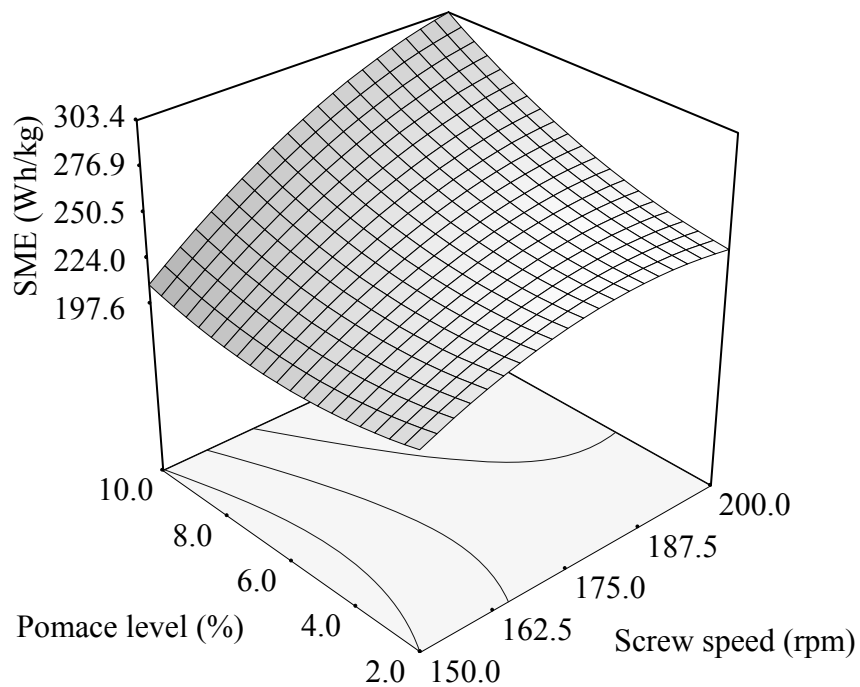


Figure 4.5 Response surface plot for the effect of screw speed and pomace level on specific mechanical energy of barley-tomato pomace extrudates at a temperature of 150°C

4.1.2 Die pressure

The equations of 2FI and second order polynomial models employed to predict the die pressure (P) developed in terms of coded variables is shown in Table 4.2 for barley and barley-pomace extrudates. ANOVA for the models as fitted (Table A.1) shows significance ($P < 0.05$) with a significant lack-of-fit for both barley and barley-tomato pomace extrudates but not significant ($P > 0.05$) lack-of-fit for barley-grape pomace extrudates. CV, which indicates the relative dispersion of the experimental points from the predictions of the model, was found 11.79 and 10.07% for die pressure of barley and barley-tomato pomace extrudates. A reasonably good coefficients of determination ($R^2 = 0.739$ and 0.874) and variation despite a significant lack-of-fit, showing that the models developed for the die pressure appeared to be adequate. The coefficient of determination was found to be 0.739 for die pressure of barley-grape pomace extrudates. It was observed that die pressure was significantly ($P < 0.05$) dependent on linear and quadratic terms of extrusion die temperature for both barley and barley-grape pomace extrudates. The regression analysis results indicate that die pressure was significant ($P < 0.05$) on linear term of temperature and interaction term of temperature and pomace level for barley-tomato pomace extrudates. Screw speed did not affect significantly ($P > 0.05$) the die pressure of extrudates as well as pomace level for barley-pomace extrudates. Similarly, Della Valle et al. (1987) found no clear influence of screw speed on product die pressure. However, other studies showed that die pressure decreased with an increase in screw speed during extrusion of yellow corn meals with wheat and oat fiber and corn grits from flint and sweet corn (Hsieh et al. 1989; Gujral et al. 2001).

The values for die pressure in extrusion cooking of barley, barley-tomato pomace and barley-grape pomace blends varied from 1559-4196, 786-6106 and 865-4256 kPa, respectively. The effect of screw speed and temperature on die pressure for barley extrudates and barley-grape pomace extrudates is given in Figures 4.6 and 4.7. Die pressure decreased with an increase in temperature for all type of extrudates. The effects of extrusion temperature on die pressure are consistent with earlier studies (Singh and Smith, 1997; Gujral et al. 2001; Singh et al. 2007). Singh and Smith (1997) compared extrusion behavior of wheat starch, whole wheat meal and oat flour. They found that the die pressure generally decreased with increasing temperature.

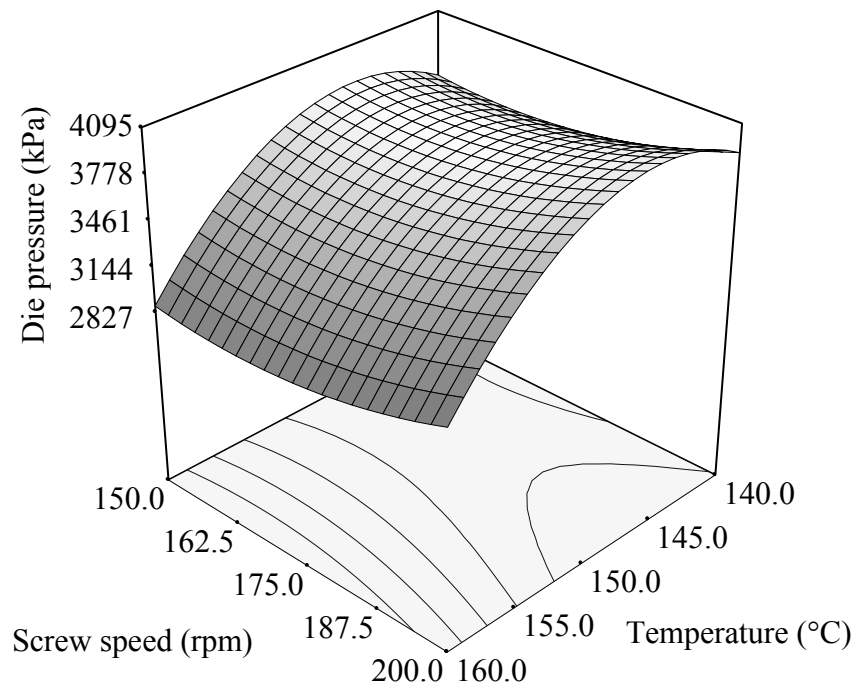


Figure 4.6 Response surface plot for the effect of temperature and screw speed on die pressure of barley extrudates

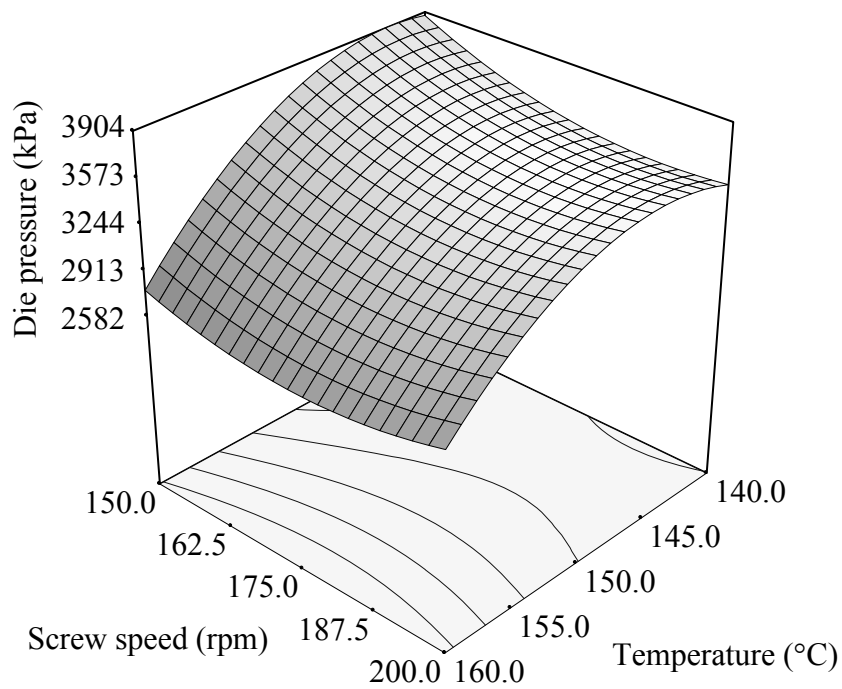


Figure 4.7 Response surface plot for the effect of temperature and screw speed on die pressure of barley-grape pomace extrudates at a pomace level of 6%

The decrease in die pressure with increase in temperature may be attributed to the decrease in viscosity of molten barley or blends (Singh and Smith, 1997; Singh et al. 1998; Ryu and Ng, 2001). Colonna et al. (1989) reported that higher temperatures increase product temperature, and in turn, decrease viscosity and pressure. This has been confirmed by temperature and pressure measurements (Chang et al. 1999). In our study, a negative correlation was also found between pressure and melt temperature ($R = -0.560$, $P < 0.05$; $R = -0.777$, $P < 0.01$; $R = -0.522$, $P < 0.05$) at the die for barley, barley-tomato pomace and barley-grape pomace extrudates (Tables A.2, A.3 and A.4). It indicates that an increase in melt temperature tended to decrease the die pressure. Della Valle et al. (1987) found that the viscosity of molten polymers generally increases as temperature decreases; this would explain why the pressure at the die rises when the temperature is reduced. Fletcher et al. (1985) reported that an increase in set temperature or screw speed resulted in increased drive power consumption, increased die temperature but decreased die pressure. Die pressure was positively correlated with SME ($R = 0.564$, $P < 0.001$; $R = 0.549$, $P < 0.05$) for barley-tomato pomace and barley-grape pomace extrudates whereas no correlation was found for barley extrudates.

The interaction effect of tomato pomace level and temperature is shown in Figure 4.8. Increasing temperature at low pomace level led to a slight decrease in die pressure, while an increase in temperature at high pomace level resulted in a dramatic decrease in die pressure. On the other hand, at low temperature, die pressures increased when pomace level increased, while at high temperature, the increase in pomace level resulted in decrease in die pressure. This indicated that the effect of pomace level on die pressure was dependent on temperature. Jin et al. (1994) found that an increase in added fiber tended to increase specific energy and die pressure but these increases were not significant for die pressure in extrusion of corn meal with soy fiber, salt and sugar. The authors stated that the increase in the die pressure with increasing fiber content probably resulted from an increased viscous dissipation of the dough mass. Meuser and Wiedmann (1989) found that increasing wheat bran in making flat bread by extrusion cooking resulted in increased SME but decreased the die pressure. They proposed that the pressure reduction was due to lower viscosity resulting from the decreasing starch content.

However, Hsieh et al. (1989) reported that incorporation of wheat fiber or oat fiber did not affect the die pressure.

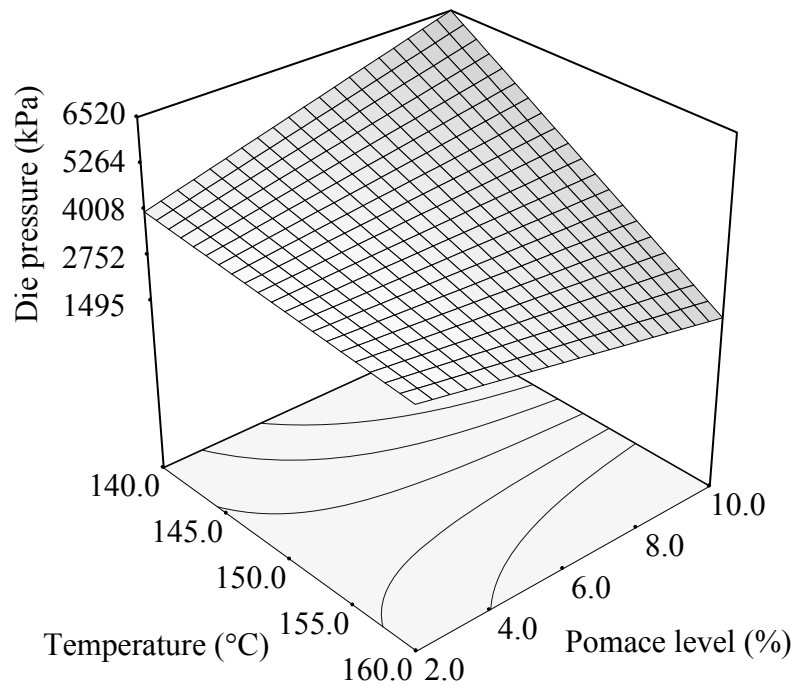


Figure 4.8 Response surface plot for the effect of pomace level and temperature on die pressure of barley-tomato pomace extrudates at a screw speed of 175 rpm

4.2 Physical Properties

4.2.1 Sectional expansion index

SEI is a measure of the cross-sectional area expansion of extrudates. The amount of expansion in food depends on the difference between the vapor pressure of water and the atmospheric pressure as well as the ability of the exiting product to sustain expansion. The pressure drop when the cooked melt suddenly goes from high pressure to atmospheric pressure causes an extensive flash-off of internal moisture and the water vapor pressure, which is nucleated to form bubbles in the molten extrudate, allows the expansion of the melt (Arhaliass et al. 2003). Multiple linear regression analysis of the experimental data yielded second order polynomial models for expansion measured as sectional expansion index in terms of coded variables for barley and barley-pomace extrudates (Table 4.3). The SEI was analyzed using ANOVA and the data are presented in Table A.5. The regression model for the influence of temperature (X_1) on SEI of extrusion of barley flour had a coefficient of determination (R^2) of 0.821. The ANOVA showed that the quadratic model was significant ($P < 0.05$), whereas lack-of-fit was not significant ($P > 0.05$) for SEI of

barley extrudates (Table A.5). Although the lack-of-fit was significant ($P < 0.05$) for models of SEI, the high coefficients of determination for SEI ($R^2 = 0.956$, $R^2 = 0.847$) with low coefficients of variation ($CV = 5.74\%$ and $CV = 8.33\%$) were found for both barley-tomato pomace and barley-grape pomace extrudates. Therefore, it is thought that the models described adequately SEI of barley-pomace extrudates. Regression analysis showed that SEI of barley extrudates was significantly ($P < 0.05$) affected by quadratic effect of temperature, while SEI of barley-pomace extrudates was significantly ($P < 0.05$) affected by both linear and quadratic effects of temperature. The linear and quadratic effects of tomato pomace level had significant effect on SEI of barley-tomato pomace extrudates, whereas only quadratic effect of grape pomace was significant for barley-grape pomace extrudates at $P < 0.05$. The interaction of temperature and tomato pomace level was found to be significant ($P < 0.05$) on SEI of barley-tomato pomace extrudates (Table 4.3).

Table 4.3 The regression models for sectional expansion index and bulk density using independent variables temperature (X_1), screw speed (X_2) and pomace level (X_3) of barley and barley-pomace extrudates

Response	Model	R^2
SEI _B	$1.90 - 0.31X_1^2$	0.821
SEI _T	$1.59 - 0.25X_1 - 0.18X_3 - 0.14X_1^2 + 0.073X_3^2 + 0.099X_1X_3$	0.956
SEI _G	$1.45 - 0.15X_1 - 0.13X_1^2 + 0.09X_3^2$	0.847
BD _B	$0.46 - 0.30X_1 + 0.12X_1^2$	0.977
BD _T	$0.53 - 0.15X_1 + 0.093X_1^2 + 0.085X_1X_3$	0.924
BD _G	$0.63 - 0.25X_1 + 0.093X_3 + 0.061X_1^2$	0.984

B: barley flour, T: tomato pomace, G: grape pomace

The measured SEI of barley flour extrudates was between 1.042 and 2.109, while it was in the range of 0.893-2.014 and 0.949-1.747 for barley-tomato pomace and barley-grape pomace extrudates. Moraru and Kokini (2003) reported that extrusion temperature plays an important role in changing the rheological properties of the extruded melts, which in turn affect the expansion volume. The effect of temperature and screw speed on SEI is given in Figure 4.9. According to the quadratic effect of temperature, increasing temperature (above 150°C) resulted in reduction in SEI of the extrudate. Images of barley extrudates are presented in Figures B.1 and B.2. After a certain level, higher temperature could lead to lower SEI but not necessarily lower overall expansion because longitudinal expansion might increase. The latter can especially be significant when a slit-die is used, as a gradual pressure drop is

encountered that could lead to nucleation of bubbles in the die and their growth along the longitudinal direction. Launay and Lisch (1983) proposed that the longitudinal and sectional expansions dependent on the melt viscosity and elasticity. The authors reported that an increased temperature would yield a lower melt viscosity and increased longitudinal expansion, while the melt viscosity would be lowered and cause a decrease in sectional expansion. Similar observations of the effect of temperature on product expansion have been reported for corn starch, corn grits and rice flour by Chinnaswamy and Hanna (1988), Ali et al. (1996) and Hagenimana et al. (2006). Chinnaswamy and Hanna (1988) found that expansion of corn starch increased as the barrel temperature increased from 110 to 140°C and declined with further increases in temperature. The increase in expansion of starch with temperature was attributed to its higher degree of gelatinization at such temperatures while reduction in expansion was attributed to molecular degradation as reported by the authors.

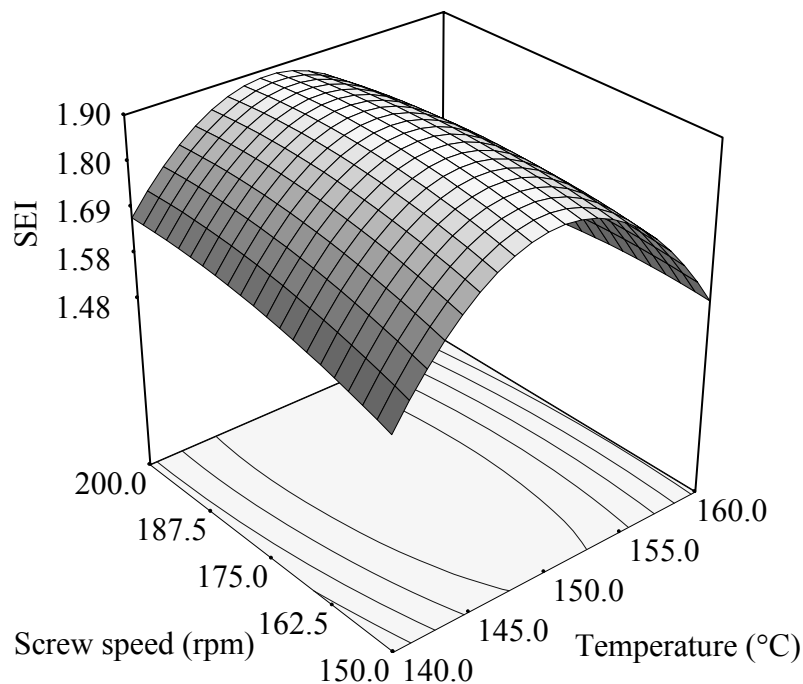


Figure 4.9 Response surface plot for the effect of temperature and screw speed on sectional expansion index of barley extrudates

It appears that SEI decreased with increasing barrel temperature for barley-pomace extrudates (Figs. 4.10 and 4.11), however volumetric expansion (inverse of bulk density) increased. This could indicate that longitudinal expansion increased

(Alvarez-Martinez et al. 1988). An inverse relationship between sectional and longitudinal expansion was reported for the study of extrusion of yellow grit and starch from different sources (Alvarez-Martinez et al. 1988; Della Valle et al. 1997; Desrumaux et al. 1998). Della Valle et al. (1997) reported that at high temperatures, the pressure of saturated vapor was higher than that of melt towards the die exit and this would favor the start of bubble growth inside the die in the direction of flow and thus higher longitudinal expansion. Hashimoto and Grossmann (2003) also reported a decrease in expansion with respect to increasing extrusion temperature for extrusion of cassava bran and cassava starch blends. Kokini et al. (1992b) found that after a critical temperature, which depends both on the type of starch and moisture content, expansion decreases with temperature, most likely due to excessive softening and potential structural degradation of the starch melt which becomes unable to withstand the high vapor pressure and therefore collapse. In addition, expansion phenomena are basically dependent on the viscous and elastic properties of melted dough (Launay and Lisch, 1983). Therefore, the elasticity loss with increasing temperature would then be one of the possible reasons for the decrease of SEI. This result is in agreement also with works of Ilo et al. (1999) and Doğan and Karwe (2003). Doğan and Karwe (2003) explained the decrease in SEI with increasing temperature by the negative effect of temperature on the elasticity of extrusion cooked melts. The authors also found that longitudinal expansion appeared to be extensively favored by lower melt viscosity at higher temperature and higher moisture level. Yuliani et al. (2006) observed decrease in expansion at high temperatures. The authors proposed two reasons for this. This was due to faster bubble collapse after the initial expansion at the die. They reported that at high temperature, melt viscosity decreased that facilitates bubble growth but as the bubble walls are very thin due to greater expansion at the lower melt viscosity, they cannot withstand the vapor pressure inside, resulting in wall fracture and rapid pressure loss allowing extrudate to collapse. The other reason was attributed by authors that at high temperature, rapid cooling of the extrudate surface could stop the growth of the bubbles, resulting in a decrease in expansion. On the other hand, some authors have approached in a different way for decreasing expansion with increasing temperature. Colonna et al. (1989) and Mendonça et al. (2000) reported that the decrease in expansion at higher temperatures could be attributed to increased dextrinization and weakening of structure.

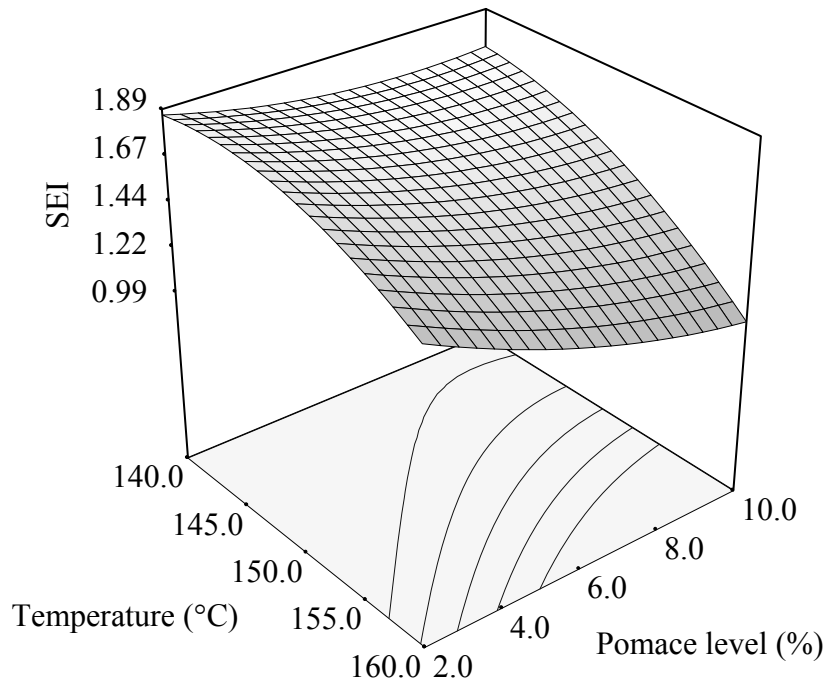


Figure 4.10 Response surface plot for the effect of pomace level and temperature on sectional expansion index of barley-tomato pomace extrudates at a screw speed of 175 rpm

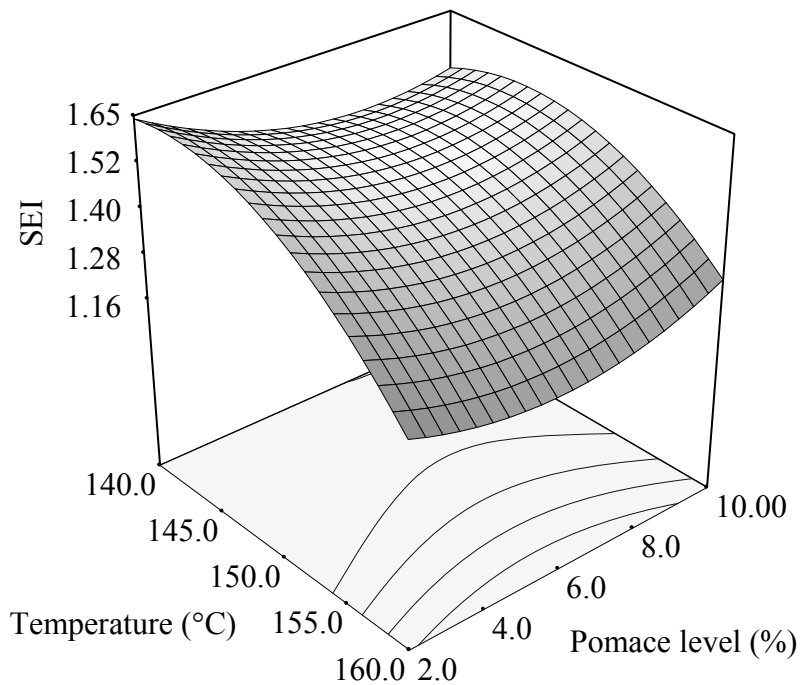


Figure 4.11 Response surface plot for the effect of pomace level and temperature on sectional expansion index of barley-grape pomace extrudates at a screw speed of 175 rpm

Kokini et al. (1992b) reported that screw speed has generally a positive effect on extrudate expansion due to increase in shear and thus decrease in melt viscosity induced by high screw speeds. Surprisingly, screw speed had no significant effect ($P>0.05$) on expansion for all extrudates in our study. The same behavior has been observed by Jin et al. (1995), Ding et al. (2005) and Liu et al. (2000) in extrusion of corn meal with soy fiber, salt and sugar, rice and oat flour with yellow corn flour. On the other hand, Ali et al. (1996) found that overall and radial expansions increased with screw speed, whereas axial expansion showed a reverse trend. Ryu and Walker (1995) found increases in the expansion ratio of wheat flour extrudates as the screw speed increased from 100 to 160 rpm, but then dramatically decreased at 180 rpm. Moraru and Kokini (2003) explained such differences might be due to significant differences in the extrusion conditions such as type of extruders and screw configuration, temperature and composition of the feed.

The effect of pomace level and temperature on SEI of barley-tomato pomace and barley-grape pomace extrudates are presented in Figures 4.10 and 4.11. Images of barley-tomato pomace and barley-grape pomace extrudates are also shown in Figures B.3, B.4, B.5 and B.6 with changes of temperature, screw speed and pomace level. As the percentage of tomato pomace in the blends increased, SEI decreased possibly due to dilution of starch with pomace and increasing level of fiber coming from pomace. We observed quadratic effect only for grape pomace on SEI of barley-grape pomace extrudates. Increasing grape pomace level up to 6% decreased SEI of extrudates and beyond that level SEI increased slightly with respect to the quadratic effect of pomace level (Figure 4.11). Chang et al. (1998) reported that as the concentration of jatobá flour in the mixture increased, the food fiber content increased while that of starch decreased, interfering the expansion of the products. The authors stated that the decrease in expansion due to increasing amounts of jatobá flour attributed to the dilution of starch content and increase in the fiber content in the blend, resulting in an increase in the mass viscosity and restricting expansion ability. In the study of Yanniotis et al. (2007), they observed that incorporation of wheat fiber into corn starch reduced the cell size, probably due to causing premature rupture of gas cells, which reduced overall expansion of extrudates. Grenus et al. (1993) found that increasing bran level in the rice flour decreased radial expansion or

product diameter, whereas the axial expansion increased with 10% rice bran followed by reduction at 20 and 30% rice bran levels. Onwulata et al. (2001b) observed that adding fiber at 50 g/kg did not change product characteristics, whereas the inclusion of fiber at 125 g/kg reduced expansion of the corn products. Moraru and Kokini (2003) suggested that at small concentration the long and stiff fiber molecules align themselves in the extruder in the direction of flow, reinforcing the expanding matrix and increasing its mechanical resistance in longitudinal direction. It has been reported that above a critical concentration, the fiber molecules disrupt the continuous structure of the melt, impeding its elastic deformation during extrusion. Fibers are also able to bind some of the moisture present in the matrix, thus reducing its availability for expansion (Moraru and Kokini, 2003). Hashimoto and Grossmann (2003) obtained the highest expansion at the lowest level of cassava bran in the blends of cassava starch. They attributed to the presence of fiber rupturing the walls of air cells and prevented the air bubbles from expanding to their full potential (Lue et al. 1991). Mendonça et al. (2000) reported that the deleterious effect of corn bran in radial expansion which was explained by inert components, such as fiber, rupturing the walls of air cells as well as the external surface of extrudates, thereby preventing the full expansion of the gas bubbles. Hsieh et al. (1991) found that increasing sugar beet fiber content in corn meal increased the axial expansion but decreased its radial expansion. A similar phenomenon has been observed for incorporation of wheat fiber or oat fiber content in corn meal by Hsieh et al. (1989). The degree of expansion is dependent on the size, number and distribution of air cells inside extrudates. With appropriate dough plasticity and elasticity, well expanded air pockets without rupture can be formed during extrusion. In contrast, if the material contains inert components (e.g. dietary fiber) that disrupt the stretching and setting of bubble films, tears and holes will appear in the wall of air cells as well as on the external surface of extrudates (Lue et al. 1990). The authors observed increasing dietary fiber content decreased the average cell size and increased the frequency of incomplete flakes and holes on the cell wall. Jin et al. (1995) found the size of air cells correlated with radial expansion of corn meal extrudates including soy fiber as in agreement with the study of Lue et al. (1991) on extrudates containing sugar beet fiber. Yanniotis et al. (2007) observed addition of pectin into corn starch reduced radial expansion of extrudates. They reported that when non-starch polysaccharides like pectin are present, they have the capacity to hydrate and consequently to

compete for and restrict the plasticizer and hence gelatinization process. Therefore, it was concluded that pectin reduced radial expansion by increasing the melt viscosity and reducing the availability of water for gelatinization. Camire and King (1991) found that cotton linter cellulose reduced expansion more than soy cotyledon fiber did. It has been reported that the nonstarch polysaccharides in both types of fiber might bind water more tightly during extrusion than do protein and starch. This binding might inhibit water loss at the die and thus reduce expansion. The another possible reason given by authors is that as the starch in the cornmeal might not have been fully gelatinized in the presence of nonstarch polysaccharides, then, it was less able to support expansion (Camire and King, 1991).

Increasing tomato pomace level at low temperature caused in a slight decrease in expansion but an increase in pomace level at high temperature resulted in a dramatic decrease in expansion (Figure 4.10). Higher temperatures would lower the viscosity of the dough mass to facilitate bubble growth. However, the presence of fiber could also reduce the elasticity and plasticity of the dough and thus decreased the sectional expansion (Hsieh et al. 1989).

The lower die pressure indicating a lower melt viscosity was consistent with lower SEI. This could be supported by the positive correlation between SEI and die pressure ($R=0.809$, $R=0.754$, $R=0.651$, $P<0.01$) for barley, barley-tomato pomace and barley-grape pomace extrudates (Tables A.2, A.3 and A.4). Sokhey et al. (1997) concluded that radial expansion (sectional expansion) depended directly on the pressure at the die nozzle. The pressure drop at the die is not directly responsible for expansion, but is the cause for bubble nucleation. It is the pressure difference between the vapor pressure of water inside nucleated bubbles and the pressure of the melt that drives expansion (Arhaliass et al. 2003). A negative correlation was observed between SEI and die melt temperature ($R= -0.494$, $P<0.05$; $R= -0.578$, $P<0.01$) for barley-tomato pomace and barley-grape pomace extrudates. Desrumaux et al. (1998) observed that expansion indices were closely correlated with die melt temperature and pressure in extrusion of corn grits. They reported that modifications in the die melt pressure and temperature changed water vapor pressure in the die and apparent viscosity of the melt. It was reported that the bubble growth is controlled by the pressure difference between the vapor pressure of water at the melt temperature

and the atmospheric pressure and by the apparent viscosity of the melt as well as the surface tension (Amon and Denson, 1984; Arhaliass et al. 2003).

4.2.2 Bulk density

Bulk density is a very important parameter in the production of expanded and formed food products. The expansion ratio and bulk density of extrudates seek to describe the degree of puffing undergone by the dough as it exits the extruder. Sectional expansion index considers expansion only in the direction perpendicular to extrudate flow, while unit bulk density considers expansion in all directions (Falcone and Phillips, 1988). It is accepted that the SEI is not a sufficient criterion for expansion by itself under tested extrusion conditions.

The regression equations for the relationship between bulk density and independent variables of temperature and pomace level obtained in terms of coded variables is presented in Table 4.3 for barley, barley-tomato pomace and barley-grape pomace extrudates. ANOVA for the model of bulk density shows significance ($P < 0.05$) for all type of extrudates and the lack-of-fit is not significant ($P > 0.05$) for both barley and barley-tomato pomace extrudates but significant ($P < 0.05$) for barley-grape pomace extrudates (Table A.5). Response surface regression model on bulk density yielded excellent fits with coefficient of determination $R^2 = 0.977$ and $R^2 = 0.924$ for barley flour extrudates and barley-tomato pomace extrudates, respectively. A high value of coefficient of determination ($R^2 = 0.984$) with a low value of coefficient of variation ($CV = 6.21\%$) of the predicted model of barley-grape pomace extrudates was observed even though having significant lack-of-fit. Bulk density of barley, barley-tomato pomace and barley-grape pomace was significantly ($P < 0.05$) affected by the linear and quadratic terms of temperature but was not significantly ($P > 0.05$) dependent on screw speed. The interaction term of temperature and tomato pomace level was found to be significant ($P < 0.05$) for barley-tomato pomace extrudates while the linear term of grape pomace was significantly ($P < 0.05$) influenced bulk density of barley-grape pomace extrudates.

Bulk density of barley flour extrudates varied widely between 0.26-1.07 g/cm³ while it ranged from 0.37-1.11 and 0.39-1.18 g/cm³ for barley-tomato pomace and barley-grape pomace extrudates based on the level of extrusion variables. The response

surface (Figure 4.12) shows that temperature had a dominant effect on bulk density, whereas screw speed had a non-significant effect. Baik et al. (2004) found that the bulk density of barley extrudates was not significantly affected by screw speed. It was also reported that screw speed had no significant effect on the bulk density of rice extrudates including rice bran, rice flour and amaranth extrudate and oat-corn puff (Grenus et al. 1993; Ilo et al. 1999; Liu et al. 2000). However, other authors have reported a significant effect of screw speed on bulk density. Seker (2005) found a reduction in bulk density of starch-soy protein extrudates with increasing screw speed. Similar findings were also observed for corn meal extrudates including soy fiber and rice extrudates by Jin et al. (1994) and Ding et al. (2006).

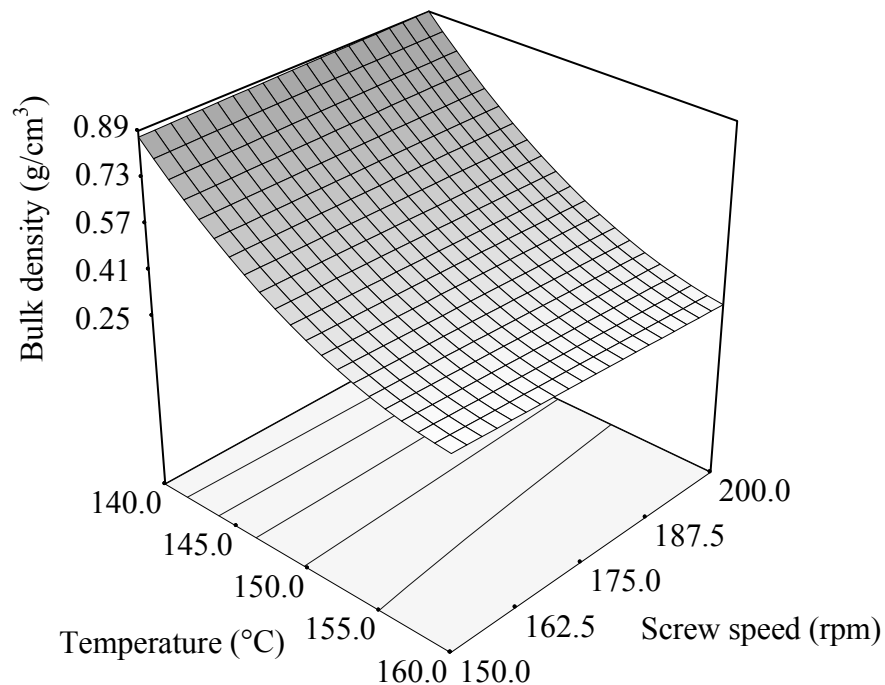


Figure 4.12 Response surface plot for the effect of screw speed and temperature on bulk density of barley extrudates

Bulk density values of extrudates decreased when temperature increased but at higher temperatures, its quadratic effect dominated. Ding et al. (2005) reported that an increase in the barrel temperature would decrease melt viscosity and the reduced viscosity effect would favor the bubble growth during extrusion. In addition, the degree of superheating of water in the extruder would increase at higher temperatures, also leading to greater expansion and hence gave low bulk density proposed by authors. Bhattacharya and Choudhury (1994) also reported an increase

in temperature markedly reduced the bulk density of rice extrudate and showed a curvilinear relationship. A decrease in bulk density with increasing temperature was observed for extrusion of rice by Hagenimana et al. (2006) and Guha and Ali (2006) as in agreement with Ilo et al. (1999) and Singh et al. (2007) for rice-amaranth extrudates and rice-pea grit extrudates. Guha and Ali (2006) explained that higher barrel temperature increases the extent of gelatinization and also the content of superheated steam that causes the rice extrudate to expand more, hence leading to the production of a low density product. Koksel et al. (2004) stated that higher temperature provides a higher potential energy for flash-off of super-heated water from extrudates as they leave the die and lose more moisture and become lighter in weight. Our results are in agreement with these studies.

Response surface plots for bulk density of barley-tomato pomace and barley-grape pomace extrudates as functions of temperature and pomace level are given in Figures 4.13 and 4.14. The effect of tomato pomace level on bulk density of barley-tomato pomace extrudates was found to be dependent on temperature. As shown in Figure 4.13, the lowest bulk density value was obtained at higher temperatures with a low level of tomato pomace, whereas the highest value was obtained at lower temperatures with a high level of tomato pomace. On the other hand, the effect of grape pomace on bulk density of barley-grape pomace extrudates was not dependent on temperature. An increase in percentage of grape pomace increased the bulk density (Figure 4.14). Increase in bulk density with increase in level of pomace may be due to the increasing fiber content of feed material. This was because the presence of fiber particles tended to rupture the cell walls before the gas bubbles had expanded to their full potential (Lue et al. 1991). Colonna and Mercier (1983) also reported that partially molten starch granules adhered to the cellulosic walls, leading to a composite wall of cellulose, gelatinised starch and cellular protein. The formation of this complex wall should restrict the product's expansion ability (Chang et al. 1998). Similar effect of fiber has been observed for extrusion of yellow corn with wheat and oat fiber, corn meal and sugar beet fiber, corn meal with soy fiber, salt and sugar, jatobá flour and cassava starch blends and corn starch with pectin and wheat fiber (Hsieh et al. 1989; Lue et al. 1991; Jin et al. 1994; Chang et al. 1998; Yanniotis et al. 2007). Sun and Muthukumarappan (2002) studied an extruded soy-based extrudates. They reported that increasing content of defatted soy flour (10-30%) into corn flour

decreased expansion ratio and increased bulk density of product. Grenus et al. (1993) found that the addition of 10% rice bran caused a small increase in product diameter but further increases in rice bran content reduced extrudate radial expansion and increased bulk density. Liu et al. (2000) reported that increases in oat flour percentage resulted in an increase in oat-corn puff bulk density. Hsieh et al. (1989) observed increases in the bulk density of both wheat fiber and oat fiber-containing corn meal extrudates when fiber content was increased in corn meals. Lue et al. (1990) studied microstructure of extruded corn meal with oat fiber or wheat fiber. They indicated that the degree of extrudate expansion was dependent on the source and amount of dietary fiber and was associated with the size of air cells inside extrudates and with the external structure of products. Increasing fiber content produced extrudates with a denser structure of reduced average cell size, increased number of holes on the cell wall, as well as increased number of apertures on the surface of extrudates. They concluded that the overall effect was a decrease in radial expansion and an increase in bulk density. Moore et al. (1990) found that density of extrudates increased as the concentration of the bran increased. The authors observed that cell number per pixel area increased greatly while average cell size decreased as bran concentration increased from 0 to 16%. It was also explained that bran interfered with bubble expansion reducing extensibility of cell walls and causing premature rupture of steam cells at a critical thickness related to particle size of bran and this would prevent formation of large cells (Moore et al. 1990). Lue et al. (1991) found the size of the air cells in the extrudate correlated with the radial expansion of the corn meal-sugar beet fiber extrudate; as the radial expansion decreased, the size of the air cells decreased. Sugar, from pomace, could be another reason for increase in bulk density of barley-grape pomace extrudates. Sugar could limit the availability of water. Limited water might also hinder gelatinization of starch, which could be another factor increasing bulk density. However, the actual mechanism has more to do with the plasticization effect of sugar leading to lower melt temperatures and thus reduce vapor pressure of water. Jin et al. (1994) reported that the addition of sugar reduced the product temperature, which might have also decreased or delayed the starch gelatinization.

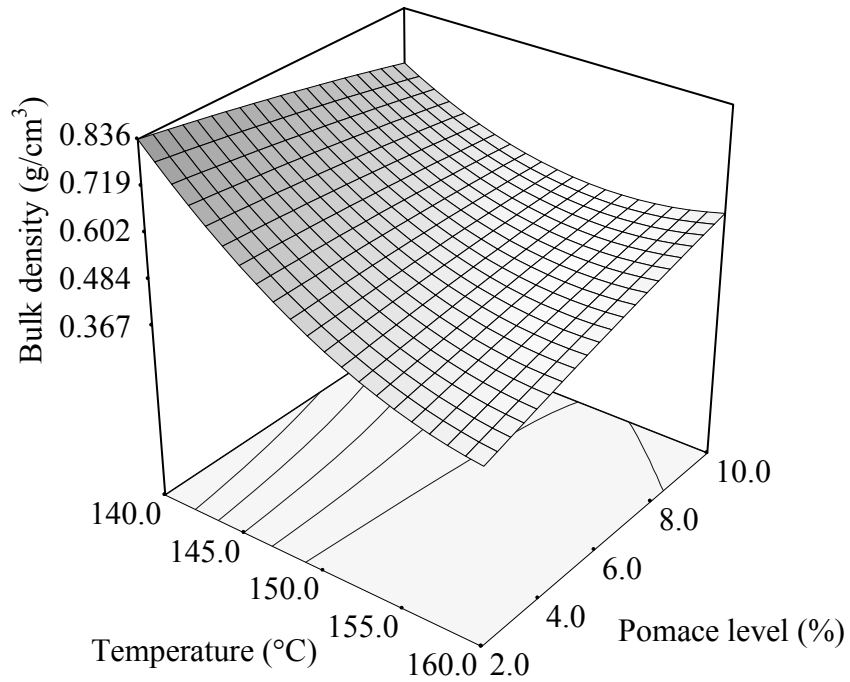


Figure 4.13 Response surface plot for the effect of pomace level and temperature on bulk density of barley-tomato pomace extrudates at a screw speed of 175 rpm

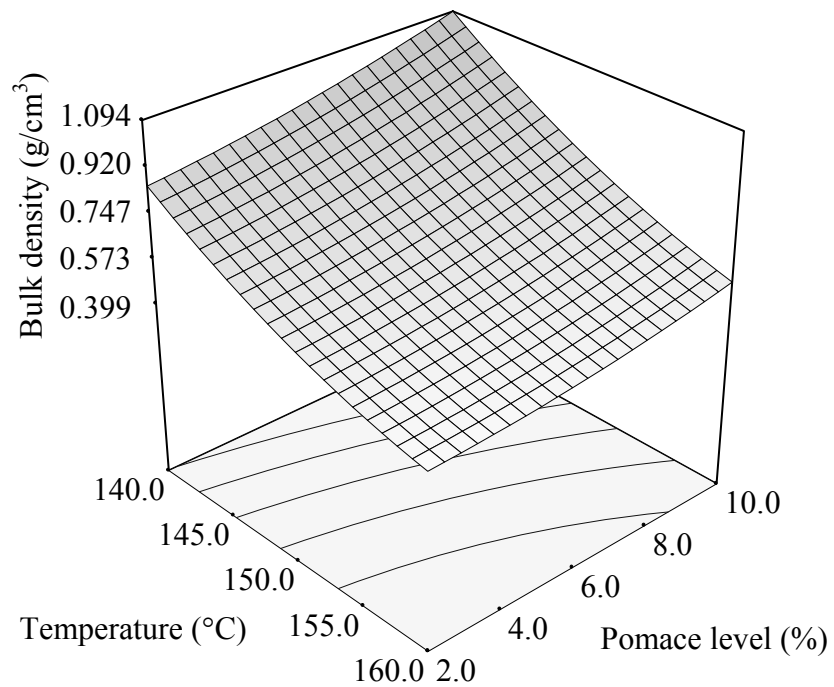


Figure 4.14 Response surface plot for the effect of pomace level and temperature on bulk density of barley-grape pomace extrudates at a screw speed of 175 rpm

4.2.3 Color parameters (Hunter L , a , b and ΔE)

Color is an important quality factor directly related to the acceptability of food products, and is an important physical property to report for extrudate products. The models developed for the lightness (L), redness (a), yellowness (b) parameters and total color change (ΔE) of barley-pomace extrudate and for Hunter L and ΔE values of barley extrudates as functions of coded independent variables are presented in Table 4.4. The significance of models of color parameters is given in ANOVA table (Table A.6). The lack-of-fit was found to be non-significant ($P>0.05$) for ΔE but significant ($P<0.05$) for Hunter L color parameter of barley extrudates. However, Hunter L model gave low coefficient of variation ($CV=0.81\%$) indicating that the experimental data could satisfactorily be explained by the model. The coefficients of determination (R^2) for Hunter L color parameter and ΔE were 0.547 and 0.749 for barley extrudates, respectively.

The lack-of-fit was not significant ($P>0.05$) for Hunter L , a color parameters and ΔE but significant for Hunter b color parameter ($P<0.05$) of barley-tomato pomace extrudates. CV was found to be 0.92, 2.81, 1.27 and 5.85% for Hunter L , a , b and ΔE values of barley-tomato pomace extrudates. The coefficients of determination (R^2) for Hunter L , a , b color parameters and ΔE of barley-tomato pomace extrudates were 0.971, 0.995, 0.992 and 0.861, respectively.

Table 4.4 The regression models for color parameters using independent variables temperature (X_1), screw speed (X_2) and pomace level (X_3) of barley and barley-pomace extrudates

Response	Model	R^2
L_B	$76.62 - 0.72X_1$	0.547
L_T	$69.95 - 3.23X_3 + 0.79X_3^2$	0.971
L_G	$72.89 - 2.07X_3 + 0.94X_3^2$	0.955
a_T	$9.88 + 3.21X_3 - 0.92X_3^2$	0.995
a_G	$3.79 + 0.91X_3$	0.847
b_T	$22.89 - 0.32X_1 + 2.88X_3 + 0.17X_1^2 - 0.86X_3^2$	0.992
b_G	$16.27 + 0.93X_3$	0.840
ΔE_B	$5.98 + 0.60X_1 - 0.58X_1^2$	0.749
ΔE_T	$8.90 - 0.31X_1 + 0.69X_3 - 0.80X_3^2$	0.861
ΔE_G	$5.47 + 0.22X_3 - 0.32X_3^2 + 0.36X_1X_3$	0.735

B: barley flour, T: tomato pomace, G: grape pomace

The lack-of-fit of the models for color parameters was not significant ($P>0.05$) but it was significant ($P<0.05$) for ΔE of barley-grape pomace extrudates. Although the lack-of-fit was found significant ($P<0.05$) for ΔE , the low coefficient of variance was observed for ΔE with a value of 6.42%. The regression analysis for the determination of the desired constants yielded a regression coefficient (R^2) of 0.955, 0.847, 0.840 and 0.735 for Hunter color parameters L , a , b and ΔE , respectively, for barley-grape pomace extrudates.

Hunter L value and ΔE of barley extrudates were significantly affected ($P<0.05$) only by temperature. It was observed that the independent variables of temperature and screw speed didn't influence significantly ($P>0.05$) both Hunter a and b color values of barley extrudates. Therefore, detailed analyses of the two color values were not presented. Tomato pomace level was an important variable in the response surface models of product color parameters as its linear and quadratic terms were significant at $P<0.05$. The color parameter Hunter b of barley-tomato pomace extrudate was significantly ($P<0.05$) affected by linear and quadratic terms of temperature. Temperature had also significant ($P<0.05$) effect on ΔE of barley-tomato pomace extrudates. According to the regression results, grape pomace level had a dominant effect on color parameters (Hunter L , a , b) and ΔE . Grape pomace level had a negative significant linear effect on Hunter L value, whereas it had a positive significant effect on Hunter a , b values and ΔE at $P<0.05$. On the other hand, the quadratic effect of grape pomace level was found to be significant ($P<0.05$) only for L value and ΔE . ΔE was also affected significantly ($P<0.05$) by interaction effect of temperature and grape pomace level. The effect of screw speed on color parameters was not significant for all type of extrudates ($P>0.05$). Liu et al. (2000) found screw speed showed no significant effect on the lightness and yellowness of the extrudate but significantly enhanced the redness of the oat-corn puff extrudate. Grenus et al. (1993) also observed the increase in screw speed had no significant effect on darkness of the extrudate. Ilo and Berghofer (1999) found that the lightness and redness were markedly dependent on barrel temperature and feed moisture content, whereas screw speed was not significant. Our observations are similar to these studies.

Hunter L value of barley flour was 80.95, whereas it was varied between 74.93 and 77.88 for extruded samples. Hunter a value, indicative of the redness of sample with positive values, ranged between 2.06 and 3.02 for barley extrudates while barley flour had Hunter a value of 1.75. The positive Hunter b value indicates the yellowness of the sample. The barley flour had a Hunter b value of 11.58 while the extruded barley samples had Hunter b values in the range of 14.53 and 16.18. The barley flour-tomato pomace extrudates had color values in the ranges: L , 65.85-76.32; a , 2.70-12.66; b , 16.10-25.63, while the barley flour-grape pomace extrudates had color values in the ranges: L : 70.70-77.93; a : 2.35-5.43; b : 14.47-17.69. The effect of temperature and screw speed on Hunter L value is given in Figure B.7. Hunter L color parameter of the barley flour extrudate decreased with increasing temperature. Similar effect of temperature on Hunter L value was found by Ilo and Berghofer (1999) in extrusion cooking of maize grits. This is possibly due to occurrence of browning reactions that increase Hunter a and b values. Apruzzese et al. (2000) found that increased temperature at constant screw speed provided a darker product. A high temperature range, where Maillard reactions become important, results in increased browning of the final product (Apruzzese et al. 2000) and hence reduction in lightness. Pelembe et al. (2002) also reported that the higher extrusion temperature resulted in darker products, probably due to Maillard reaction favored by the high temperature and relatively low water in the extruder. Bhattacharya et al. (1997) reported brightness of rice based extrudates decreases as a result of extrusion processing, particularly with temperature. They observed that an increase in temperature from 100 to about 140°C markedly decreased the brightness of the samples but beyond 140°C, the decrease in brightness values was rather low. Gutkoski and El-Dash (1999) found that as the extrusion temperature increased, the luminosity (L^*) was lowered to a minimum at 120°C and then increased again. Ilo et al. (1999) observed during extrusion cooking the L^* value decreased, whereas a^* and b^* values increased.

The effect of level of tomato pomace on both Hunter L and a color parameters is given in Figures B.8 and B.9. Among the color parameters, Hunter L and a values showed marked changes due to addition of tomato pomace. Image of extrudates containing 0 and 12.7% of tomato pomace is given in Figure B.10. Temperature had no significant effect on Hunter L and a values (Figures B.8 and B.9). An increase in

tomato pomace level decreased Hunter L value of the samples and increased Hunter a value of samples as expected due to the lycopene pigment in the tomato pomace. Liu et al. (2000) reported that the redness of the oat-corn extrudate was enhanced with the percent oat flour and attributed to higher redness of oat flour than corn flour. The increase in darkness could be attributed to the darkness of the bran compared to the nearly white rice flour. In study of Wu et al. (2007), color of extruded flaxseed-corn puff with respect to lightness, redness and yellowness was affected significantly by flaxseed meal content. They obtained the darker (lower in lightness), redder, but less yellow extrudate with the higher the flaxseed meal content. Sun and Muthukumarappan (2002) found L^* value decreased with increasing defatted soy flour content, whereas a^* and b^* values increased significantly with increasing soy flour content. The effect of temperature and tomato pomace level is shown in Figure B.11. The change in yellowness (Hunter b value) decreased with increasing temperature which is in agreement with the results of Ilo and Berghofer (1999). They reported that the changes in yellowness during extrusion cooking of yellow maize induced by the effects of two different reactions: the nonenzymatic browning and pigment destruction. They also concluded that some of the carotenoids might have been damaged by the thermal treatment and some browning might have made up the color loss. Increasing tomato pomace content resulted in a significant ($P < 0.05$) increase in the extrudate Hunter b value.

The effect of grape pomace level for color parameters is presented in Figures B.12, B.13 and B.14. Image of extrudates containing 0 and 12.7% of grape pomace is given in Figure B.15. The reduction in lightness with increasing grape pomace level may be resulted from occurrence of browning reaction such as Maillard reactions and caramelization, because of contribution of more sugar (from grape pomace) that favored the browning reaction (Maga, 1989). The increase in a value of the extrudates may be associated with Maillard reaction and destruction of heat sensitive pigments. The change in b value increased with increasing grape pomace level which may be due to the yellowish pigments present in the pomace. Temperature and screw speed did not affect significantly ($P > 0.05$) all three color parameters (Hunter L , a , b) of barley-grape pomace extrudates. Sacchetti et al. (2004) observed the snack's lightness (L^*) tended to decrease with the increase in chestnut flour (that is dark) content in the initial blend. They found that extrusion temperature appeared to have

little effect on the product L^* and a^* values with low chestnut flour, whereas the extrusion temperature caused product browning with increased level of chestnut flour. This was attributed to the high reducing sugars content of chestnut flour, which could promote color changes due to Maillard reaction development according to extrusion temperature. Analogue changes of the a^* and b^* parameters due to the Maillard reaction development was also reported by authors. Ilo and Berghofer (1999) also reported that the changes in lightness and redness during extrusion cooking of yellow maize grits showed to represent only the effects of browning reactions. A significant decrease in lightness and increase in redness and yellowness were observed for extrudates made from extrusion of unfermented maize-finger millet blend (Onyango et al. 2004a). They stated that reducing sugars formed as a result of mechanical shear of starch reacted with amino acids forming Maillard products and therefore caused changes in color parameters.

Hunter L value was negatively correlated with Hunter a and Hunter b values for barley, barley-tomato pomace and barley-grape pomace extrudates (Tables A.2, A.3 and A.4). The same correlation between parameters L and a was observed by Ilo and Berghofer (1999) in extrusion cooking of maize grits. On the other hand, Hunter a and b values were positively correlated with each other for extrudates. The Hunter L value was positively correlated with SEI of barley-tomato pomace extrudates (Table A.3) but similar correlation was not observed for other barley and barley-grape pomace extrudates. An increase in expansion gives more bright color in extrudates due to air cells rather than dull color. The ΔE value of barley extrudates varied from 4.51 to 7.27 while it was in the range of 5.56-9.99 and 4.21-6.31 for barley-tomato pomace and barley-grape pomace extrudates. The effects of independent variables on ΔE value are shown in Figure 4.15. The total color change of barley extrudate increased with increasing temperature. Increasing process temperature in extrusion cooking increased the rate of browning reactions which increased the total color difference (Ilo et al. 1999). Although the screw speed was not significant independent variable, a slight increase in color change with increasing screw speed was observed (Figure 4.15). The effect of temperature and tomato pomace level on ΔE value of extrudates is shown in Figure 4.16. Results of regression analysis show that color change of barley-tomato pomace extrudates was most dependent on tomato pomace content. An increase in tomato pomace level caused an increase in ΔE value

with respect to the Hunter *a* and *b* values of barley-tomato pomace extrudates. Response surface plot for total color change as functions of the grape pomace level and temperature is given in Figure 4.17. Color change increased as grape pomace level increased. The interaction between temperature and grape pomace level was found to be significant ($P < 0.05$). The low value of total color change was obtained at high temperatures with a low percentage of grape pomace. At low grape pomace level, the increase in temperature resulted in a slight decrease in ΔE value, while at high grape pomace level, the increase in temperature led to large rise in ΔE value of barley-grape pomace extrudates. This may be related to the increasing amount of sugar coming from grape pomace because the carbohydrate of grape pomace was mostly sugar (Table 4.1). When the grape pomace was increased in the blend, the temperature increase may cause the product browning and thus increased ΔE value at high pomace level and temperature. A negative correlation was observed between total color change and lightness but positive correlation was found for redness and yellowness for barley, barley-tomato pomace and barley-grape pomace extrudates (Tables A.2, A.3 and A.4). Similarly, Sacchetti et al. (2004) reported the interaction effect of temperature and chestnut flour content on color parameters. They found that temperature affected slightly the color parameters (L^* and a^*) at low chestnut flour content but increase in temperature caused product browning at high chestnut flour content.

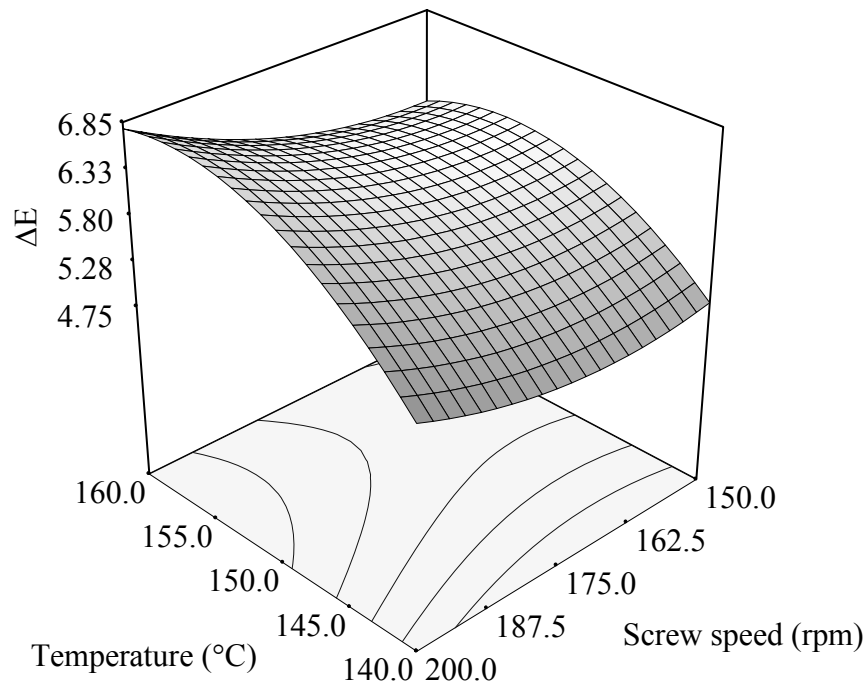


Figure 4.15 Response surface plot for the effect of screw speed and temperature on total color change of barley extrudates

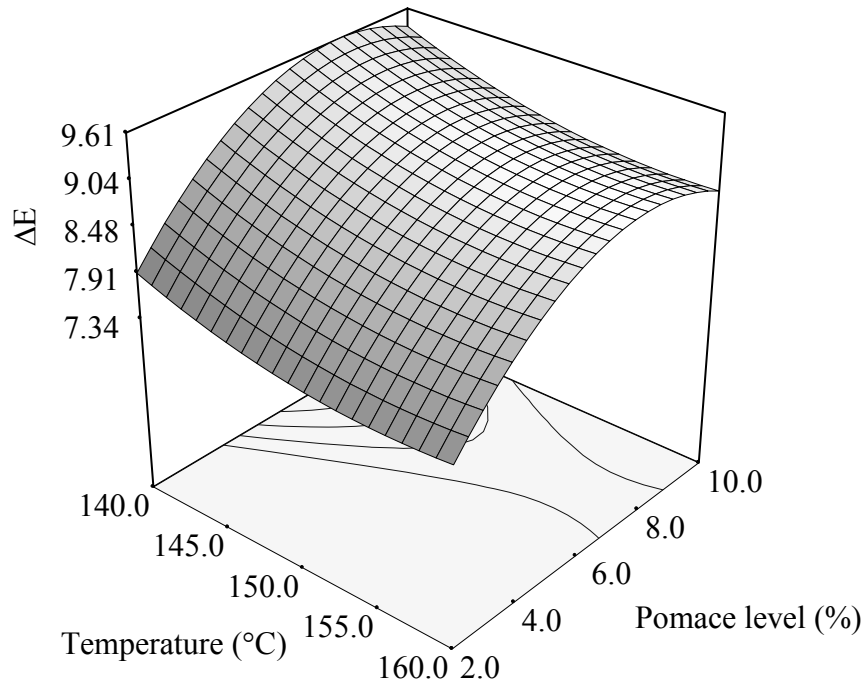


Figure 4.16 Response surface plot for the effect of pomace level and temperature on total color change of barley-tomato pomace extrudates at a screw speed of 175 rpm

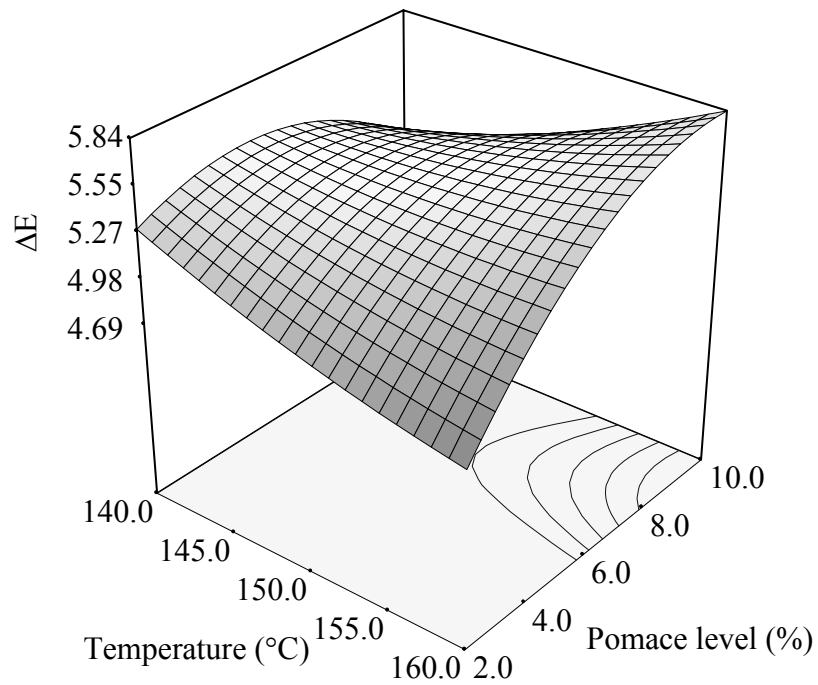


Figure 4.17 Response surface plot for the effect of pomace level and temperature on total color change of barley-grape pomace extrudates at a screw speed of 175 rpm

4.3 Functional Properties

4.3.1 Water absorption index

Equations derived from multiple regression analysis relating water absorption index with coded levels of the variables are shown in Table 4.5 for barley-tomato pomace and barley-grape pomace extrudates. No significant model was found for the WAI of barley flour extrudates. Therefore, it was not presented in the Table 4.5. ANOVA for models of the WAI is given in Table A.7. The lack-of-fit was found to be significant ($P < 0.05$) for the WAI of barley-grape pomace extrudates with low coefficient of variation ($CV = 2.33\%$), whereas the lack-of-fit was not significant ($P > 0.05$) for the WAI of barley-tomato pomace extrudates. The coefficients of determination (R^2) for the WAI of barley-tomato pomace and barley-grape pomace extrudates were 0.557 and 0.739, respectively. The negative coefficients of the linear terms of temperature and pomace level (Table 4.5) indicated that the WAI of barley-tomato pomace extrudates decreases with increasing these variables. Statistical analysis showed that grape pomace level had significant effect on the WAI of barley-grape pomace extrudates but the effect of temperature was not significant ($P > 0.05$). The WAI of barley-grape pomace extrudates was significantly affected by interaction of screw speed and grape pomace level ($P < 0.05$). The effect of screw speed on WAI of barley-tomato pomace extrudates was not significant ($P > 0.05$). Similar result was observed by Seker (2005) for native starch/soy protein mixture extrudate. González-Soto et al. (2007) found that the screw speed did not show an effect on WAI of banana starch extrudates as well as in the study of Ding et al. (2005) and Ding et al. (2006) for rice and wheat extrudates.

Table 4.5 The regression models for functional properties using independent variables temperature (X_1), screw speed (X_2) and pomace level (X_3) of barley and barley-pomace extrudates

Response	Model	R^2
WAI_T	$6.54 - 0.12X_1 - 0.18X_3$	0.557
WAI_G	$6.37 - 0.19X_3 + 0.18X_2X_3$	0.739
WSI_B	$8.74 - 0.51X_1 + 0.95X_2 - 0.98X_1^2$	0.872
WSI_T	$9.66 - 1.15X_1 + 1.03X_2 + 0.98X_3$	0.831
WSI_G	$10.80 - 0.69X_1 + 1.49X_3 + 0.68X_3^2$	0.865

B: barley flour, T: tomato pomace, G: grape pomace

The WAI measures the volume occupied by the granule or starch polymer after swelling in excess water, while the WSI determines the amount of free polysaccharide or polysaccharide released from the granule after addition of excess water (Sriburi and Hill, 2000). The measured WAI of barley extrudates ranged between 6.42 and 7.26 g gel/g dry sample. The independent variables of processing conditions such as temperature and screw speed didn't affect significantly ($P>0.05$) WAI of barley flour extrudates. The WAI of barley-tomato pomace and barley-grape pomace extrudates varied between 6.10-7.03 and 5.85-6.97 g gel/g dry sample for the various extrusion conditions studied.

The effect of temperature and pomace level on WAI of barley-tomato pomace extrudates is shown in Figure 4.18. Water absorption depends on the availability of hydrophilic groups which bind water molecules and on the gel-forming capacity of macromolecules (Gomez and Aguilera, 1983). Increasing temperature significantly ($P<0.05$) decreased the WAI of extrudates. Pelembe et al. (2002) found lower value of WAI at the higher extrusion temperature. A decrease in WAI with increasing temperature was probably due to decomposition or degradation of starch molecules (Pelembe et al. 2002). Mercier and Feillet (1975) reported that the WAI for waxy corn and other starches decreased with increasing extrusion temperature from 70 to 225°C. They indicated that WAI decreases with the onset of dextrinization. Badrie and Mellows (1991) found that increasing temperature from 100-105°C to 120-125°C lowered WAI of cassava extrudates. Lee et al. (1999) found rapidly increasing WAI with increasing barrel temperature up to 90°C but decreased at temperatures higher than 90°C in the range of 80-100°C. Guha et al. (1997) observed a decrease in WAI when temperature was increased in the range of 80-120°C. It might be related to the degradation of starch that causes a reduction in the water holding capacity of the molecules as a result of decrease in molecular size (Guha et al. 1997). It was reported that one can expect more undamaged polymer chains and a greater availability of hydrophilic groups which can bind more water at low temperature and resulted in higher values of WAI (Gomez and Aguilera, 1983; Guha et al. 1997). Similar results were reported by Ding et al. (2005) and Ding et al. (2006) for rice and wheat extrudates. Ding et al. (2006) also stated that the WAI decreases with increasing temperature if dextrinization or starch melting prevails over the gelatinization phenomenon.

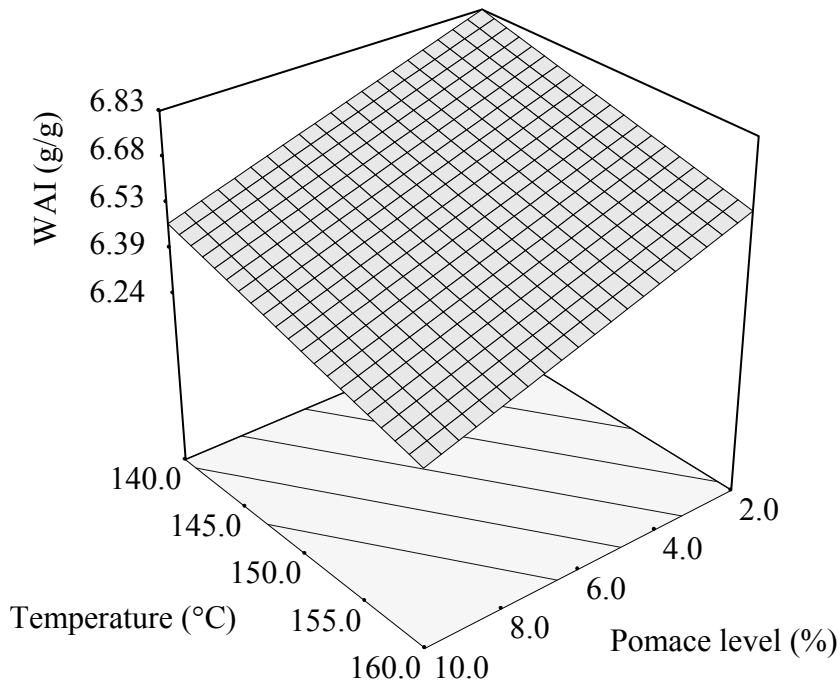


Figure 4.18 Response surface plot for the effect of pomace level and temperature on water absorption index of barley-tomato pomace extrudates at a screw speed of 175 rpm

Hashimoto and Grossmann (2003) showed that increasing extrusion temperature from 150 to 180°C decreased the WAI of cassava bran/cassava starch extrudates, which was probably due to an increase in starch degradation. A decrease in WAI with the increase in extrusion temperature at low moisture content was also reported by Kirby et al. (1988). On the other hand, opposite results have been observed for WAI of extruded whole pinto bean meal, quinoa extrudates and rice based extrudates including pea grits (Baladrán-Quintana et al. 1998; Doğan and Karwe, 2003; Singh et al. 2007). They found that WAI increased with increasing temperature.

The WAI decreased significantly ($P < 0.05$) as the percentage of tomato pomace increased. This may be attributed to relative decrease in starch content with addition of pomace and competition of absorption of water between pomace and available starch. This result is in agreement with those of Artz et al. (1990). They reported a decrease in water holding capacity when the ratio of fiber/corn starch increased in extrusion of corn fiber and corn starch blend. It was concluded that gelatinized corn starch has a much greater water holding capacity than either hemicellulose or cellulose, the major components of corn fiber, thus, any reduction in the gelatinized starch should reduce the water holding capacity of the extrudate (Artz et al. 1990). A

decrease in water absorption capacity of potato fiber with extrusion was observed by Camire and Flint (1991). In addition, Singh et al. (2007) observed a decrease in WAI with addition of pea grits in extrusion of rice. They explained that a decrease in WAI was due to the dilution of starch in rice pea blends. Gujska and Khan (1991) found that addition of hull to the high starch protein fraction of pinto bean flour resulted in a significant decrease of WAI, probably a result of reduction of starch in the starting material. Ahmed (1999) reported that WAI of corn based flax snack decreased as the percent of flaxseed increased. Jin et al. (1995) also reported that increasing fiber content from 0-20% resulted in products with low WAI. However, Chang et al. (1998) indicated that as the concentration of jatobá flour increased, the WAI continuously increased at higher moisture contents. They explained that a moderate extrusion treatment disrupts structures and therefore creates pores that water can penetrate. Increasing the grape pomace level resulted in decrease in WAI of barley-grape pomace extrudates similar to that of barley-tomato pomace extrudates. A decrease in WAI may be attributable to increase in fiber content due to pomace addition and resulted in decrease in starch content as mentioned previously. Another possible reason may be because of sugar incorporation from grape pomace. Similar results were reported for extrusion cooking of fermented maize-finger millet blend with different sugars and chestnut-rice flour blend by Onyango et al. (2004a) and Sacchetti et al. (2004). Onyango et al. (2004a) reported that increasing concentration of sugars, apart from glucose, increased starch disruption and hence reduced the volume occupied by the granule or starch polymer after swelling in excess water.

The effect of pomace level and screw speed on WAI of barley-grape pomace extrudates is shown in Figure 4.19. The interaction effect of grape pomace level and screw speed was significant ($P < 0.05$) but temperature had no significant influence on WAI of barley-grape pomace extrudates. Increasing screw speed at low grape pomace level decreased WAI of barley-grape pomace extrudates but at high pomace level, an increase in screw speed led to rise in WAI. This indicated that the effect of screw speed was dependent on grape pomace level. Hashimoto and Grossmann (2003) found that WAI of cassava bran/cassava starch extrudates decreased and then increased when screw speed increased from 120 to 150 and to 180 rpm. They explained this phenomenon such that there might have been some starch degradation and the further increase at higher shear conditions is probably due to the structural

modification of fiber. Guha et al. (1997) reported that samples extruded at lower screw speed showed relatively high WAI values than at higher screw speeds. This was attributed to the high residence time at low screw speed permitting enhanced extent of cooking. Jin et al. (1995) stated that at low shear rate (low screw speed), there were more undamaged polymer chains and a greater availability of hydrophilic groups which could bind more water molecules and resulted in higher values of WAI. Mezreb et al. (2003) observed increasing WAI of wheat extrudates when the screw speed was changed from 200 to 300 rpm but decreased at 500 rpm. The authors accounted WAI as the phenomena of gelatinization and molecules melting and explained by low residence time allowing sufficient gelatinization at 500 rpm for reduction in WAI at high screw speed. Badrie and Mellowes (1991) also observed the lower WAI of cassava extrudate on increasing screw speed from 425 to 520 rpm and the higher WAI of cassava extrudate on further increase of screw speed from 520 to 560 rpm. They attributed to increase in shear rate resulting in structural modification for lower value of WAI and to shorter residence time for higher value of WAI with increasing screw speed as opposed to Mezreb et al. (2003).

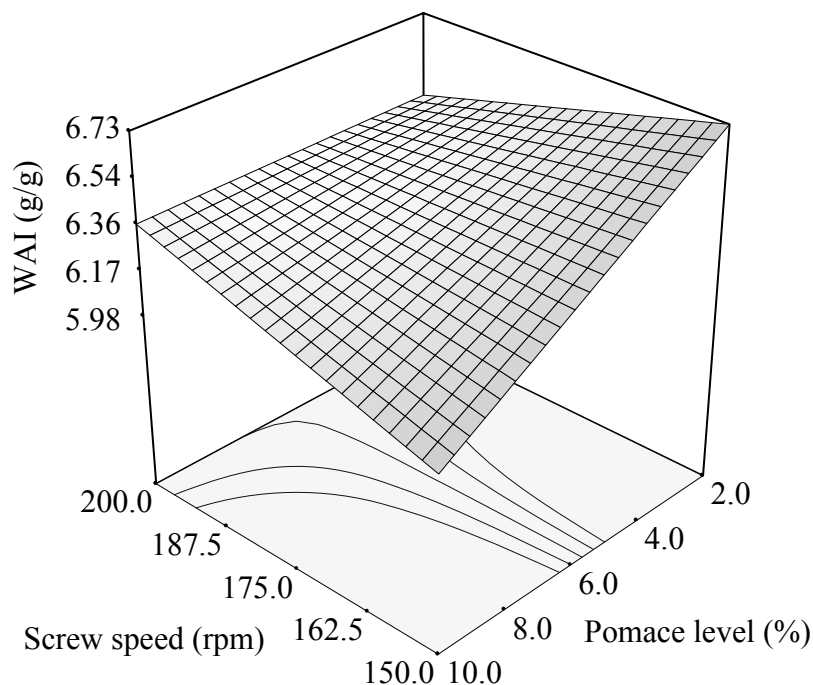


Figure 4.19 Response surface plot for the effect of pomace level and screw speed on water absorption index of barley-grape pomace extrudates at a temperature of 150°C

4.3.2 Water solubility index

The relationship between WSI and independent variables obtained from regression analysis is expressed in Table 4.5. The significance of models of WSI of barley, barley-tomato pomace and barley-grape pomace extrudates is given in Table A.7. The lack-of-fit was not significant ($P>0.05$) for WSI of barley and barley-grape pomace extrudates but significant ($P<0.05$) for barley-tomato pomace extrudates with low coefficient of variation ($CV=7.82\%$). The coefficients of determination of the predicted models for WSI of barley, barley-tomato pomace and barley-grape pomace extrudates were 0.872, 0.831 and 0.865, respectively. The negative linear and quadratic effects of temperature had significant effect ($P<0.05$) on WSI of barley extrudates while linear term of temperature only was significant ($P<0.05$) for WSI of barley-tomato pomace and barley-grape pomace extrudates. The significant positive linear effect of screw speed on WSI was obtained for barley and barley-tomato pomace extrudates, whereas it was not significant for barley-grape pomace extrudates at $P>0.05$. The linear and quadratic terms of pomace level were significantly affected WSI of barley-grape pomace extrudates but the linear term of pomace level was significant for WSI of barley-tomato pomace extrudates only ($P<0.05$).

Water solubility gives information about degradation while water absorption is more related to the swelling capability of granules. The values of WSI varied from 6.27 to 9.67% for barley extrudates while it ranged from 7.08 to 12.99 and 7.85 to 15.79 % for barley-tomato pomace and barley-grape pomace extrudates, respectively. WSI of barley extrudates first increased with increase in temperature up to a certain point (150°C) and then decreased (Figure 4.20). Similar behavior was observed for WSI of yam extrudates by Sebio and Chang (2000). However, WSI of barley-tomato pomace and barley-grape pomace extrudates decreased with increasing temperature which is in agreement with work of Gutkoski and El-Dash (1999) in extruded oat products. An increase in the amount of dextrinized starch during extrusion cooking may result in an increase in WSI as reported by Doğan and Karwe (2003). However, molecular interactions between degraded starch, protein and lipid components, which in turn lead to an increase in molecular weight, may decrease the solubility, thus WSI (Doğan and Karwe, 2003). An increase in WSI with increasing temperature was reported by other authors (Gujral et al. 2001; Yuliani et al. 2006; Hagenimana et al.

2006; Singh et al. 2007) which opposes with our findings. Hagenimana et al. (2006) reported that the combination of harsh conditions and low moisture contents caused an increase in the amount of degraded starch granules resulting in an increased formation of water-soluble products.

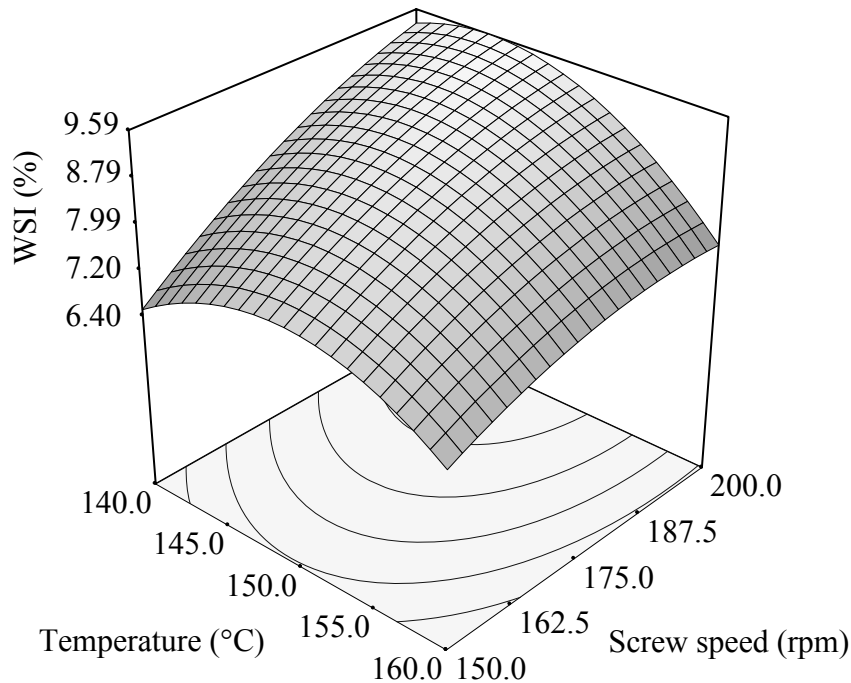


Figure 4.20 Response surface plot for the effect of screw speed and temperature on water solubility index of barley extrudates

The effect of temperature and screw speed on WSI of barley-tomato pomace extrudates is shown in Figure 4.21. The WSI increased significantly ($P < 0.05$) with increasing screw speed for both barley and barley-tomato pomace extrudates (Figures 4.20 and 4.21) but not significantly affected WSI of barley-grape pomace extrudates. The increase in WSI with increasing screw speed was consistent with the results reported by other authors (Jin et al. 1995; Guha et al. 1997; Sebio and Chang, 2000; Gujral et al. 2001; Doğan and Karwe, 2003; Mezreb et al. 2003). Jin et al. (1995) obtained higher WSI when screw speed increased from 150 to 350 rpm for corn meal extrudates. Gujral et al. (2001) found similar effects of screw speed for WSI of sweet corn and flint corn grits extrudates in the range of 100 to 150 rpm. The solubility of yam flour increased with screw speed, from 40 to 280 rpm, as reported by Sebio and Chang (2000). Jin et al. (1995) reported that WSI depends on the quantity of soluble molecules which is related to the degradation. Lee et al. (1999) also stated that water solubility usually increases when starch chains degrade into

smaller fragments. Guha et al. (1997) reported a higher screw speed resulted in more fragmentation than a lower screw speed and thus increased WSI of rice extrudates. The higher WSI of extrudate with increasing screw speed may be related to increasing SME input with screw speed. Higher SME input causes greater restriction to material flow resulting in breakdown of polymers to small molecules with higher solubility (Choudhury and Gautam, 1998). Smith (1992) observed the increase in WSI with decreasing molecular weight or that the molecular weight falls with increasing SME. Mezreb et al. (2003) found that the WSI increased as screw speed increased from 200 to 300 rpm for wheat extrudates and from 300 to 500 rpm for corn extrudates. The authors reported that the increase of screw speed induced a sharp increase of specific mechanical energy, the high mechanical shear degraded macromolecules, and so the molecular weight of starch granules decreased and hence increased WSI. As the water solubility increases there is a linear decrease in intrinsic viscosity which reflects a decrease in the average molecular weight of the amylose and amylopectin chains (Kirby et al. 1988). With the agreement of other authors, Kirby et al. (1988) found also the greater water solubility with large mechanical energy inputs. These results can be confirmed by correlation between WSI and SME ($R=0.804$, $R=0.782$, $P<0.05$) for barley and barley-tomato pomace extrudates (Tables A.2 and A.3). Similar correlation was also reported by Kirby et al. (1988) and Choudhury and Gautam (1998).

The effect of pomace level and temperature on WSI of extrudates from barley-grape pomace and barley-tomato pomace are illustrated in Figures 4.22 and 4.23. Increasing level of pomace raised WSI of barley-tomato pomace and barley-grape pomace extrudates. This may be related to the modification of fiber, coming from pomace, due to extrusion and causes increase in WSI. It was reported that starch and/or fiber degradation was sufficient to increase in solubility (Hashimoto and Grossmann, 2003). Larrea et al. (2005b) extruded orange pulp for using source of fiber in the preparation of biscuit-type cookies. They observed that WSI of extruded pulp increased by extrusion. It could be attributed to the presence of compounds having a low molecular weight (Larrea et al. 2005b) and therefore, the presence of soluble material in pomace may cause increase in WSI of extrudates.

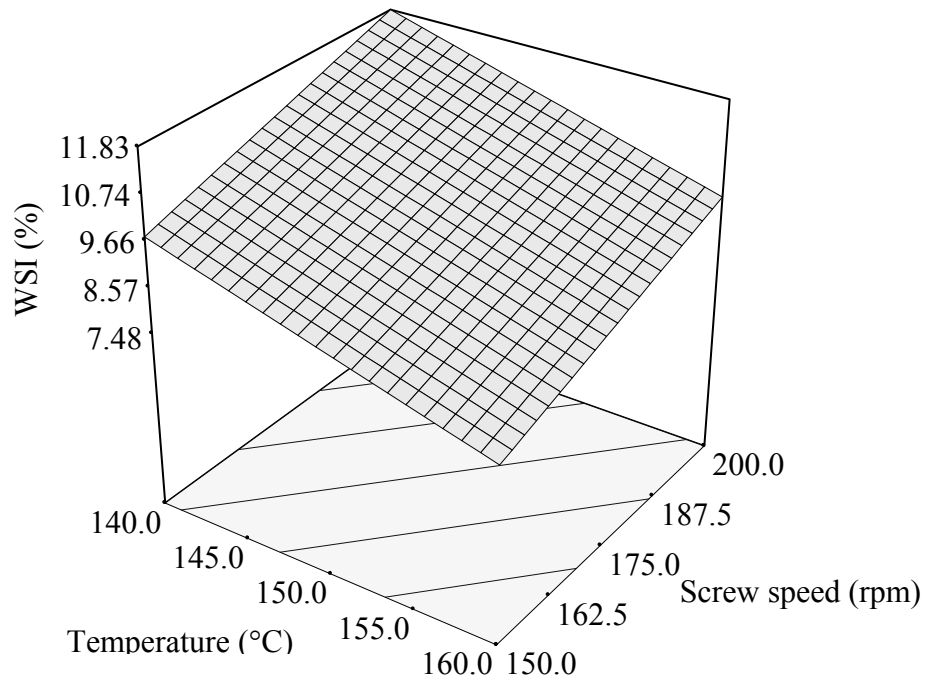


Figure 4.21 Response surface plot for the effect of screw speed and temperature on water solubility index of barley-tomato pomace extrudates at a pomace level of 6%

Jin et al. (1995) observed an increase in WSI of corn meal extrudates as fiber content increased from 0 to 20%. In contrast to these findings, Hashimoto and Grossmann (2003) found a decrease in WSI when bran content was increased. The highest value of WSI of barley-grape pomace extrudates was found with the extrudates containing highest level of grape pomace (12.7%). This result may be related to increased quantity of sugar in the system. Because, in addition to fiber, sugar content of barley-grape pomace extrudates increases as grape pomace level increased. Sacchetti et al. (2004) found increased WSI as percentage of chestnut flour was increased. Onyango et al. (2004a) suggested that an increase in WSI with increasing sugar concentration was a sign of increased solubilization of starch. Mezreb et al. (2006) also reported that adding sucrose up to 10% increased the quantity of water-soluble fractions and resulted in an increase in WSI for wheat extrudates. On the other hand, Jin et al. (1995) found that increasing sugar content decreased WSI of corn meal extrudates including soy fiber.

WSI of barley extrudates was correlated with SEI ($R=0.620$, $P<0.05$) (Table A.2). Badrie and Mellowes (1991) found similar correlation for cassava extrudates. Colonna et al. (1989) reported that water solubility of starch increases with expansion. WSI of barley-tomato pomace and barley-grape pomace extrudates was positively correlated with bulk density ($R=0.542$, $R=0.478$, $P<0.05$). Jin et al. (1995) observed correlation between bulk density and functional properties (WAI and WSI) ($r=0.99$, $P<0.001$). They suggested that WAI and WSI are related to the expansion of extrudates and higher expansion had a higher WSI. Rayas-Duarte et al. (1998) reported that the higher values of expansion index were associated with higher water absorptions. It was observed that WSI of barley-tomato pomace extrudates was negatively correlated with die melt temperature (Table A.3).

Gutkoski and El-Dash (1999) found temperature being inversely proportional to WSI in extruded oat products. An increase in WSI coincides with the decrease in WAI for barley-grape pomace extrudates, which was proved by a negative correlation between WSI and WAI ($R= -0.511$, $P<0.05$) (Table A.4). This agrees well with the results of Badrie and Mellowes (1991) where they found a negative correlation between WSI and WAI for cassava extrudates. Iwe (1998) studied effects of extrusion process on functional properties of mixtures of full-fat soybean and sweet potato. He found that the water solubility indices of samples decreased as water absorption index increased. Van der Burgt et al. (1996) reported the same relationship between WAI and WSI. They plotted WSI against the WAI and saw continuous decrease of WSI with an increase in WAI. Kirby et al. (1988) also observed strong correlation between two indices for maize grits extrudates in addition to the other authors.

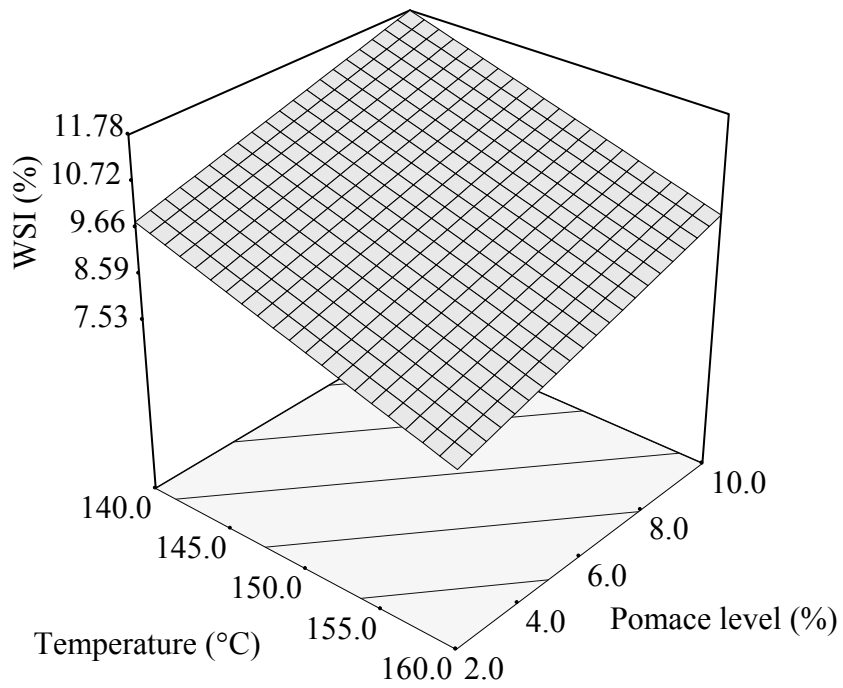


Figure 4.22 Response surface plot for the effect of pomace level and temperature on water solubility index of barley-tomato pomace extrudates at a screw speed of 175 rpm

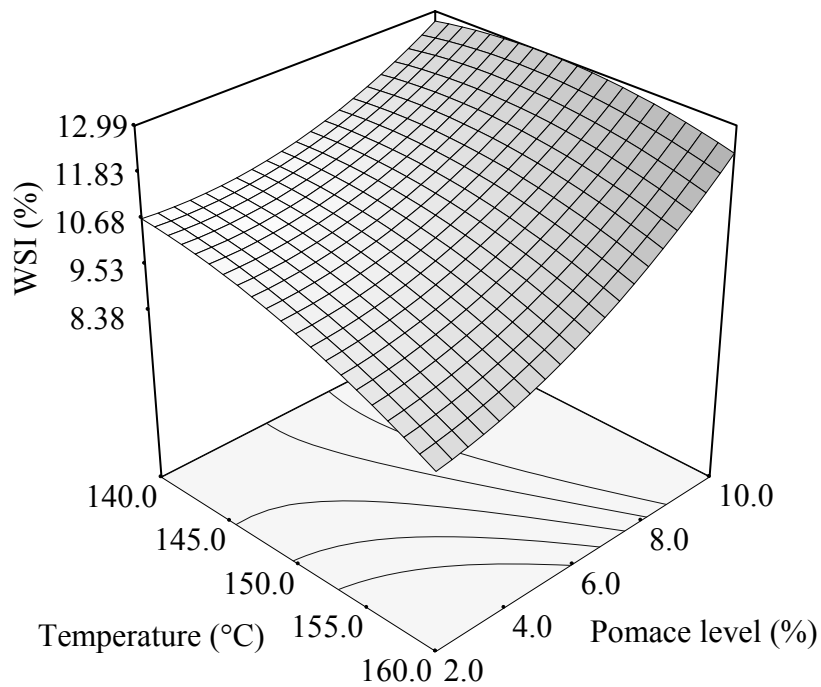


Figure 4.23 Response surface plot for the effect of pomace level and temperature on water solubility index of barley-grape pomace extrudates at a screw speed of 175 rpm

4.4 Textural Properties

4.4.1 Peak force (Hardness)

The textural property of extrudate was determined by measuring the force required to break the extrudate (Singh et al. 1994). The higher the value of maximum peak force required in gram, which means the more force requires to breakdown the sample, the higher the hardness of the sample to fracture (Li et al. 2005). The regression equations of hardness measured as peak force for barley, barley-tomato pomace and barley-grape pomace extrudates determined in terms of coded variables are given in Table 4.6. ANOVA results for quadratic models of peak force are shown in Table A.8. Acceptable coefficient of determination values ($R^2=0.984$, $R^2=0.988$, $R^2=0.967$) were obtained for significant models of peak force for barley, barley-tomato pomace and barley-grape pomace extrudates with significant lack-of-fit variation. On the basis of analysis of variance, the selected models adequately represented the data for peak force. Peak force of extrudates was significantly ($P<0.05$) dependent on linear and quadratic terms of temperature (Table 4.6). Screw speed had no significant ($P>0.05$) effect on peak force of barley and barley-grape pomace extrudates but it was significantly ($P<0.05$) affected those of barley-tomato pomace extrudates. The interaction term of temperature and tomato pomace level was found to be significant ($P<0.05$) on peak force but the linear and quadratic terms of tomato pomace level have no significant effect ($P>0.05$) on peak force of barley-tomato pomace extrudates. The quadratic effect of grape pomace was significant ($P<0.05$), whereas linear effect was not significant ($P>0.05$) for peak force of barley-grape pomace extrudates.

Table 4.6 The regression models for textural parameters using independent variables temperature (X_1), screw speed (X_2) and pomace level (X_3) of barley and barley-pomace extrudates

Response	Model	R^2
PF _B	$8.17 - 8.74X_1 + 5.16X_1^2$	0.984
PF _T	$7.70 - 5.79X_1 - 0.67X_2 + 4.13X_1^2 + 1.93X_1X_3$	0.988
PF _G	$6.72 - 7.40X_1 + 3.78X_1^2 + 1.47X_3^2$	0.967
S _B	$8.52 - 4.85X_1 + 1.30X_1^2$	0.979
S _T	$8.00 - 3.54X_1 - 0.47X_2 + 1.35X_1^2 + 0.55X_2^2 + 0.86X_1X_3$	0.974
S _G	$7.73 - 4.50X_1 + 1.21X_3 + 1.56X_1^2 + 0.89X_3^2$	0.955
D _{iB}	$0.83 - 0.084X_1 + 0.21X_1^2$	0.846
D _{iT}	$0.93 + 0.096X_1^2 + 0.039X_1X_3$	0.899
D _{iG}	$0.77 - 0.032X_1 - 0.11X_3 + 0.069X_1^2 + 0.051X_3^2$	0.947

B: barley flour, T: tomato pomace, G: grape pomace

The influence of operating conditions on the changes in peak force of barley extrudates is shown in Figure 4.24. The peak force measured as hardness of barley extrudates varied between 5.0 and 29.2 while ranged from 5.6 to 29.8 and 4.6 to 27.2 N for barley-tomato pomace and barley-grape pomace extrudates, respectively. A decrease in product hardness with increasing temperature was observed for barley and barley-pomace extrudates. For example; a decrease in die temperature increased the product hardness yielding a maximum at about 133°C, 175 rpm screw speed and 6% tomato pomace level. This result is in line with bulk density where an increase in density was observed at these conditions. This is in agreement with the results of other researchers. Sebio and Chang (2000) obtained low hardness of yam flour extrudates with the samples extruded at higher barrel temperatures. Lee et al. (1999) reported that breaking stress increased as barrel temperature decreased for corn starch extrudates. Doğan and Karwe (2003) found increased hardness of quinoa extrudates as die temperature decreased. Yuliani et al. (2006) reported that increasing the temperature resulted in a decrease in hardness of extrudates from mixtures of starch and D-limonene. Yao et al. (2006) found that extrusion at higher temperatures resulted in cereal with less hardness for extrudates from two oat lines. Mendonça et al. (2000) obtained lower hardness at high temperatures for corn meal extrudates including corn bran. Similarly, Ryu and Walker (1995) observed that breaking strength and bulk density decreased over the temperature range of 140 to 160°C in extrusion cooking of wheat flour. Lee et al. (1999) found that breaking stress increased as barrel temperature decreased and a negative correlation with expansion ratio, indicating extrudates became brittle when extrusion was performed at high temperatures. Ding et al. (2005; 2006) reported that increasing temperature would decrease melt viscosity, but it also increases the vapor pressure of water. This favors the bubble growth which is the driving force for expansion that produces low density products and thus decreasing hardness of extrudate. Yuliani et al. (2006) also reported that an increase in temperature would decrease the melt viscosity causing more bubble growth and decreased the bubble wall thickness although some bubbles may collapse and fracture. This resulted in a lower bulk density and hence lower hardness of extrudates. The extrudate with the higher density would have relatively thicker cell walls and an overall lower porosity (Barrett et al. 1994), which is directly related with hardness of samples. Agbisit et al. (2007) observed increase in cell diameter and decrease in cell number density with higher overall expansion. The

authors found high negative correlations between mechanical properties and average cell diameter. Sacchetti et al. (2005) reported correlation between hardness and density of extruded cereal blend. These findings agree with the results of current research, as we found positive correlation ($R=0.984$, $R=0.925$, $R=0.859$, $P<0.01$) between hardness and bulk density for barley, barley-tomato pomace and barley-grape pomace extrudates, respectively (Tables A.2, A.3 and A.4). High density product naturally offers high hardness evident by high correlation between product density and hardness. Rayas-Duarte et al. (1998) found a negative correlation between expansion index and breaking strength which means that decreased breaking strength of extrudates was associated with high expansion index and low bulk density. Veronica et al. (2006) observed negative relationship among expansion ratio, hardness and breaking strength for puffed snacks from maize-soybean mixture. They found increased breaking strength and hardness values when expansion decreased.

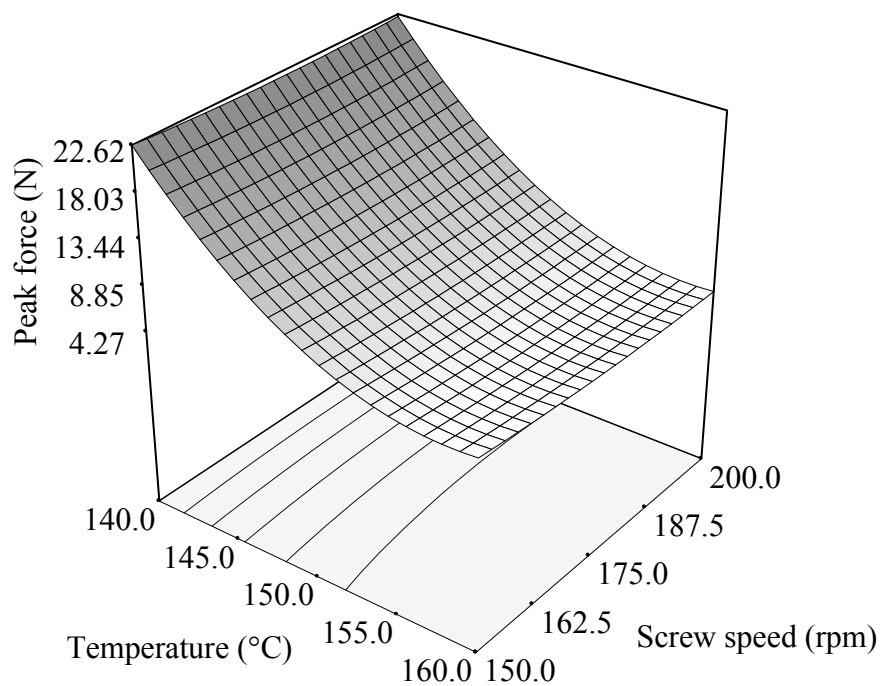


Figure 4.24 Response surface plot for the effect of screw speed and temperature on peak force of barley extrudates

Increasing screw speed slightly decreased hardness of the barley flour-tomato pomace extrudate, particularly at higher temperatures (Figure B.16). Liu et al. (2000) found that the hardness of the extruded oat-corn flour increased as the screw speed decreased. They explained that a higher screw speed caused increase in product temperature, which usually leads to a higher expansion and decrease in hardness. Wu

et al. (2007) observed a decrease in hardness of extrudate from flaxseed-corn meal blend with increasing screw speed from 200 to 400 rpm. Increasing screw speed would increase SME which have positive influence on expansion index and would cause reduction in hardness. Ryu and Ng (2001) found that with the increase in the SME input, apparent elastic moduli and breaking strength in bending and breaking strength in compression were decreased for wheat flour and whole cornmeal extrudates. They concluded that extrudates puffed at higher SME input could have a softer, more brittle and crispier texture since the apparent elastic modulus and breaking strength were relatively lower than those of lower SME input. Ding et al. (2006) found decreased hardness of wheat extrudate with increasing screw speed especially at higher barrel temperatures. They attributed this to lower melt viscosity of the mix obtained due to increasing screw speed and resulted in a less dense, softer extrudate.

The effect of temperature and pomace level on peak force of barley-tomato pomace and barley-grape pomace extrudates is shown in Figures 4.25 and 4.26. The changes on peak force as a function of tomato pomace level were dependent on temperature. Response surface plot showed that a decrease in die temperature with increasing level of tomato pomace increased the product hardness, whereas lower product hardness was observed at higher temperatures. On the other hand, the effect of grape pomace on peak force of barley-grape pomace extrudates was not dependent on temperature. Increasing grape pomace level resulted in increase in peak force of extrudates with respect to quadratic effect (Figure 4.26). An increase in percentage of grape pomace caused increase of bulk density of extrudates as a result of fiber or sugar coming from pomace. Therefore, it was expected an increase in peak force or hardness of extrudates with increasing level of grape pomace. Onwulata et al. (2001b) found reduced expansion and breaking strength by inclusion of fiber at 125 g/kg. They reported that reduced expansion and increased breaking strength are characteristics of fiber substituted products resulting from reduced elasticity due to presence of fiber. Liu et al. (2000) observed that extrudate with higher oat flour contents had a higher hardness than extrudate with a lower oat flour content possibly due to increasing fiber level with oat. Hsieh et al. (1989) reported that increasing fiber content in corn meal decreased the radial expansion and increased the bulk density and breaking force for the extrudates. Yanniotis et al. (2007) found increased

hardness and decreased porosity while adding more fibers. The change of hardness with fiber is related to the effect of these materials on the cell wall thickness. Fiber causes less porous matrix, the thicker the cell wall and the harder the extrudate (Yanniotis et al. 2007). Ahmed (1999) obtained increased bulk density and breaking strength of extrudates over the flax addition range 5 to 20%. Jin et al. (1995) also reported that the breaking strength increased with increasing sugar and soy fiber contents. They attributed to a more compact extrudate with thick cell walls and small air cell size by incorporation of sugar and fiber into corn meal. The authors found a negative correlation between breaking strength and radial expansion of extrudates. The incorporation of fiber in extruded product results in increased density and hardness of extrudates as reported by Onwulata et al. (2000).

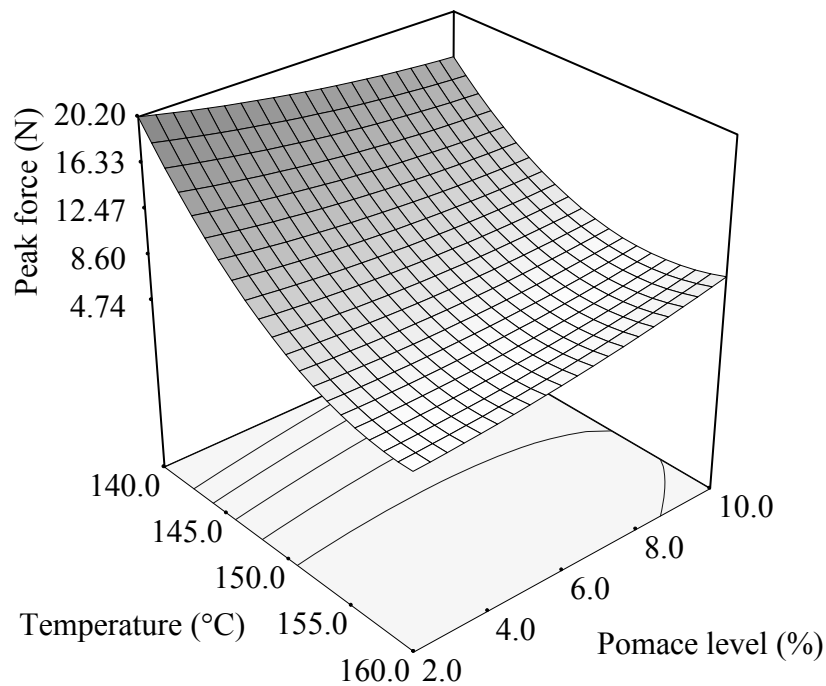


Figure 4.25 Response surface plot for the effect of pomace level and temperature on peak force of barley-tomato pomace extrudates at a screw speed of 175 rpm

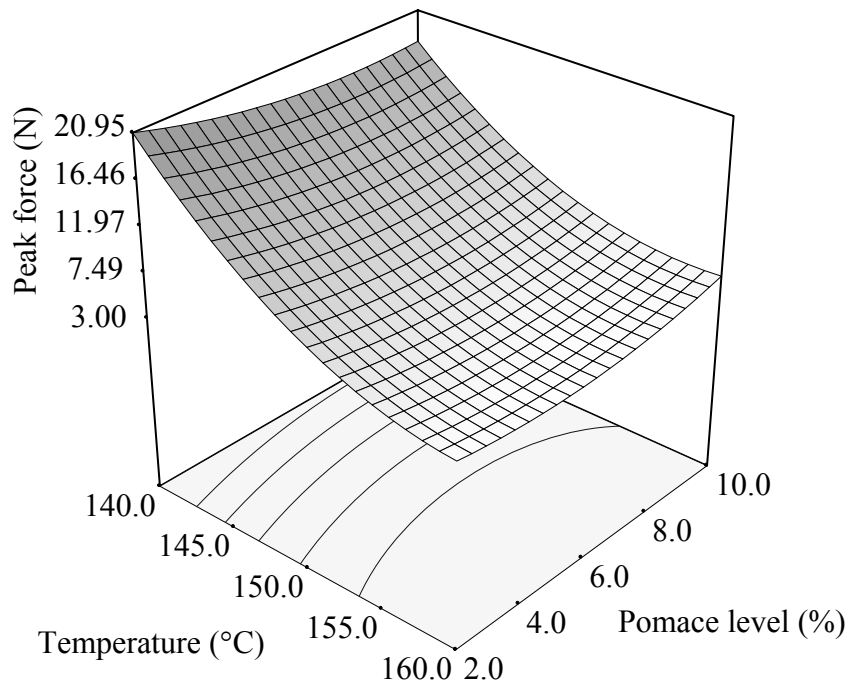


Figure 4.26 Response surface plot for the effect of pomace level and temperature on peak force of barley-grape pomace extrudates at a screw speed of 175 rpm

4.4.2 Slope (Crispness)

Crispness is associated with a low-density cellular structure that is brittle and generates a high-pitched noise when fractured (Le Meste et al. 2002). Vincent (1998) reported that crispness may be associated with a rapid drop in force which is associated with rapid propagation of fracture, which, in turn, necessitates that material is brittle. The slope of force-distance curve before the first major fracturability peak was measured as the crispness of the extrudates (Jackson et al. 1996). The lower the slope, the crisper the product is considered. Multiple regression equations for slope of barley, barley-tomato pomace and barley-grape pomace extrudates in terms of coded variables are given in Table 4.6. ANOVA results for models are summarized in Table A.8. The lack-of-fit was significant ($P < 0.05$) for the quadratic models of slope for barley and barley-grape pomace extrudates but not significant ($P > 0.05$) for barley-tomato pomace extrudates. However, coefficients of determination (R^2) of the predicted models for slope of barley, barley-tomato pomace and barley-grape pomace extrudates were 0.979, 0.974 and 0.955, respectively. The linear and quadratic effects of temperature were found to be significant ($P < 0.05$) for slope of barley extrudates and barley-pomace extrudates. Screw speed was significant ($P < 0.05$) as both linear and quadratic for slope but it did not affect

significantly ($P>0.05$) slope of barley extrudates and barley-grape pomace extrudates. The slope of barley-tomato pomace was significantly ($P<0.05$) influenced by interaction of temperature and tomato pomace level while the slope of barley-grape pomace extrudates was significantly ($P<0.05$) affected by grape pomace level with linear and quadratic terms.

The values of slope measured as crispness varied between 5.0 to 16.0 N/mm for barley extrudates while it was in the range 5.5-17.1 and 4.3-19.6 N/mm for barley-tomato pomace and barley-grape pomace extrudates, respectively. The effect of temperature and screw speed on slope of barley extrudates is given in Figure 4.27. The slope appeared to decrease with increasing temperature meant increase in crispness of barley extrudates. This trend was similar for both barley-tomato pomace and barley-grape pomace extrudates (Figures 4.28 and 4.29). The hardness and crispness of expanded extrudate is a perception of the human being and is associated with the expansion and cell structure of the product (Ding et al. 2005). It was reported that the parameters controlling the mechanical properties of cellular material such as density, cell wall thickness, cell size and cell number are expected to predict the product crispness (Hutchinson et al. 1989; Guraya and Toledo, 1996; Roudaut et al. 2002). In this study, increasing temperature decreased peak force and bulk density of extrudates. It is noted that when progressive increase in temperatures resulted in pores in the structure due to formation of air cells and the surface appeared flaky and porous and hence decreased hardness (Bhattacharya and Choudhury, 1994). Duizer and Winger (2006) reported that less force required in breaking a product that is very crisp. Therefore, it was expected that crispness of extrudates to increase with increasing temperature because it is related to the cellular structure of extrudates and thus bulk density and hardness. This can also be supported by correlation between slope and bulk density and peak force. Positive correlation was found between slope and bulk density ($R=0.983$, $P<0.01$; $R=0.984$, $P<0.05$; $R=0.943$, $P<0.01$) and peak force ($R=0.961$, $R=0.949$, $R=0.919$, $P<0.01$) for barley, barley-tomato pomace and barley-grape pomace extrudates, respectively. It would be expected that high density and high hardness would give high slope and thus lower crispness of extrudates. Ding et al. (2005) suggested that increasing temperature would decrease melt viscosity, which favors the bubble growth and produce low density products with small and thin cells, thus increasing the crispness of extrudate. Rayas-Duarte et al.

(1998) also stated that low breaking strength values are usually related to a large number of small cells per unit area with thinner cell walls, resulting in a crispy texture. Agbisit et al. (2007) found that both average crushing force and crispness work had marked to high negative correlations with cell diameter. Both crushing force and crispness work increased with a decrease in cell diameter, indicating that more force and work needed to deform/fracture smaller-size cells.

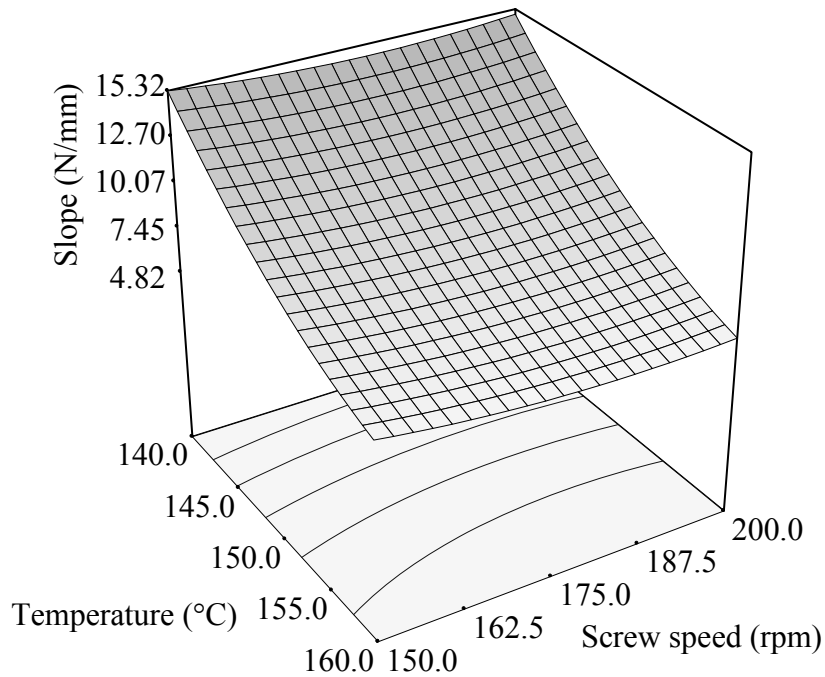


Figure 4.27 Response surface plot for the effect of screw speed and temperature on slope of barley extrudates

Increasing screw speed decreased the slope and therefore increased crispness of barley-tomato pomace extrudates (Figure 4.28). This can be attributed to decrease in extrudate hardness when screw speed increased. Low value of crispness for barley-tomato pomace extrudates was obtained by increasing temperature with decreasing pomace level (Figure B.17). The effect of grape pomace level on slope of barley-grape pomace extrudates is presented in Figure 4.29. Increasing grape pomace content in extrusion cooking of barley flour-grape pomace blends increased the slope of force-time curve. This means that highest pomace content causes less crispy extrudate. This may be due to presence of more fiber in the grape pomace. Fiber reduces the cell size, probably by causing premature rupture of gas cells, which reduces the overall expansion and results in less porous structure (Lue et al. 1991; Yanniotis et al. 2007) and therefore less crispy texture. Jin et al. (1995) investigated

structure of corn meal extrudates including soy fiber by using scanning electron microscopy. It was revealed that the air bubbles were smaller and the cell was thicker as increasing fiber level. They also reported that increasing the fiber content resulted in a less expanded, more compact texture in extrudate. Lue et al. (1990) observed increasing dietary fiber content decreased the average cell size and increased the frequency of incomplete flakes and holes on the cell wall. They also concluded that these holes and incomplete flakes were indicative of poor bubble formation during puffing. Moore et al. (1990) reported that the effect of bran on cell expansion and cell structure results from the fact that bran particles reduce extensibility of cell walls and in causing premature rupture of the cell walls, bran would create more broken and small cells.

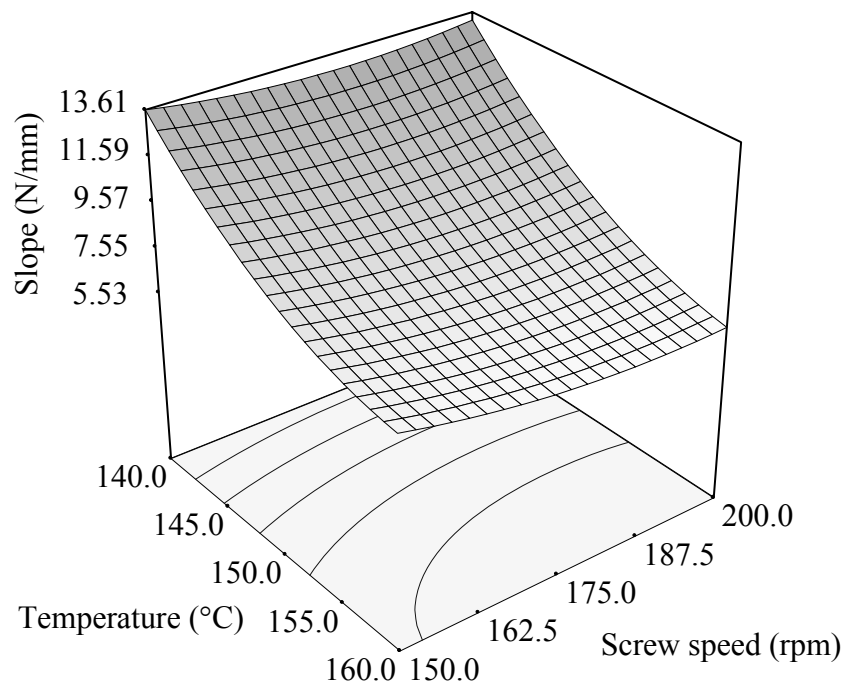


Figure 4.28 Response surface plot for the effect of screw speed and temperature on slope of barley-tomato pomace extrudates at a pomace level of 6%

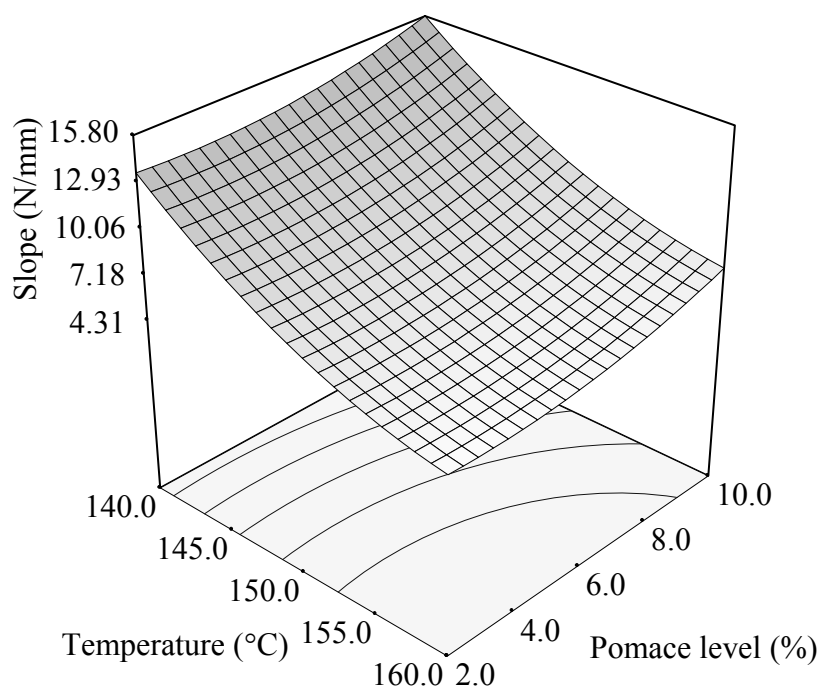


Figure 4.29 Response surface plot for the effect of pomace level and temperature on slope of barley-grape pomace extrudates at a screw speed of 175 rpm

4.4.3 Distance (Brittleness)

The distance that is required in breaking extrudates, measured as brittleness, was determined with the shortest distance being most brittle (Chanvrier et al. 2007). A good general rule is that if a product cracks at a smaller deformation it is more brittle (Texture Technologies (b)). The quadratic models for distance (D), which is a textural attribute, of barley, barley-tomato pomace and barley-grape pomace extrudates in terms of coded levels of the variables are given in Table 4.6. Although the lack-of-fit was significant for models (Table A.8) except for barley-tomato pomace extrudates, the coefficients of variation were found to be in the levels of 10.25, 4.46 and 4.38% for distance model of barley, barley-tomato pomace and barley-grape pomace extrudates, respectively. The coefficients of determination (R^2) of quadratic models for distance were 0.846, 0.899 and 0.947 for barley, barley-tomato pomace and barley-grape pomace extrudates, respectively. Temperature was significantly ($P < 0.05$) affected distance of extrudates. The distance of barley-tomato pomace extrudates was significantly ($P < 0.05$) influenced by the interaction effect of temperature and tomato pomace level. Grape pomace had a significant effect ($P < 0.05$) on the distance of barley-grape pomace extrudates. There was no significant effect ($P < 0.05$) of screw speed on barley and barley-pomace extrudates.

The measured value of distance for barley extrudates was ranged between 0.74 and 1.23 mm while it was in the range of 0.87-1.23 and 0.69-1.06 mm for barley-tomato pomace and barley-grape pomace extrudates, respectively. The effect of screw speed and temperature on distance of barley extrudates is given in Figure 4.30. The brittle behavior associated with crispness was mostly dependent on temperature for barley extrudates but also pomace level for barley-pomace extrudates as well. As temperature increased up to 150°C, the distance was decreased and thus brittleness increased. However, further increase in temperature of 150°C increased distance and therefore decreased brittleness of barley and barley-tomato pomace extrudates (Figure 4.31). On the other hand, this trend was different for barley-grape pomace extrudates and further increase in temperature to 155°C caused increase in distance slightly and this would decrease brittleness (Figure 4.32). Increasing screw speed decreased distance but was not significant ($P>0.05$). The most brittle barley extrudate was obtained at 150°C and screw speed of 210 rpm. It was found that temperature was the main factor affecting expansion as well as bulk density of the extrudates with a greater extent. It would be expected that low bulk density and low hardness value with increased temperature would produce lower distance to break and thus higher brittleness of extrudates. Gambus et al. (1999) obtained lower hardness and higher brittleness of extrudate connected with smaller force and work which confirmed a more delicate structure of extrudate made from different starches. The distance of barley extrudates was negatively correlated with SEI ($R= -0.706$, $P<0.01$) but positively correlated with bulk density ($R=0.677$, $P<0.05$) and peak force ($R=0.753$, $P<0.01$) as well as slope ($R=0.585$, $P<0.05$). Similar correlations between the distance and SEI ($R= -0.553$, $P<0.05$) and peak force ($R=0.478$, $P<0.05$) were observed for barley-tomato pomace extrudates. However, the distance of barley-grape pomace extrudates was correlated with peak force only ($R=0.487$, $P<0.05$). These correlations suggested that the more brittle extrudate, the lower bulk density, the higher expansion, the lower slope and the lower the force required rupturing it.

The effect of pomace level and temperature on distance of barley-tomato pomace and barley-grape pomace extrudates is shown in Figures 4.31 and 4.32. The breaking distance of barley-tomato pomace extrudates was lower at low pomace level with high temperature. The decrease in brittleness (high in distance) for barley-tomato pomace extrudates can be explained by change in the microstructure where cell size

distribution and orientation, which is associated with expansion, bulk density and cell wall properties of extrudates. It was reported that increased fiber produced thicker cell wall, reduced average cell size and decreased expansion, increased density and hardness or breaking strength of extrudates as reported earlier (Hsieh et al. 1989; Jin et al. 1995; Lue et al. 1990; Hsieh et al. 1991; Lue et al. 1991; Yanniotis et al. 2007). Thick cell walls are inherently less fragile and less likely to rupture than thin cell walls (Barrett and Ross, 1990). However, increasing grape pomace level decreased distance of barley-grape pomace extrudates and hence increased brittleness of samples. This is very interesting result because increasing grape pomace level increased bulk density, peak force and slope. Chanvrier et al. (2007) found smaller distance for wholemeal breakfast cereals; that is, they were more brittle. They suggested that this might be due to the high amount of particles (brans, insoluble fibers) in products, which favors the initiation of product fracture. They also observed by light microscopy that the cohesion between the starch matrix and the particles appeared to be weak.

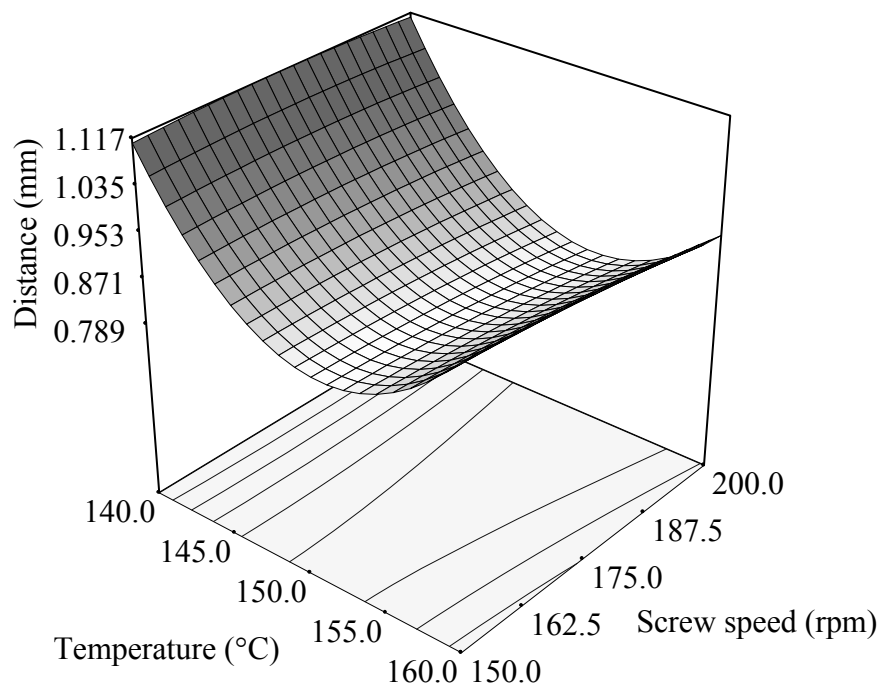


Figure 4.30 Response surface plot for the effect of screw speed and temperature on distance of barley extrudates

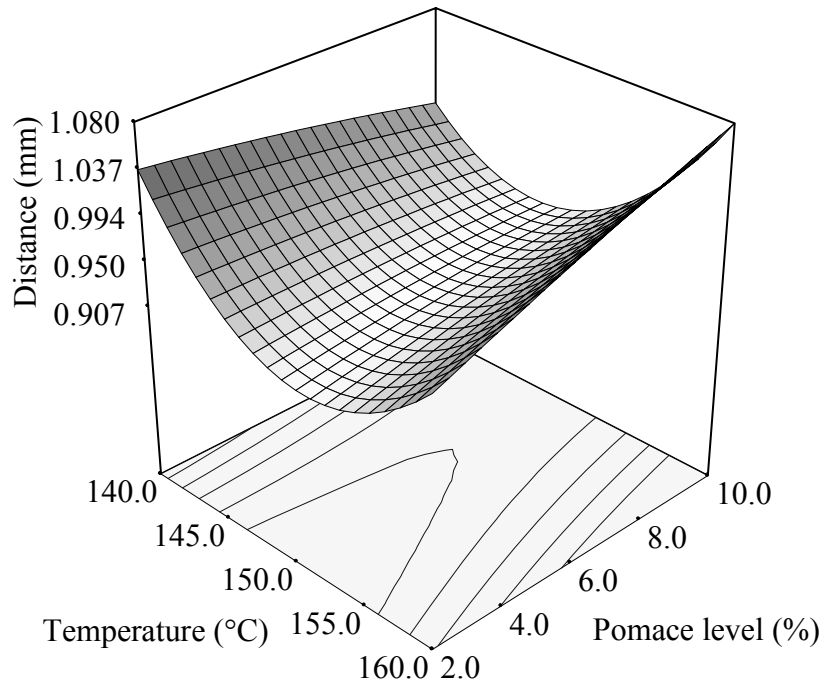


Figure 4.31 Response surface plot for the effect of pomace level and temperature on distance of barley-tomato pomace extrudates at a screw speed of 175 rpm

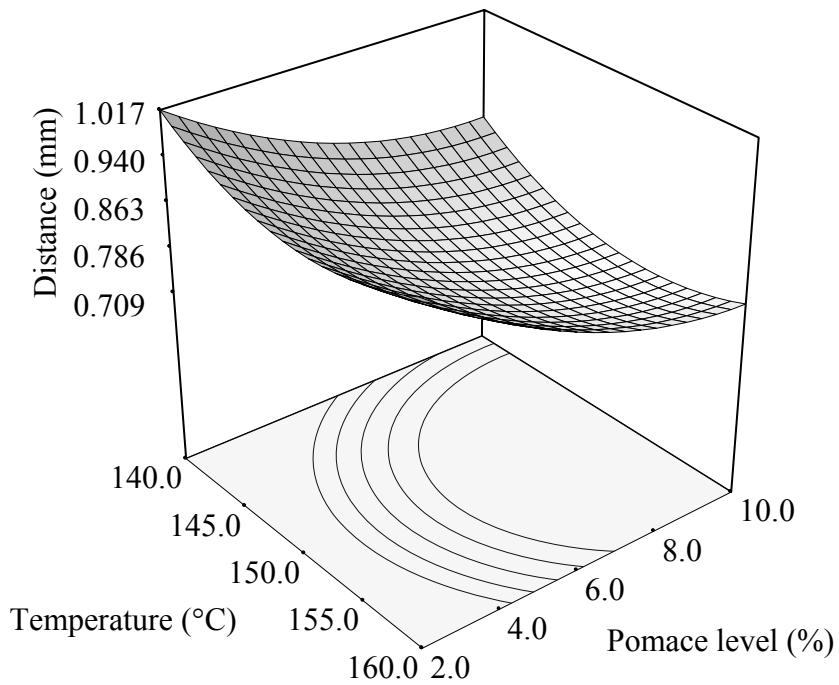


Figure 4.32 Response surface plot for the effect of pomace level and temperature on distance of barley-grape pomace extrudates at a screw speed of 175 rpm

4.5 Sensory Analysis

4.5.1 Barley extrudates

Mean scores for sensory attributes of extrudates are given in Table 4.7. The appearance of extrudates was evaluated in terms of color and porosity. Sensory color of extrudates (A, B and C) produced at 150°C with different screw speeds did not show significant ($P>0.05$) difference and also extrudates (D and E) produced at 160°C and 150, 200 rpm were not significantly different ($P>0.05$). However, significant difference ($P<0.05$) was found between the extrudates produced at 150°C and 160°C (Table 4.7). The highest score (5.20) of porosity was obtained for extrudate D (160°C, 150 rpm). Extrudate D was significantly ($P<0.05$) different from extrudates A, B and C but not significantly ($P>0.05$) different from extrudates E and F. Taste of extrudates was evaluated in terms of flavor and bitterness. There was no significant difference between bran flavor of extrudates as well as bitterness. Mean score for bran flavor was in the range of 3.31 and 3.77. Sensory score for bitterness was ranged from 1.62 to 2.00. The low score showed that panelists did not perceive any bitterness taste in all extrudates. As shown in Table 4.7, low scores of off-odor (1.54-1.88) were observed for all extrudates and they were not significantly ($P>0.05$) different.

Table 4.7 Mean scores for sensory attributes of barley extrudates produced by different extrusion conditions

Sensory attributes	Barley extrudates					
	A ¹	B	C	D	E	F
Appearance						
Color	4.05 ^a	4.45 ^a	4.14 ^a	5.14 ^b	5.37 ^b	5.40 ^b
Porosity	4.34 ^a	4.25 ^a	4.37 ^a	5.20 ^b	4.91 ^{ab}	4.62 ^{ab}
Taste						
Bran flavor	3.31 ^a	3.77 ^a	3.54 ^a	3.45 ^a	3.37 ^a	3.31 ^a
Bitterness	1.97 ^a	1.97 ^a	2.00 ^a	1.62 ^a	1.91 ^a	1.85 ^a
Off-odor	1.80 ^a	1.71 ^a	1.88 ^a	1.60 ^a	1.77 ^a	1.54 ^a
Texture						
Hardness	3.97 ^{ab}	3.86 ^a	4.00 ^{ab}	4.83 ^c	4.75 ^{bc}	4.47 ^{abc}
Crispness	4.22 ^{ab}	3.94 ^a	4.29 ^{abc}	5.44 ^d	4.97 ^{cd}	4.88 ^{bcd}
Brittleness	3.55 ^a	3.27 ^a	3.70 ^a	5.05 ^b	5.08 ^b	5.00 ^b
Overall acceptability	3.50 ^a	3.75 ^a	3.50 ^a	5.69 ^b	5.13 ^b	5.47 ^b

¹Means within a row with different superscripts are significantly different ($P<0.05$).

A: 150°C, 140 rpm; B: 150°C, 175 rpm; C: 150°C, 210 rpm; D: 160°C, 150 rpm; E: 160°C, 200 rpm; F: 164°C, 150 rpm

The texture of extrudates was assessed in hardness, crispness and brittleness. Extrudate D had highest hardness score (4.83) compared to the other extrudates. It was significantly different ($P < 0.05$) from extrudates A, B and C but not significantly ($P > 0.05$) different from extrudates E and F. The higher sensory scores indicated that panelists preferred extrudates D, E and F more compared to extrudates A, B and C with respect to hardness. Sensory hardness was negatively correlated with instrumentally measured peak force ($R = -0.833$) and slope ($R = -0.864$) values at $P < 0.05$ (Table A.9). This means that increase in peak force (hardness) and slope (crispness), from force-distance curve, caused a decrease in preference of panelists in hardness of extrudates. Sensory hardness was positively correlated with porosity ($R = 0.969$, $P < 0.01$). Panelists preferred extrudate D with highest score (5.44) for sensory crispness compared to all the extrudates, whereas it was not significantly ($P > 0.05$) different from extrudates E and F. Instrumentally measured peak force was positively correlated with slope (indication of crispness). There was a negative correlation ($R = -0.817$, $P < 0.05$) between sensory crispness and slope measured as instrumental crispness while a positive correlation ($R = 0.972$, $P < 0.01$) was observed between sensory crispness and sensory hardness. Sensory crispness was also positively correlated ($R = 0.970$, $P < 0.01$) with porosity. The expanded extrudate with low bulk density would have larger cells with thinner cell walls resulting in a crispy texture and hence resulted in low value of crispness force (slope) and high score of sensory crispness. Since the structure of extrudate becomes more open with larger cells, hardness decreases, color becomes brighter and it gets higher sensory preferences. Dogan and Kokini (2007) reported that crispness is associated with cellular foods in the glassy state and structural parameters like porosity, pore size distribution, thickness and the strength of the cell wall and phase behavior are potentially important factors that affect mechanical behavior. Although the differences were not significant ($P > 0.05$) between extrudates D, E and F, extrudate E had highest score (5.08) for sensory brittleness and extrudates A, B and C had lower scores (3.55, 3.27 and 3.70). Extrudates A, B and C were significantly different ($P < 0.05$) in sensory brittleness from extrudates D, E and F (Table 4.7). Sensory brittleness was negatively correlated with peak force ($R = -0.824$, $P < 0.05$) and slope ($R = -0.914$, $P < 0.05$) whereas it was positively correlated with sensory hardness ($R = 0.965$, $P < 0.01$) and sensory crispness ($R = 0.943$, $P < 0.01$) (Table A.9). This means

that when instrumental peak force and slope of extrudates decreased, preference of panelists in sensory brittleness as well as sensory hardness and sensory crispness increased. Sensory brittleness was also positively correlated with porosity ($R=0.883$, $P<0.05$). Extrudate D had highest preference in terms of overall acceptability over other extrudates. Sensory scores for overall acceptability of extrudates A, B and C were significantly different ($P<0.05$) from extrudates D, E and F (Table 4.7).

Overall acceptability of extrudates was positively correlated with sensory hardness ($R=0.931$, $P<0.01$), sensory crispness ($R=0.924$, $P<0.01$), sensory brittleness ($R=0.952$, $P<0.01$), sensory color ($R=0.948$, $P<0.01$) and porosity ($R=0.877$, $P<0.05$) but negatively correlated with slope ($R= -0.931$, $P<0.01$). These correlation results confirmed that all sensory attributes would affect the overall acceptability depending on texture and appearance.

4.5.2 Barley-tomato pomace extrudates

The mean values of sensory panel ratings of barley-tomato pomace extrudates are presented in Table 4.8. Extrudates with different level of tomato pomace had better score than that of extrudate with 0% pomace. Extrudate D with 10% tomato pomace had the highest level of acceptance for color which was significantly ($P<0.05$) different from other extrudates, whereas it received low score for porosity. A negative correlation was observed between porosity and distance from force-distance curve ($R= -0.907$, $P<0.05$) (Table A.10). The higher the porosity of samples would be expected to give the lower the breaking distance of extrudates. Vincent (1998) suggested that if the material is brittle, the fracture will travel quickly, resulting in sudden unloading of the muscles; this is seen as a sudden drop in load on a force-deflection curve. High sensory score for porosity was perceived by extrudate A from barley flour but not significant ($P>0.05$) than that of extrudates B, C and E. There were no significant differences ($P>0.05$) in bran flavor, bitterness and off-odor scores among extrudates. Tomato flavor score changed with changing percentage of tomato pomace in extrudates. However, tomato flavor was perceived as weak (3.02) by panelists for highest level of pomace. No bitter taste and off-odor were detected by panelists for barley-tomato pomace extrudates.

Extrudates B and D from blends of barley flour- tomato pomace at 2 and 10% tomato pomace had higher preference for texture evaluated as hardness, crispness and brittleness while extrudate E had lower score due to possibly having high level of tomato pomace level (12.7%). Sensory crispness was negatively correlated with slope measured instrumentally ($R = -0.902$, $P < 0.05$) but positively correlated with sensory hardness ($R = 0.994$, $P < 0.01$) and brittleness ($R = 0.978$, $P < 0.01$). Sensory hardness showed a positive correlation with sensory brittleness ($R = 0.993$, $P < 0.01$). An increase in slope, that means less crispy in texture, caused decrease in preference of sensory crispness as expected. Increasing in preference of sensory hardness and brittleness increased preference in sensory crispness. Crispness might be associated with a rapid drop in force which is associated with rapid propagation of fracture which, in turn, necessitates that the material is brittle (Vincent, 1998). The extrudate with the higher density would have relatively thicker cell walls and an overall lower porosity. It should offer a greater resistance to break and lead to higher peak force, slope and breaking distance. The overall acceptability of the barley-tomato pomace extrudate was the lowest (3.94) in extrudate A and highest (5.23) in extrudate D. Overall acceptability was positively correlated with sensory hardness ($R = 0.884$, $P < 0.05$), brittleness ($R = 0.916$, $P < 0.05$) and color ($R = 0.959$, $P < 0.05$).

Table 4.8 Mean scores for sensory attributes of barley-tomato pomace extrudates produced by different extrusion conditions

Sensory attributes	Barley-tomato pomace extrudates				
	A ¹	B	C	D	E
Appearance					
Color	3.64 ^a	4.70 ^{bc}	4.85 ^{bc}	5.08 ^d	4.23 ^{ab}
Porosity	4.76 ^a	4.23 ^{ab}	4.61 ^{ab}	3.82 ^c	4.50 ^{ab}
Taste					
Bran flavor	3.67 ^a	3.79 ^a	3.23 ^a	3.44 ^a	3.82 ^a
Bitterness	1.85 ^a	1.61 ^a	1.94 ^a	1.88 ^a	2.00 ^a
Tomato flavor	1.67 ^a	1.50 ^a	2.70 ^b	2.50 ^b	3.02 ^b
Off-odor	1.79 ^a	1.79 ^a	1.70 ^a	1.58 ^a	1.88 ^a
Texture					
Hardness	3.91 ^a	5.32 ^b	4.58 ^c	5.26 ^b	3.44 ^a
Crispness	4.23 ^a	5.17 ^c	4.61 ^{bc}	5.00 ^c	3.82 ^a
Brittleness	4.03 ^a	4.94 ^c	4.55 ^{bc}	5.08 ^c	3.70 ^a
Overall acceptability	3.94 ^a	4.85 ^b	4.94 ^b	5.23 ^b	4.08 ^a

¹Means within a row with different superscripts are significantly different ($P < 0.05$).

A: 0% pomace level, 150°C, 175 rpm; B: 2% pomace level, 160°C, 200 rpm; C: 6% pomace level, 150°C, 217 rpm; D: 10% pomace level, 160°C, 200 rpm; E: 12.7% pomace level, 150°C, 175 rpm

4.5.3 Barley-grape pomace extrudates

The mean scores of sensory attributes for selected extrudates are shown in Table 4.9. Appearance of extrudates was evaluated in terms of color and porosity by panelists. Sensory scores for color decreased as grape pomace level was increased. Extrudate E had the lowest score (2.25) when compared to the other extrudates. The highest score (5.12) of color was obtained for extrudate B with 2% grape pomace level. Color of extrudate E was significantly ($P<0.05$) different from those of extrudates A, B, C and D. It is obvious that extrudate E received lower sensory score for porosity due to highest level of grape pomace. Porosity was positively correlated with sensory color (Table A.11). Porosity is directly related with the expansion of product. If the product expands less, a compact structure appears to be dull and hence it results in low score in sensory color. There was a significant difference ($P<0.05$) in porosity between extrudate E and extrudates A and B but extrudate E was not significantly different ($P>0.05$) from C and D.

Table 4.9 Mean scores for sensory attributes of barley-grape pomace extrudates produced by different extrusion conditions

Sensory attributes	Barley-grape pomace extrudates				
	A ¹	B	C	D	E
Appearance					
Color	4.93 ^a	5.12 ^a	4.19 ^b	4.59 ^{ab}	2.25 ^c
Porosity	4.59 ^a	4.62 ^a	4.35 ^{ab}	4.37 ^{ab}	3.68 ^b
Taste					
Bran flavor	3.59 ^a	3.93 ^a	3.65 ^a	3.68 ^a	3.25 ^a
Bitterness	1.59 ^a	1.46 ^a	1.62 ^a	1.46 ^a	1.46 ^a
Sweetness	2.43 ^a	2.25 ^a	3.43 ^b	3.65 ^b	4.65 ^c
Off-odor	1.56 ^a	1.34 ^a	1.65 ^a	1.21 ^a	1.68 ^a
Texture					
Hardness	3.90 ^a	5.53 ^b	4.18 ^a	5.09 ^b	2.71 ^c
Crispness	4.34 ^a	5.56 ^b	4.46 ^a	5.59 ^b	3.31 ^c
Brittleness	4.03 ^a	5.81 ^b	4.40 ^a	5.15 ^c	2.93 ^d
Overall acceptability	4.65 ^a	5.53 ^b	4.84 ^{ab}	5.34 ^{ab}	2.40 ^c

¹Means within a row with different superscripts are significantly different ($P<0.05$).

A: 0% pomace level, 150°C, 175 rpm; B: 2% pomace level, 160°C, 200 rpm; C: 6% pomace level, 150°C, 217 rpm; D: 10% pomace level, 160°C, 150 rpm; E: 12.7% pomace level, 150°C, 175 rpm

Taste of extrudates was evaluated as bran flavor, bitterness and sweetness. There were no significant differences ($P>0.05$) in bran flavor and bitterness in selected extrudates. Low sensory scores were observed for bran flavor (3.25-3.93) and bitterness (1.46-1.62). More sweet taste was detected for extrudate E with 12.7%

grape pomace level which is expected and it was significantly different ($P < 0.05$) from other extrudates (Table 4.9). Extrudates received low sensory scores for off-odor (1.21-1.68). This means that panelists did not perceive any off-odor from extrudates.

Texture of extrudates was assessed in terms of hardness, crispness and brittleness. Extrudates B and D had higher preference with higher sensory scores in hardness (5.53-5.09), whereas extrudate E had lowest sensory score (2.71). They were significantly different ($P < 0.05$) in sensory hardness from extrudates A and C. Sensory hardness was negatively correlated with peak force and slope measured instrumentally at $P < 0.05$ (Table A.11). An increase in peak force measured as hardness and slope of extrudates as crispness resulted in decrease in preference of panelists in sensory hardness. Grape pomace levels of 2 and 10% had higher preference in sensory crispness (5.56-5.59), whereas 12.7% of grape pomace gave lower sensory score (3.31). A decrease in preference of sensory crispness of extrudate E (12.7% pomace) may be attributed to the increasing fiber content resulted in a less expanded, more compact texture in extrudate. Peak force, measured as hardness, was positively correlated ($R = 0.941$, $P < 0.05$) with slope (as crispness) measured instrumentally. However, peak force was negatively correlated ($R = -0.943$, $P < 0.05$) with sensory crispness which agrees with Seymour and Hamann (1988) and Norton et al. (1998). Seymour and Hamann (1988) investigated crispness and crunchiness of five moisture foods subjected to three humidity conditions. They showed that maximum shear/compressive force and work done to failure respectively correlated inversely with crispness and crunchiness of low moisture snack foods. The sample has been placed between incisors and bitten though; crispness has been evaluated as the level of higher pitched noise. Norton et al. (1998) demonstrated a method to determine the contribution to overall line length of the force-compression curve by fractures of specific dimensions. The method has been applied to a series of puffed rice samples extruded with various water contents to create different textures and to investigate the relationship between the mechanical method and perceived texture, samples were assessed by a sensory panel. They found a negative correlation of maximum force with sensory crispness. Mohamed et al. (1982) investigated the crispness of fried food products using instrumental and sensory evaluation methods. They pointed out that the poor correlation between sensory crispness and

instrumental hardness could be associated to the fact that a hard product did not necessarily mean a crisp one. A negative correlation was observed between slope and sensory crispness, sensory color and porosity (Table A.11). This is expected because crispness is related with texture formed. The expanded extrudate would have larger cells with thinner cell walls, resulting in a crispy texture and hence resulted in low value of crispness force (slope) and high score of sensory crispness. Since the structure of extrudate becomes more open with larger cells, hardness decreases, color becomes brighter and it gets higher sensory preferences. Sensory crispness was positively correlated with sensory hardness (Table A.11). Mean sensory scores of extrudates A and C were not significantly different ($P>0.05$) in sensory brittleness, whereas extrudates B, D and E were significantly different ($P<0.05$). Sensory brittleness in preference was the lowest (2.93) in extrudate E and the highest (5.81) in extrudate B. High correlations were found between sensory brittleness and peak force, slope, sensory hardness and sensory crispness. Sherman and Deghaidy (1978) reported that the maximum force at fracture of low moisture foods correlated inversely with panelists' evaluations of brittleness either in the mouth or using the fingers as found in this study.

The overall acceptability of extrudate B showed higher preference (5.53) but this was not significantly different ($P>0.05$) from extrudates D (5.34) and C (4.84). The lowest score (2.40) was observed for extrudate E with highest level of grape pomace level with a significant difference ($P<0.05$) over other extrudates. High correlations were observed between overall acceptability and all sensory attributes and textural properties except brittleness (Table A.11). A negative correlation between overall acceptability-peak force and overall acceptability-slope indicates that extrudates with small peak force and slope have better overall acceptability.

4.6 Chemical Analysis

4.6.1 Antioxidant activity and total phenolic content

Phenolic compounds in barley include phenolic acids (benzoic and cinnamic acid derivatives), flavonoids, proanthocyanidins, tannins and amino phenolic compounds, all of which are known to possess antioxidant and antiradical properties (McMurrough et al. 1996; Goupy et al. 1999; Hernanz et al. 2001; Bonoli et al. 2004; Papetti et al. 2006). Yu et al. (2001) identified the major phenolic acids in barley as

derivatives of benzoic acid as *p*-hydroxybenzoic, vanillic and protocatechuic acids and coumaric, caffeic, ferulic and chlorogenic acids as cinnamic acid derivatives. Goupy et al. (1999) reported that besides polyphenols, barley extracts contained other antioxidants, carotenoids (lutein and zeaxanthin) and tocopherols (α , δ and γ). Phenolic acids, tocopherols and tocotrienols are known to be strong antioxidants (Gao et al. 2002).

DPPH is a stable free radical that accepts an electron or hydrogen radical to become a stable molecule. DPPH radical scavenging is one of the important methods to evaluate antioxidant activity of phenolic compounds. The reduction in the DPPH radical is measure of antioxidant activity (Thippeswamy and Akhilender Naidu, 2005). Antioxidant reacts with DPPH and converts it to 1,1-diphenyl-2-picrylhydrazine. The degree of discoloration indicates the scavenging potentials of the antioxidant extract (Chidambara Murthy et al. 2002).

Antioxidant activity as measured by the DPPH method was 43.17, 27.57 and 82.23% while total phenolic contents, expressed as ferulic acid equivalents, were 5.29, 4.66 and 9.15 mg/g dry sample in the extracts obtained from barley flour, tomato and grape pomaces, respectively. Several authors have studied total phenolic contents of barley samples. Amarowicz et al. (2007) indicated that barley possess marked antioxidant and antiradical capacities as compared to other grains such as wheat, rye, and triticale. Madhujith et al. (2006) found that the total phenolic content of Falcon barley fractions ranged 0.39 to 6.26 mg ferulic acid/g of defatted material while that of AC Metcalfe ranged from 0.17 to 4.16 mg ferulic acid/g of defatted material. On the other hand, Madhujith and Shahidi (2006) observed that total phenolic content measured according to Folin-Ciocalteu's method ranged from 13.58 to 22.93 mg of ferulic acid/g of defatted material in different barley varieties. The differences between these results and those in the present work may be attributed to the different varieties and to the different extraction solvent and tests used to evaluate the total phenolic content. The antioxidant activities and total phenolics of extrudates from barley flour are shown in Figures 4.33 and 4.34. Extrusion cooking significantly reduced both antioxidant activities and total phenolics in all barley extrudate samples by 60-68 and 46-60% compared to that of the unprocessed barley flour. The losses observed in antioxidant activities due to extrusion are consistent with those reported

by Dlamini et al. (2007). Such findings were supported by Grela et al. (1999) and Zieliński et al. (2001). It was previously shown that extrusion caused a significant reduction in the content of the natural antioxidants α - and γ -tocopherol, β -carotene and lutein in extruded grass pea seeds (Grela et al. 1999).

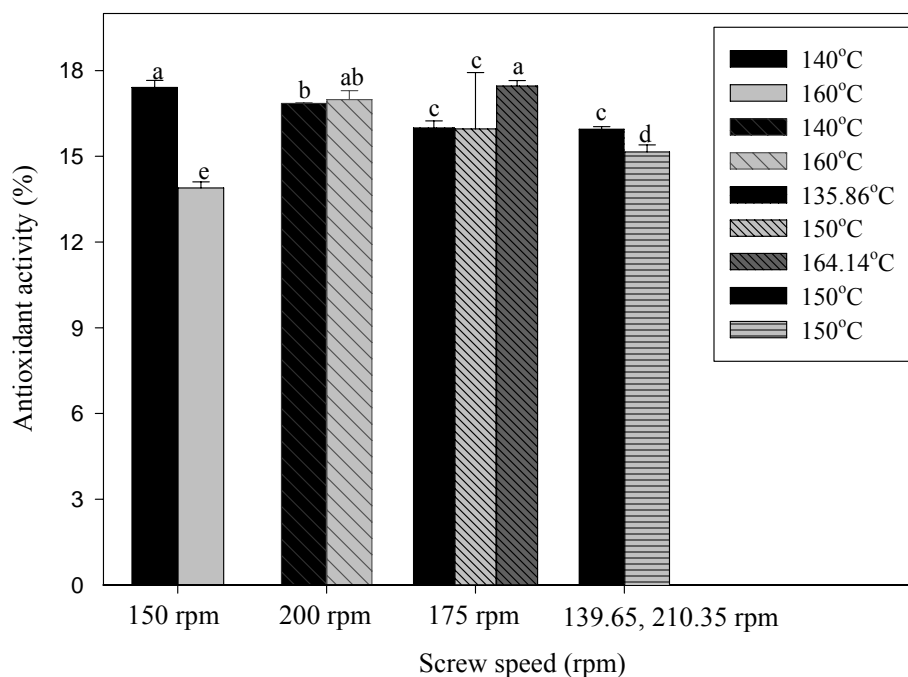


Figure 4.33 Remaining antioxidant activities of barley flour extrudates at different extrusion conditions. Values with different superscripts were significantly different at $P < 0.05$. (Antioxidant activity of barley flour was 43.17%)

Zadernowski et al. (1999) stated that the disadvantage of natural antioxidants is their low resistance to high temperatures since heating over 80°C destroys their antioxidant properties. The loss of antioxidants has been attributed to both evaporation and decomposition at elevated temperatures as reported by Hamama and Nawar (1991). They concluded that phenolic antioxidants exhibited significant decomposition at elevated temperatures and give rise to a number of breakdown products which in turn can further decompose. Zieliński et al. (2001) found that extrusion caused a significant decrease in tocopherols and tocotrienols from 63 to 94% depending on the type of cereal grains. They also reported that the least resistant to extrusion processing was α -tocopherol and α -tocotrienol. The remaining tocopherols were more stable; however, the degree of their degradation was up to 50%. Zieliński et al. (2006) stated that a reduction in the overall antioxidant

properties of extruded dehulled buckwheat seeds was due to the loss of naturally occurring antioxidants. Extrusion conditions resulted in degradation of phenolic compounds that observed in oat extrudates and in reduction of their antioxidative properties (Zadernowski et al. 1999). They observed about 50% degradation of phenolic compounds during extrusion. It was suggested that the damage of significant amounts of phenolic compounds during extrusion might be the cause for the lack of antioxidant properties (Zadernowski et al. 1999). Alonso et al. (2000) found reduction in polyphenols in peas by extrusion cooking. The authors claimed that the high temperature of extrusion might alter their molecular structure and either reduce their chemical reactivity or decrease their extractability due to a certain degree of polymerization. Extrusion also decreased the antioxidant activity of bean extrudates compared to the unprocessed bean (Korus et al. 2007). They found that the effect of extrusion on the total phenolic content of beans depended on the cultivar and one variety showed a 14% increase in the amount of phenolics in extrudates compared to raw beans, while the other two exhibited a decrease by 19 and 21%. It was stated that extrusion caused a reduction in total phenolics of extruded oat cereals by 24-46% (Viscidi et al. 2004). Dlamini et al. (2007) reported that extrusion cooking significantly reduced measurable total phenols and tannins for both whole and decorticated tannin sorghums. Some authors, however, have observed positive effect of extrusion on phenolic content of cereals (Zieliński et al. 2001; Gumul and Korus, 2006). Gumul and Korus (2006) concluded that the increase in the level of phenolic acids (particularly ferulic acid) after extrusion can be attributed to the release of the acid and derivatives from the cell walls of the plant material.

The conditions of extrusion had an unclear effect on the antioxidant activity which meant that there was no general trend with extrusion temperature and screw speed. It can be seen that barley flour extruded at a lower temperature, i.e. 140°C, showed a higher antioxidant activity than that extruded at 160°C with a screw speed of 150 rpm, whereas at both extrusion temperatures and 200 rpm (Figure 4.33), nearly the same antioxidant activities were determined in extrudate samples. The extrudate produced at 150°C temperature and 139.65 rpm screw speed displayed high antioxidant activity when compared to that of extrudate obtained at 150°C temperature and 210.35 rpm screw speed. The phenolic contents of barley flour extrudates ranged from 2.10 to 2.87 mg ferulic acid/g dry sample which were not

statistically different ($P>0.05$) from each other except that extrusion at 160°C and 200 rpm screw speed (Figure 4.34).

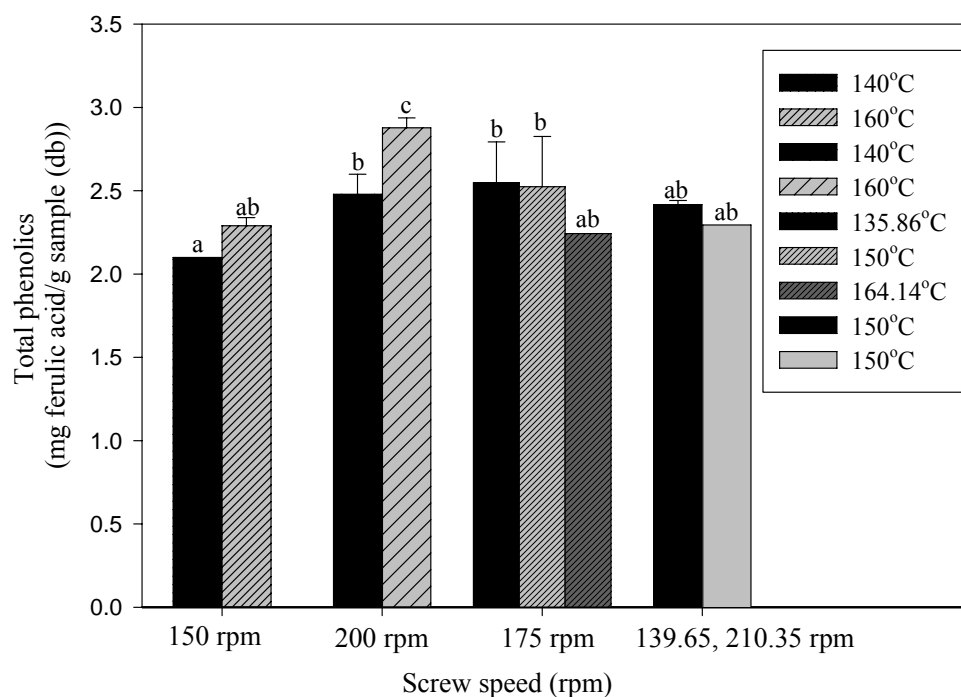


Figure 4.34 Total phenolics of barley flour extrudates at different extrusion conditions. Values with different superscripts were significantly different at $P<0.05$. (Total phenolic content of barley flour was 5.29 mg/g sample)

Antioxidant activity was not significantly correlated with phenolic content. A similar lack of correlation between the antioxidant activity and phenolic contents was observed by Camire et al. (2005, 2007) in extruded corn including ginkgo extract, steamed potato peels or wheat bran and white cornmeal cereals containing fruit powder. Maillard et al. (1996) found no clear correlation between the level of phenolic compounds and the antioxidant activity in seven different varieties of barley. Madhujith and Shahidi (2006) reported that DPPH radical scavenging capacities did not strongly correlate with total phenolic content, indicating that factors other than total phenolic content might play a role in the antioxidant activity of the extracts of barley samples. It has been reported that all the phenolics do not have the same antioxidant activity, some are powerful, others are weak and they may develop antagonistic or synergistic effects with themselves or with other constituents of the extracts (Rice-Evans et al. 1996; Moran et al. 1997; Lien et al. 1999; Zieliński and Kozłowska, 2000). Other than phenolic compounds, protein in barley that was extracted in aqueous methanol or the combined action of phenolics and protein in the

sample might contribute to antioxidant activity (Zieliński and Kozłowska, 2000; Madhujith and Shahidi, 2006). Cereal proteins have been reported to exert a strong antioxidant activity (Iwama et al. 1987). Folin-Ciocalteu reagent detects all phenolic groups found in extracts, including those found in extractable proteins (Shahidi and Naczk, 1995). Arts et al. (2002) suggested that the interaction of flavonoids with proteins might mask part of the total antioxidant activity. The degree of this masking depends on both the type of polyphenol and the type of protein (Arts et al. 2002). Korus et al. (2007) also have not observed a significant correlation between the DPPH radical scavenging power and the flavonoid and phenolic acid contents.

The regression equation obtained for antioxidant activity of barley flour-tomato pomace extrudates as a function of pomace level and screw speed in terms of coded variables is presented in Table 4.7. The coefficient of determination (R^2) for antioxidant activity was 0.637 with a low value of coefficient of variation (CV=8.46%) of the predicted model. The lack of fit was not significant ($P>0.05$) for antioxidant activity (Table A.12). The interaction of pomace level and screw speed had a significant effect ($P<0.05$) on antioxidant activity of barley flour-tomato pomace extrudates. However, temperature did not affect significantly ($P>0.05$) antioxidant activity of barley-tomato pomace extrudates.

The effect of pomace level and screw speed on the antioxidant activity is shown in Figure 4.35. The antioxidant activity value of samples decreased with increase in screw speed at low pomace levels. Similar effect of screw speed on antioxidant activity was observed by Ozer et al. (2006). Increasing screw speed resulted in an increase in mechanical energy input to the system with increasing shearing effect (Ozer et al. 2006). The authors suggested that the increased shearing effects were more dominant than the effect of residence time on the destruction of antioxidant activity over the extrusion condition even though increased screw speeds associates with decreased residence times.

Table 4.10 The regression models for chemical analyses using independent variables temperature (X_1), screw speed (X_2) and pomace level (X_3) of barley and barley-pomace extrudates

Response	Model	R ²
AA _T	$16.17 + 1.86X_2X_3$	0.637
AA _G	$13.34 - 1.11X_2^2 - 1.0X_1X_3$	0.785
TP _G	$2.43 + 0.27X_3$	0.564
DG _B	$86.09 - 2.98X_1 + 4.17X_2 - 4.44X_1^2$	0.860
DG _T	$95.79 - 4.99X_1 - 4.41X_1^2 - 5.51X_3^2 + 6.03X_1X_2 - 6.81X_1X_3$	0.780
DG _G	$90.77 - 3.52X_1 + 4.29X_2 - 5.30X_1^2$	0.751
SD _T	$448.56 - 27.06X_1 - 23.94X_3 - 21.66X_1^2 + 23.24 X_1X_3$	0.883
SD _G	$378.95 - 26.05X_3 + 28.37X_1^2 + 20.08X_3^2$	0.782
BG _B	$3.39 - 0.90X_1 + 0.15X_1X_2$	0.617
BG _T	$2.98 - 0.23X_3$	0.688
BG _G	$3.17 - 0.045X_1 - 0.083X_3 + 0.053X_2^2$	0.821

B: barley flour, T: tomato pomace, G: grape pomace

Although a reduction in the antioxidant activity of barley flour-tomato pomace extrudates in overall appeared to occur as a result of extrusion process, antioxidant activity increased when pomace level increased at high screw speed. This could be attributed to the contribution of antioxidants present in tomato pomace. There is no available data about what happened in tomato pomace with extrusion cooking but the effect of thermal processing on tomato has been reported by several authors. Sahlin et al. (2004) found that boiling, baking and frying of tomatoes resulted in a significant reduction in the ascorbic acid, total phenolic and lycopene contents when compared to respective raw cultivar. Zanoni et al. (1999) reported a significant loss (10%) of lycopene after air drying at 110°C, whereas no significant loss occurred during drying at 80°C. Goula and Adamopoulos (2005) investigated the stability of lycopene from tomato pulp during spray drying. They concluded that lycopene loss ranged between 8.07 and 20.93% and the extent of loss increased with increases in air inlet temperature. In another study, Toor and Savage (2006) found a reduction in the hydrophilic antioxidant activity by 28-38% in semi-dried tomatoes compared with the fresh. They stated that decline in total phenolics and ascorbic acid during processing could likely be responsible for decreases in antioxidant activity in the semi-dried tomatoes. The main antioxidants in tomatoes are carotenoids, ascorbic acid and phenolic compounds (Giovanelli et al. 1999). Hanson et al. (2004) also reported that tomato is important source of certain antioxidants including lycopene,

β -carotene and vitamin C. Toor and Savage (2005) reported that the skin and seed fractions of tomato are a very rich source of antioxidant compounds and the incorporation of the skin and seeds fraction during home consumption or processing could lead to about a 40-53% increase in the amount of all the major antioxidants in the final product. Knoblich et al. (2005) analyzed tomato peel and seed by-products and they concluded that tomato peel by-products was a rich source of lycopene, several times more concentrated than was present in seed by-products.

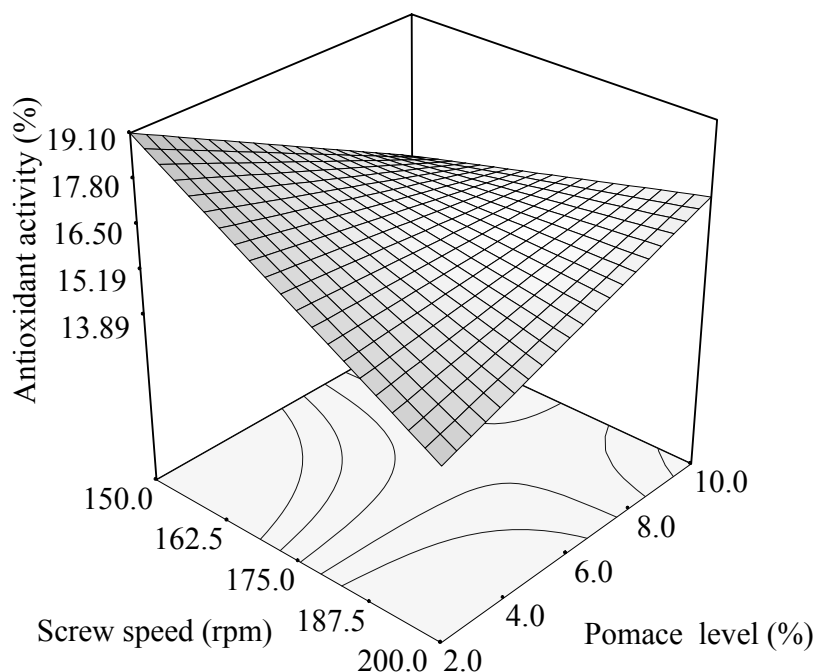


Figure 4.35 Response surface plot for the effect of pomace level and screw speed on the antioxidant activity of barley-tomato pomace extrudates at a temperature of 150°C

Our results showed that total phenolic content of barley-tomato pomace extrudates did not fit a model to explain the effects of extrusion conditions and pomace levels despite the significant changes in the antioxidant activities with those factors occurred. Although no significant change among total phenolic content of extrudates was observed, extrusion cooking decreased significantly ($P < 0.05$) total phenolics when compared to raw materials. However, Sensoy et al. (2006) found that roasting did not affect the phenolic content of either dark or white buckwheat flour but antioxidant activity decreased by roasting, whereas extrusion did not cause any change. Ozer et al. (2006) reported that phenolic content of extruded snack food did not change by extrusion processing. The total phenolics of the barley flour-tomato pomace extrudates that were analyzed varied from 2.09 to 2.81 mg ferulic acid per g

dry sample. Shen et al. (2007) found that the phenolics isolated from fresh and heat treated tomatoes were gallic acid, chlorogenic acid, caffeic acid, myricetin and naringenin. They reported that crude phenolic extracts of tomato had fair antioxidant activity either with or without heat treatments and synergistically promoted the antioxidation of lycopene. Luthria et al. (2006) identified three phenolic acids which are caffeic acid, *p*-coumaric acid and ferulic acid in tomato extracts. According to these authors, caffeic acid was the predominant phenolic acid, whereas the content of ferulic acid was the lowest of three phenolic acids in all tomato extracts.

The equations of second and first order polynomial models employed to predict the antioxidant activity and total phenolic content of barley-grape pomace extrudates developed in terms of coded variables are given in Table 4.10. Regression analysis showed that antioxidant activity was significantly ($P < 0.05$) affected by quadratic term of screw speed and interaction term of temperature and pomace level while total phenolic content affected significantly ($P < 0.05$) from pomace level only. An analysis of variance (ANOVA) for the models is summarized in Table A.12. The lack of fit was found to be non-significant ($P > 0.05$) for both antioxidant activity and total phenolic content with low coefficients of variation (7.48 and 10.14%) of the predicted models. The coefficients of determination for antioxidant activity and total phenolic content were found to be 0.785 and 0.564, respectively.

Response surface plot for antioxidant activity as a function of temperature and pomace level is given in Figure 4.36. Similar to barley-tomato pomace extrudates, an increase in the screw speed resulted in lower antioxidant activity of extrudate samples. A higher temperature of extrusion and a higher level of grape pomace led to an increase in antioxidant activity of barley flour-grape pomace extrudates. Gumul and Korus (2006) found high antioxidant potential of the rye bran extrudates produced at a temperature of 120 or 180°C. The reason of the high antioxidant potential of the extrudates was partly accounted for the presence of the high molecular weight products of Maillard's reaction which are formed at higher temperatures and act as antioxidants (Nicoli et al. 1999; Gumul and Korus, 2006). An increase in antioxidant activity with increasing pomace level can be attributed to phenolic compound in grape pomace. This result was also supported by significant increase in total phenolic content by increasing level of grape pomace (Figure 4.37).

Kedage et al. (2007) analyzed 11 varieties of grapes including Thompson seedless grape and identified different phenolics and flavonoids such as gallic acid, catechin, caffeic acid, hydrocaffic acid, *o*-coumaric acid, *p*-coumaric acid, rutin and quercetin. Grape pomace has been reported as rich in phenolic compounds (Chidambara Murthy et al. 2002). Lu and Fo (1999) found a variety of polyphenols in Chardonnay grape pomace included phenolic acids, phenolic alcohol, flavan-3-ols and flavonoids. No information about polyphenols' stability and antioxidant activity of grape pomace during extrusion has been reported. However, Larrauri et al. (1997) studied effect of drying temperature (60, 100 and 140°C) on the stability of polyphenols and antioxidant activity of red grape pomace peels. They found that when drying temperature was 100 and 140°C, significant reduction in both total extractable polyphenols and condensed tannins by 18.6 and 32.6% was observed as well as a decrease of 28 and 50% in the antioxidant activity of the samples. The reduction of the polyphenol content from 100 to 140°C was attributed to thermal degradation. Maillard and Berset (1995) explained the decrease of bound phenolic acids in three ways: release of bound phenolic compounds; partial degradation of lignin which could lead to the release of phenolic acid derivatives; and/or the beginning of thermal degradation of the phenolic compounds. The effect of temperature and screw speed on phenolic content of extrudates was not significant ($P>0.05$). In general, extrusion decreased both antioxidant activity and total phenolic content of barley flour-grape pomace extrudates when compared to the unprocessed raw materials. However, Zieliński et al. (2001) observed an increase in the phenolic acids content of whole grains of wheat, barley, rye and oat reached from 200 to 300% after extrusion at a temperature of 120-200°C. They found ferrulic acid as a predominant compound in raw whole-grain as well as in extruded grain. Zieliński et al. (2006) reported a two-fold higher content of phenolic acids for the dehulled buckwheat seeds after extrusion. No correlation was observed between antioxidant activity and total phenol contents of extracts obtained from grape pomace extrudates. Similarly, Lafka et al. (2007) found no correlation between antioxidant activity and phenol content in winery wastes.

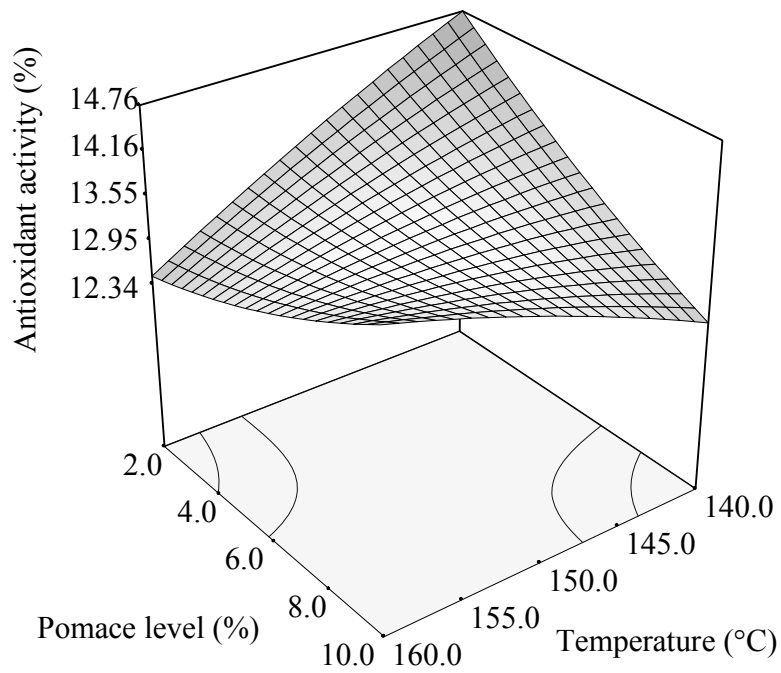


Figure 4.36 Response surface plot for the effect of temperature and pomace level on the antioxidant activity of barley-grape pomace extrudates at a screw speed of 175 rpm

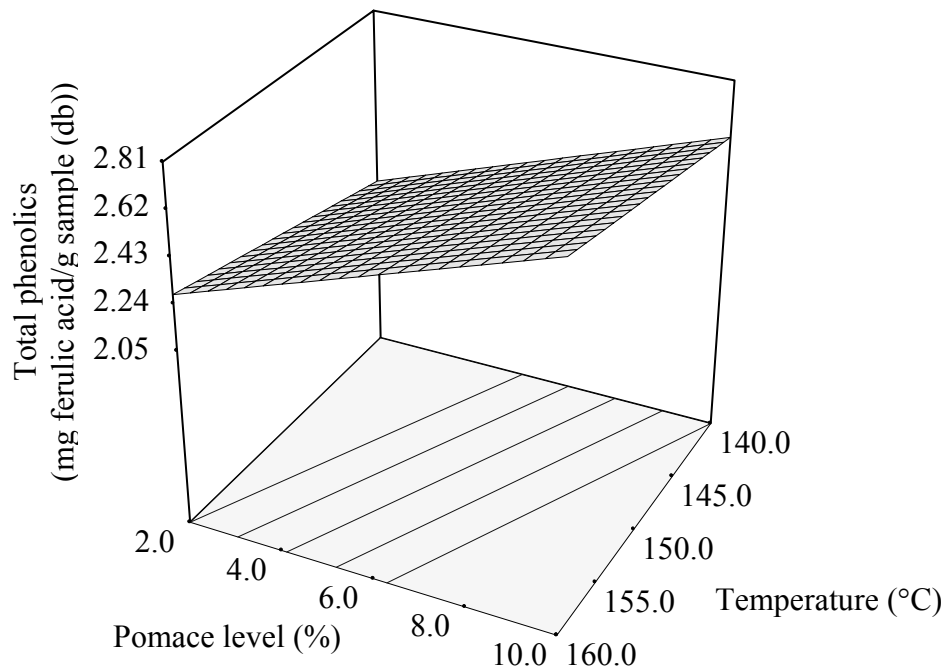


Figure 4.37 Response surface plot for the effect of temperature and pomace level on the total phenolic content of barley-grape pomace extrudates at a screw speed of 175 rpm

4.6.2 Gelatinization

Gelatinization of starch can be determined by several methods such as starch-iodine complexing method (Gomez and Aguilera, 1983), loss of birefringence (Baks et al. 2007) and differential scanning calorimetry (DSC) (Souza and Andrade, 2002). The iodine complexing method is based on the fact that gelatinized starch could solubilize in water more easily and uptake iodine faster than the ungelatinized starch to form an iodine-starch complex. The color of the iodine-starch complex depends on the molecular size (Lai and Kokini, 1991). The models developed for the degree of gelatinization of barley and barley-pomace extrudates as functions of coded independent variables are presented in Table 4.10. ANOVA for models of degree of gelatinization is given in Table A.12. The coefficients of determination for degree of gelatinization of barley, barley-tomato pomace and barley-grape pomace extrudates were 0.860, 0.780 and 0.751, respectively. The lack-of-fit was found to be non-significant ($P>0.05$) for degree of gelatinization of barley and barley-tomato pomace extrudates, whereas significant ($P<0.05$) for barley-grape pomace extrudates. Although the lack-of-fit was significant for grape pomace extrudates, the coefficient of variance was found to be low (6.55%). The coefficients of variance for barley and barley-tomato pomace extrudates were 3.48 and 7.79%, respectively. Temperature had significant ($P<0.05$) both linear and quadratic effects on all types of extrudate while screw speed had significant ($P<0.05$) linear effect on barley and barley-grape pomace extrudates on degree of gelatinization. The interactions of temperature with screw speed and pomace level on degree of gelatinization were found to be significant ($P<0.05$) for barley-tomato pomace extrudates.

The influence of temperature and screw speed on the extent of gelatinization determined by iodine complexing method for barley extrudates is shown in Figure 4.38. The degree of gelatinization of barley extrudates increased with temperature at 150°C with a screw speed of 200 rpm, but a decrease was observed by further increasing temperature. Similar results were reported for corn starch and rice flour extrudates by Owusu-Ansah et al. (1983) and Guha et al. (1998). Owusu-Ansah et al. (1983) observed that gelatinization was affected in anomalous manner; decreasing with increasing temperature. The authors attributed its cause to the choice of screw profile, which led to complete gelatinization at low temperature, probably because of mechanical shear associated with the profile used. Guha et al. (1998)

reported that this reduction could be due to a higher extent of degradation produced by a higher applied mechanical (shear) force on account of the screw profile selected, or due to a reduced swelling on account of the formation of resistant starch, starch-lipid complexes, starch-protein complexes (Nwabueze, 2006) or even retrograded amylose. However, other researchers observed that an increase in temperature increased the starch gelatinization (Bhattacharya and Hanna, 1987; Cai and Diosady, 1993). An increase in screw speed resulted in increase in the degree of gelatinization of barley extrudates (Figure 4.38). The results are in agreement with the findings of Cai and Diosady (1993), Guha et al. (1998) and Doğan and Karwe (2003) who observed an increase in degree of gelatinization with increase in screw speed during extrusion of wheat starch, rice and quinoa extrudates. This confirms that shear is a significant contributor to starch gelatinization (Cai and Diosady 1993; Doğan and Karwe, 2003). Increased shear effects at higher screw speeds would lead to higher gelatinization possibly by increasing SME. Degree of gelatinization was positively correlated ($R=0.682$, $P<0.05$) with SME of barley extrudates. Similar correlation was observed between the degree of gelatinization and SME by Doğan and Karwe (2003).

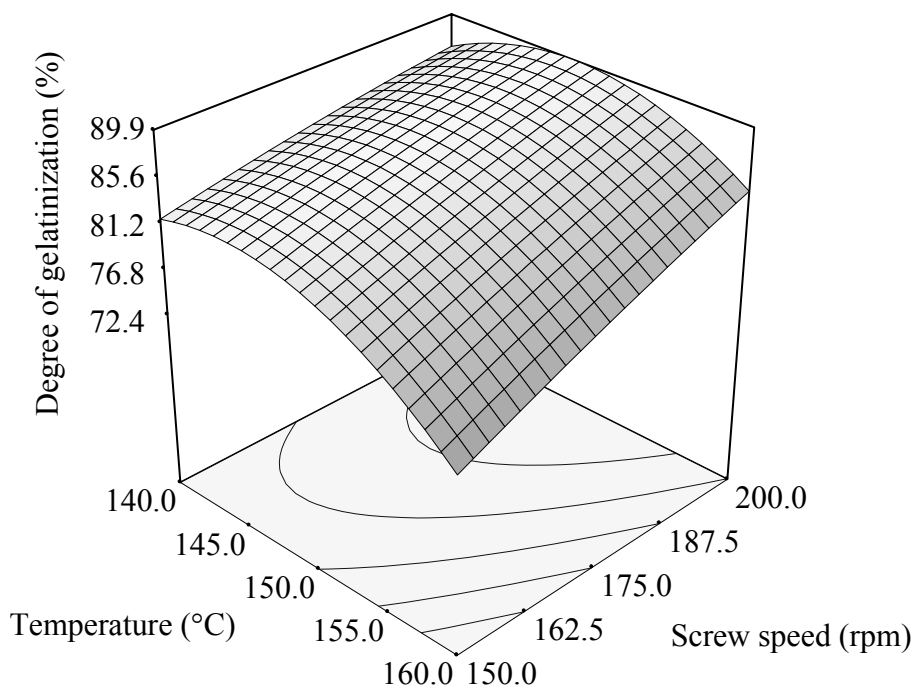


Figure 4.38 Response surface plot for the effect of screw speed and temperature on the degree of gelatinization of barley extrudates

Ilo et al. (1996) found the starch gelatinization of extruded maize grits increased with increasing the SME during extrusion. Kokini (1993) reported that as the ratio of specific mechanical energy to specific total energy increases during extrusion, the degree of gelatinization increases. This is the expected result, indicating the role of mechanical energy in the conversion of starch (Kokini, 1993). Starch gelatinization and breakdown depend on mechanical and thermal energy inputs (Choudhury and Gautam, 1998). The degree of gelatinization was correlated ($R=0.788$, $P<0.01$) with WSI of barley extrudates. Gomez and Aguilera (1983) found that degree of gelatinization correlated very well with WSI ($R=0.963$) indicating that in the case of extruded products, degree of gelatinization should be considered as an indicator of the extent of starch degradation rather than as an index of gelatinization.

The effect of temperature on the degree of gelatinization for barley-pomace blends extrudates was similar to that of barley extrudates. The interactions of temperature with screw speed and pomace level are shown in Figures 4.39 and 4.40. At low screw speed, the increase in temperature decreased the degree of gelatinization while at high screw speed, the increase in temperature resulted in an increase in degree of gelatinization. It was reported that a higher screw speed generated a higher friction and, therefore, a higher energy input into the material, which resulted in a longer cooking zone and a higher degree of starch gelatinization at the end of barrel even though the residence time of the material was shorter (Lin et al. 1997). Diosady et al. (1985) found that a higher degree of cooking was observed on the extrudates as a result of increasing screw speed. It is known that the mechanical energy input (shear force) is one of the major factors contributing to the starch degradation (Diosady et al. 1985). The shear force, which physically tears apart starch granules during extrusion allows faster transfer of water into the interior starch molecules and would affect the degree of gelatinization (Burros et al. 1987). On the other hand, Govindasamy et al. (1996) observed that increasing or decreasing the screw speed around 410 rpm resulted in a rise in the degree of gelatinization. They reported that raising screw speed (325 to 410 rpm) increased shear rate but lowered residence time, which reduces swelling making the granule less susceptible to shearing action. However, at higher screw speeds (410 to 485 rpm), the shearing action presumably predominates over residence time accounting for the enhanced gelatinization (Govindasamy et al. 1996). Bhattacharya and Hanna (1987) found an insignificant

effect of screw speed on gelatinization in the range of 93.5 to 166.5 rpm for whole waxy and ordinary corn. Positive correlation was found between the degree of gelatinization and SME ($R=0.477$, $P<0.05$) as well as SEI ($R=0.694$, $R<0.01$) of barley-tomato pomace extrudates. This is in agreement with results of Doğan and Karwe (2003) who found high correlation between gelatinization and SEI and SME. Increasing the temperature at low tomato pomace level led to an increase in degree of gelatinization, while increasing the temperature at high pomace level lowered degree of gelatinization (Figure 4.40). Lue et al. (1991) found that addition of sugar beet fiber (0-30%) did not influence the degree of gelatinization. Yanniotis et al. (2007) stated that when non-starch polysaccharides like pectin are present, they have the capacity to hydrate and consequently to compete for and restrict the plasticizer and hence the gelatinization process by increasing the melt viscosity and reducing the availability of water for the gelatinization process. The effect of temperature and screw speed was found to be significant ($P<0.05$) for barley-grape pomace extrudates. An increase in temperature decreased the degree of gelatinization, whereas increase in screw speed caused to an increase in gelatinization (Figure 4.41). Grape pomace level did not affect the degree of gelatinization. Degree of gelatinization for barley-grape pomace extrudates was correlated with SME ($R=0.599$, $P<0.01$) and expansion ($R=0.468$, $P<0.05$) as well.

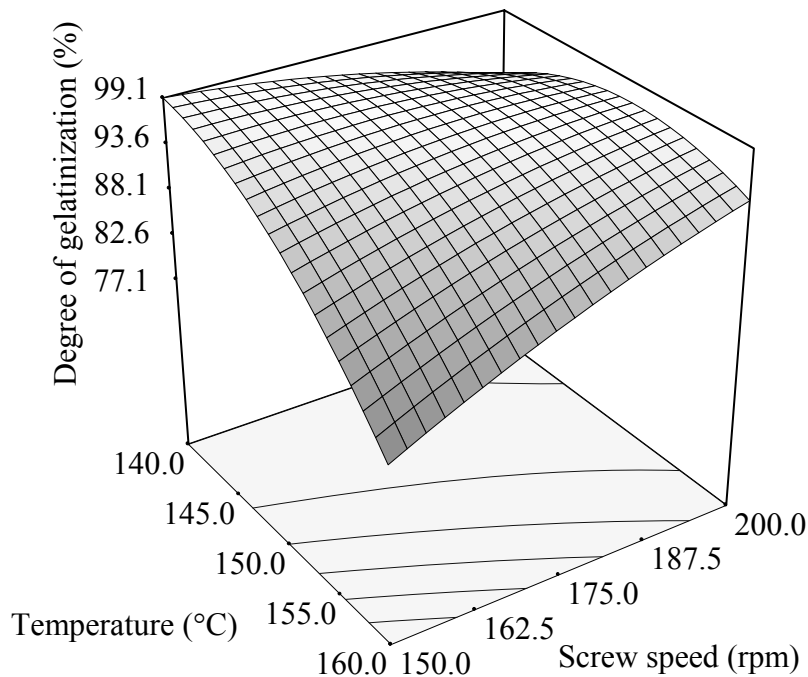


Figure 4.39 Response surface plot for the effect of screw speed and temperature on the degree of gelatinization of barley-tomato pomace extrudates at a pomace level of 6%

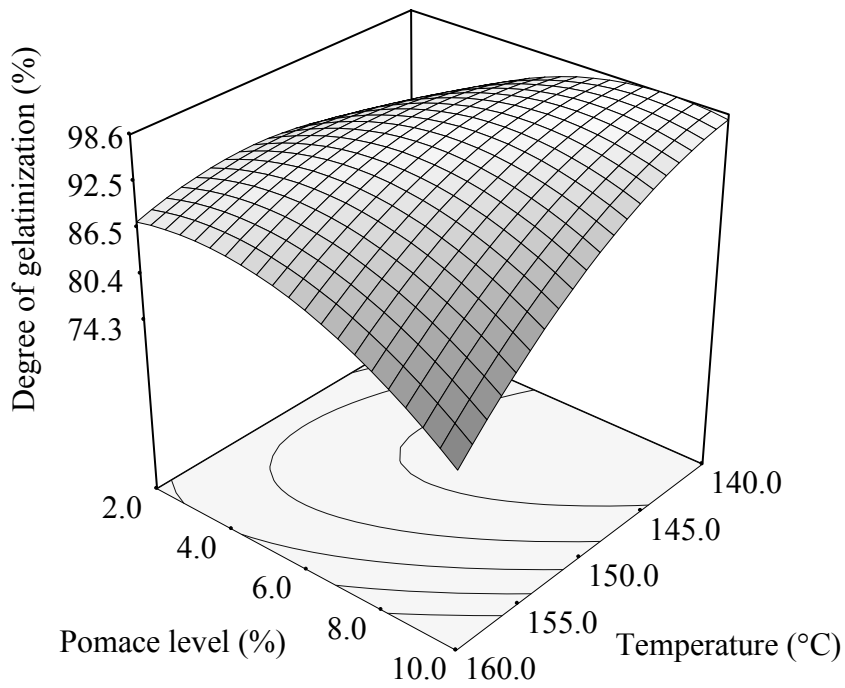


Figure 4.40 Response surface plot for the effect of temperature and pomace level on the degree of gelatinization of barley-tomato pomace extrudates at a screw speed of 175 rpm

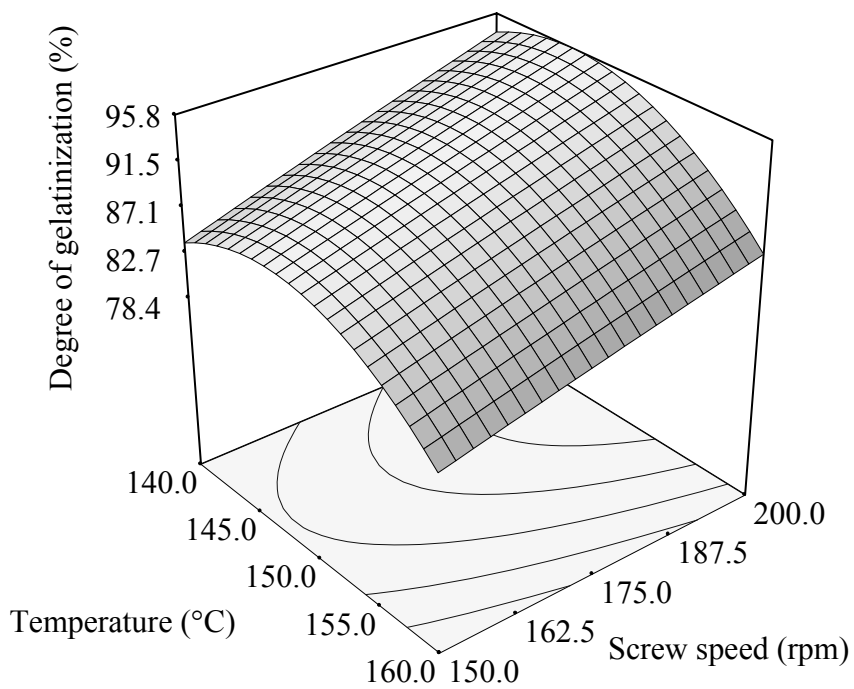


Figure 4.41 Response surface plot for the effect of screw speed and temperature on the degree of gelatinization of barley-grape pomace extrudates at a pomace level of 6%

The instruments, DSC and polarized light microscopy were used further to study and clarify gelatinization of extrudates. The principle in DSC is a sample and an inert material are both heated in a uniform way. The temperature difference between the sample and the reference is then converted to the enthalpy change. The results are then interpreted for starch systems as being related to the breaking of starch-starch hydrogen bonds in favor of starch-water bonds with increased entropy (Lai and Kokini, 1991). The major changes occurring in starch during the extrusion process are the disruption of the crystalline regions in the granule followed by the loss of starch granule birefringence. Polarizing light microscopy can be used to observe these changes (McHugh and Huxsoll, 1999). The loss of crystallinity is termed as gelatinization and is evidenced by the disappearance of birefringence in the form of Maltese crosses (Linko, 1992).

Gelatinization properties of barley flour and barley flour-pomace blends evaluated by DSC are presented in Table 4.11. Gelatinization temperature (T_p) of barley flour was found to be 68.5°C. The T_p values of barley flour-pomace blends varied within a narrow range 67.2-69.6°C when compared to that of barley flour. Björck et al. (1990) reported that gelatinization temperature of five different barley samples varied between 62 and 69°C. The peak temperature for isolated starch from normal, waxy and high amylose hull-less barley cultivars was found in the range of 64.9 to 68.8°C by Waduge et al. (2006). Czuchajowska et al. (1998) stated that the first transition temperature, >60°C, corresponded to endotherms of starch gelatinization while the second transition, >100°C, corresponded to the amylose-lipids complex. There was apparently little effect of the addition of tomato and grape pomaces on the peak temperatures (T_p) (Table 4.11). However, the enthalpy of gelatinization (ΔH_g) tended to decrease when pomace was added. There was a significant ($P < 0.05$) difference between the enthalpy of gelatinization of barley flour and barley flour-pomace blends. Similar results were observed by Cleary and Brennan (2006) in β -glucan enriched pasta. It was reported that certain polysaccharides restrict the swelling of starch granules and consequently restrict starch gelatinization, though immobilization of water, resulting in an increase in gelatinization temperature (Tester and Sommerville, 2003) and a decrease in enthalpy (Cleary and Brennan, 2006). Tudorică et al. (2002) found that the enthalpy of a flour/fiber complex decreased with increasing fiber concentration. Symons and Brennan (2004) observed a decrease

in enthalpy values of wheat starch with substitution of 5% barley β -glucan fiber fraction when compared with the control. Our results are in agreement with these findings.

Table 4.11 Gelatinization characteristics of barley flour and barley flour-pomace blends¹

Samples	Gelatinization transition				Amylose-lipid complex transition	
	T _o ² (°C)	T _p ² (°C)	T _c ² (°C)	ΔH_g ² (J/g)	T _{pcx} ³ (°C)	ΔH_{cx} ³ (J/g)
Barley flour	62.4 ^{abc}	68.5 ^{abc}	79.5 ^c	6.9 ^a	101.9 ^d	0.96 ^a
Blends						
BF-TP (2%)	62.4 ^{abc}	68.5 ^{abc}	77.6 ^{abc}	4.8 ^b	93.3 ^a	1.46 ^a
BF-TP (6%)	61.1 ^a	67.3 ^{ab}	77.5 ^{ab}	5.0 ^b	95.9 ^{bc}	2.18 ^a
BF-TP (10%)	62.5 ^{abc}	67.8 ^{abc}	76.5 ^a	4.2 ^b	95.4 ^{bc}	1.84 ^a
BF-TP (12.7%)	61.4 ^{ab}	67.2 ^a	77.3 ^{ab}	4.1 ^b	95.3 ^{bc}	2.19 ^a
BF-GP (2%)	62.7 ^{abcd}	68.5 ^{abc}	77.7 ^{abc}	4.2 ^b	94.2 ^{ab}	1.70 ^a
BF-GP (6%)	63.1 ^{bcd}	68.4 ^{abc}	77.8 ^{abc}	4.9 ^b	96.4 ^c	1.72 ^a
BF-GP (10%)	63.6 ^{cd}	69.2 ^{bc}	79.1 ^{bc}	4.5 ^b	95.8 ^{bc}	1.81 ^a
BF-GP (12.7%)	64.4 ^a	69.6 ^c	77.8 ^{abc}	3.8 ^b	95.7 ^{bc}	1.65 ^a

BF: Barley flour, TP: Tomato pomace, GP: Grape pomace

¹ Within columns means with same subscript are not significantly different (P<0.05)

² T_o, T_p, T_c and ΔH_g represent the onset, peak, conclusion and enthalpy of gelatinization

³ T_{pcx} and ΔH_{cx} represent the peak melting temperature of the amylose-lipid complex and the enthalpy of melting of the amylose-lipid complex

The amylose-lipid complex melting peak temperature (T_{pcx}) of barley flour was 101.9°C while those of barley flour-pomace blends were in the range from 93.3 to 96.4°C (Table 4.11). The value of T_{pcx} was in agreement with reported values for other barley studies (Yoshimoto et al. 2002; Waduge et al. 2006). Björck et al. (1990) also found T_{pcx} value changing between 93.7 and 105.5 for different types of barley samples.

The thermograms of barley flour and selected barley flour extrudates are shown in Figure 4.42 and also some examples for barley-tomato pomace and barley-grape pomace extrudates were given in Figure 4.43. Table 4.12 also shows transition peak temperature (T_{pcx}) and enthalpy (ΔH_{cx}) of amylose-lipid complex for some selected barley and barley-pomace extrudates. The extrudates produced at low and high temperatures and screw speeds as well as at high pomace level (12.7%) for barley and barley-pomace blends were selected. Barley flour had endothermic gelatinization

peak at 68.5°C (Figure 4.42). No peak was detected for barley flour extrudates indicating 100% gelatinization. However, iodine complexing method revealed that degree of gelatinization of barley flour extrudates is not more than 90% (Figure 4.38). This result was similar to the studies of Gomez and Aguilera (1984) and Blanche and Sun (2004). Gomez and Aguilera (1984) found no peak in the temperature range of 25-115°C for extrudate samples and postulated that the amount of native starch left in extruded samples is minimal and undetectable by the DSC procedure. Chanvrier et al. (2007) observed no residual gelatinization enthalpy by DSC for wholemeal products after extrusion at 110°C. However, they found that there was still some crystallinity left as detected by X-ray diffraction, which was not measurable by DSC, although no Maltese cross typical of native starch granules within the samples observed by polarized microscopy. The detected crystallinity in contrast to the DSC results was found as a V-type structure formed during processing, which was attributed to the formation of amylose-lipid complexes (Chanvrier et al. 2007).

Table 4.12 Thermal behavior of barley flour extrudates and barley-pomace blends extrudates¹

Samples	Amylose-lipid complex transition	
	T _{pcx} ² (°C)	ΔH _{cx} ² (J/g)
Barley extrudate		
136°C / 175 rpm	84.0 ^a	3.14 ^a
150°C / 140 rpm	84.7 ^a	2.42 ^a
150°C / 210 rpm	86.9 ^b	1.97 ^a
164°C / 175 rpm	90.9 ^c	2.36 ^a
Barley-tomato pomace extrudate		
133°C / 175 rpm / 6%	80.7 ^a	0.85 ^a
150°C / 133 rpm / 6%	84.6 ^b	0.97 ^a
150°C / 175 rpm / 12.7%	86.9 ^c	2.02 ^a
167°C / 175 rpm / 6%	88.4 ^c	1.34 ^a
Barley-grape pomace extrudate		
133°C / 175 rpm / 6%	89.0 ^a	1.79 ^a
150°C / 133 rpm / 6%	89.8 ^a	3.22 ^a
150°C / 175 rpm / 12.7%	90.1 ^a	1.92 ^a
150°C / 217 rpm / 6%	89.4 ^a	2.33 ^a
167°C / 175 rpm / 6%	89.4 ^a	2.58 ^a

¹ Within columns means with same subscript are not significantly different (P<0.05)

² T_{pcx} and ΔH_{cx} represent the peak melting temperature of the amylose-lipid complex and the enthalpy of melting of the amylose-lipid complex

Blanche and Sun (2004) observed no gelatinization peak on DSC thermograms obtained for ground corn starch extrudates. The authors concluded that the results indicate that starch granules were completely gelatinized as a result of extrusion, or starch molecules might be broken down or depolymerized during heat shearing, or that DSC was not sensitive enough to detect the few granules that remained intact after processing. However, they found that an endotherm on DSC thermograms at approximately 110°C. The authors concluded that this endotherm shows the possible formation of amylose-lipid complexes during extrusion based on literature information. It has been reported that the higher temperature and low moisture content strongly contributed to starch degradation and favored amylose-lipid complex formation. This was explained such that with fewer water molecules surrounded by amylose chains might be able to come into closer contact with lipids, hence increasing complex formation and also high temperatures might favor molecular movement and generate higher complexation (Ho and Izzo, 1992; Blanche and Sun, 2004). Bindzus et al. (2002) suggested that during extrusion, starch undergoes a melting process, which results in a loss of partially crystalline structure. Following melting of the starch, high shear and high temperature conditions result in molecular fragmentation and formation of amylose-lipid complexes. Lee et al. (1999) found no endothermic peak of corn starch extrudates produced at 90 and 100°C on DSC thermograms. Although no gelatinization peak was detected, an endotherm was observed on all selected barley extrudates at 84.0-90.9°C while 80.7-88.4°C and 89.0-90.1°C for barley-tomato pomace and barley-grape pomace extrudates, respectively. It has been reported that cereal starches containing natural fatty acids and amylose form an amylose-lipid complex when extruded (Mercier et al. 1980). Hagenimana et al. (2006) also observed amylose-lipid complexes at 103°C on rice flour extrudates analyzed by differential scanning calorimeter. Bhatnagar and Hanna (1994) reported that extrusion temperature was the most significant factor affecting amylose-lipid complex formation. Highest levels of lipid binding occurred at barrel temperatures of 110-140°C, 140 rpm screw speed and 19% feed moisture content according to these authors. They found the melting temperature of complexes was around 107°C. Schweizer et al. (1986) found the second endothermic transition only for extruded and drum-dried wheat flour with and without soya oil (2%) and linoleic acid (1%) that demonstrated complete gelatinization of their starch. The transition

temperatures of amylose-lipid complexes were in the range of 87.6-84.1 for extruded flour and flour with addition of lipids while this range was 93.7-92.3 for drum-dried wheat flour and flour with addition of lipids.

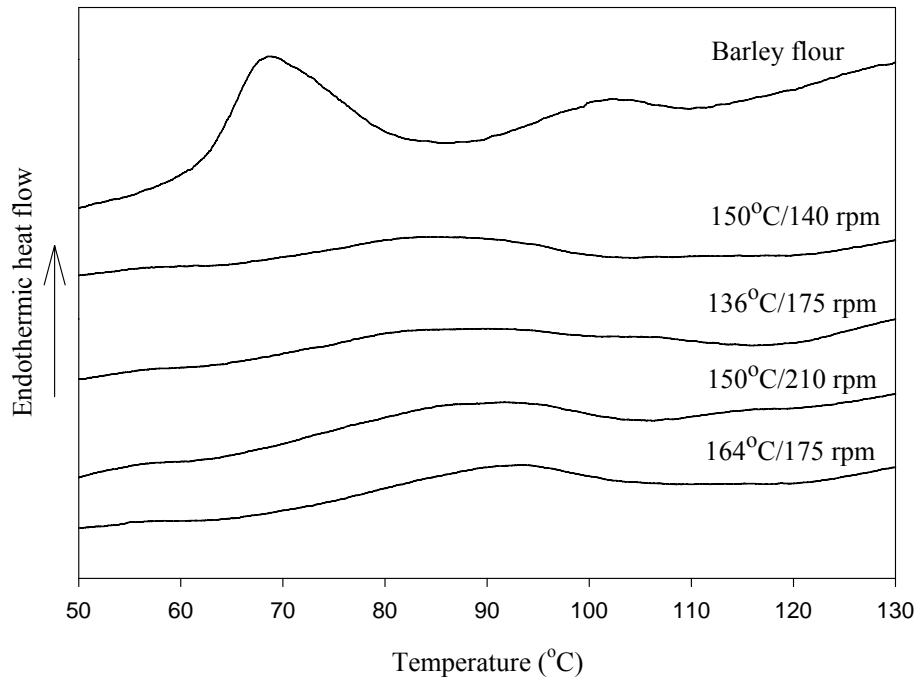


Figure 4.42 Differential scanning calorimetry thermograms of barley flour and barley flour extrudates produced at different extrusion conditions

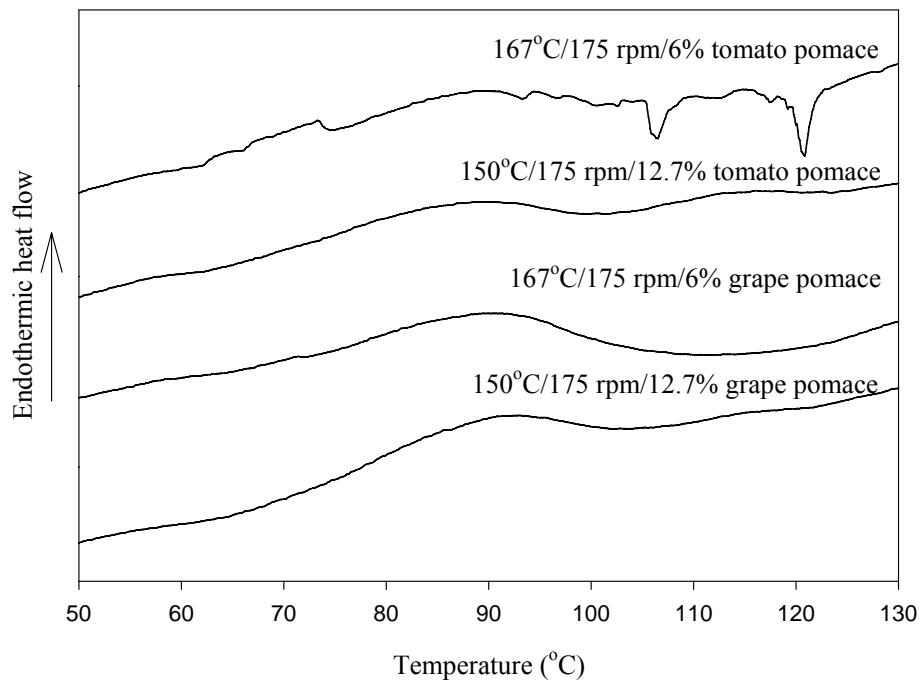


Figure 4.43 Differential scanning calorimetry thermograms of barley-tomato pomace and barley-grape pomace extrudates produced at different extrusion conditions

Starch polymers (amylose and amylopectin) are tightly packed in granules with a high degree of molecular order and are associated by hydrogen bonding. Raw granules contain highly crystalline regions and are birefringent in polarized light. The granules are insoluble in cold water. When exposed to heat in the presence of water, the granules undergo an irreversible swelling and destruction of the internal crystalline structure and birefringence is lost. This transformation is termed gelatinization (Holm et al. 1988). Figure 4.44 displays the typical birefringence pattern observed in raw barley flour. The process of gelatinization leads to loss of birefringence and hence a loss of the Maltese cross pattern depicted in Figures 4.45-4.47. The results obtained through measurements of iodine complexing method indicated that there was still an ungelatinized starch granule although no gelatinization peak was observed by DSC. This was confirmed by microscopic examination of the sample, which revealed the partial existence of Maltese cross in barley, barley-tomato pomace and barley-grape pomace extrudates.

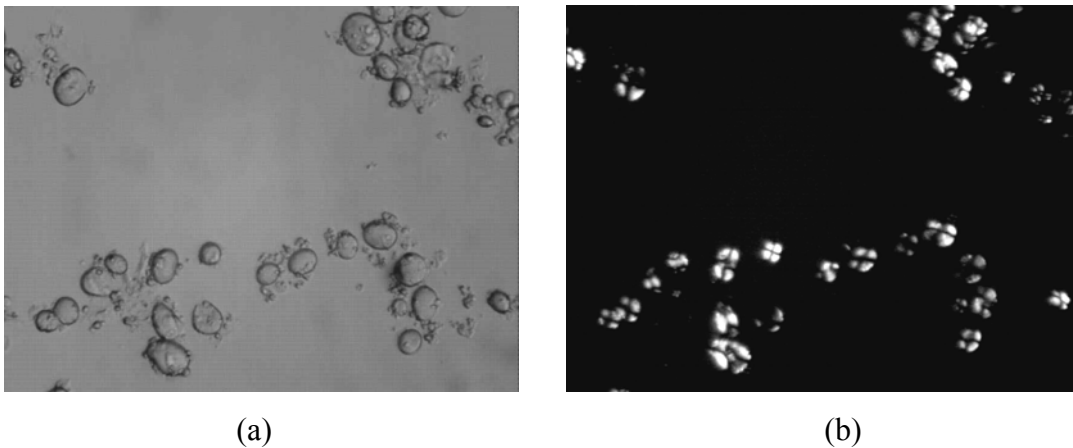
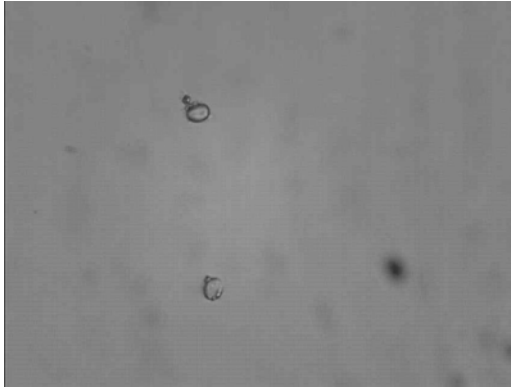


Figure 4.44 Image of barley flour under bright field (a) and polarized light (b)

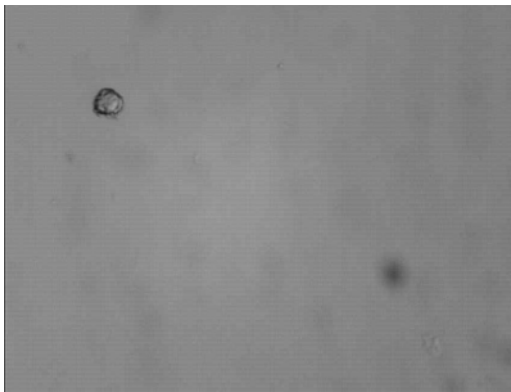


(a)



(b)

Figure 4.45 Image of barley extrudate produced at 164°C and 175 rpm screw speed under bright field (a) and polarized light (b)

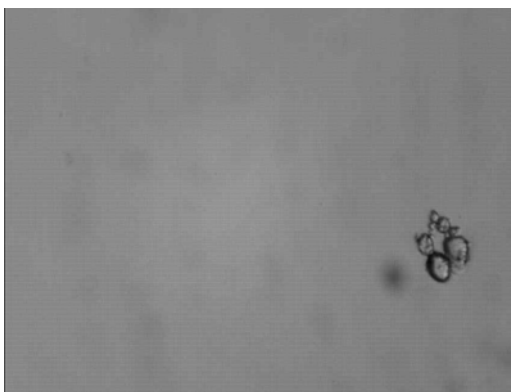


(a)

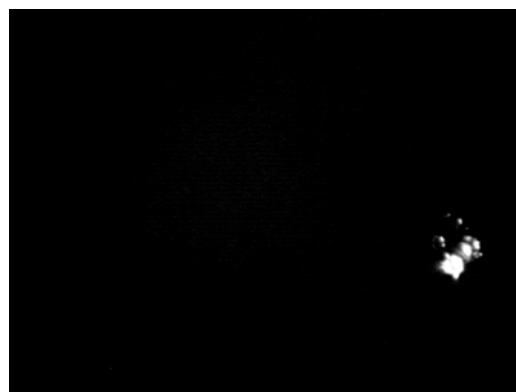


(b)

Figure 4.46 Image of barley-tomato pomace extrudate produced at 160°C, 150 rpm screw speed and 2% tomato pomace under bright field (a) and polarized light (b)



(a)



(b)

Figure 4.47 Image of barley-grape pomace extrudate produced at 167°C, 175 rpm screw speed and 6% grape pomace under bright field (a) and polarized light (b)

4.6.3 *In vitro* starch digestibility

Gelatinization destroys the compact granular structure by breaking of inter- and intramolecular hydrogen bonds and allows different degrees of swelling and absorption of water as a result fully hydrated starch molecules leach from granule. Therefore, the availability of starch granules to digestive enzymes increases to different levels with increasing degree of gelatinization (Holm et al. 1988). The regression equations for the relationship between *in vitro* starch digestibility and independent variables of temperature and tomato and grape pomace levels obtained in terms of coded variables are presented in Table 4.10. The coefficients of determination were 0.883 and 0.782 for barley-tomato pomace and barley-grape pomace extrudates with a low value of coefficient of variation (4.49 and 6.82%) of the predicted model, respectively. However, the lack-of-fit was significant ($P < 0.05$) for model of barley-tomato pomace extrudate. Linear effect of temperature and tomato pomace level as well as the quadratic effect of temperature were found to significantly influence *in vitro* starch digestibility of barley flour-tomato pomace extrudates at $P < 0.05$. The interaction term between temperature and tomato pomace level was also significant ($P < 0.05$) for barley-tomato pomace extrudates. Both the quadratic effects of temperature and grape pomace level in addition to linear effect of pomace level were significant ($P < 0.05$) on *in vitro* starch digestibility of barley flour-grape pomace extrudates. Screw speed had no significant effect on starch digestibility of barley-pomace extrudates. Dahlin and Lorenz (1993) observed less pronounced and non significant effect of screw speed on carbohydrate digestibility of all grains studied.

There was no significant model that fit *in vitro* starch digestibility of barley extrudates. The starch digestibility of barley extrudates varied between 447.2 and 560.6 mg maltose per g dry sample while it was 169.5 mg maltose per g dry sample for barley flour. Extrusion cooking significantly increased *in vitro* digestibility of barley starch as in agreement with those of other authors who have found that extrusion treatment significantly increased the *in vitro* digestibility of pea starch (Alonso et al. 2000). They attributed the improvement in *in vitro* starch digestibility to the relatively high temperatures (145°C) coupled with high shear as a result of extrusion treatment. It has been reported that heat treatment increases susceptibility of starch to amylase by promoting hydration, loss of structural integrity and partial

solubilization of starch molecules (Onyango et al. 2004b). Østergård et al. (1989) found considerably higher *in vitro* susceptibility of starch to α -amylase in boiled or extruded barley flour than that of the corresponding raw flour. Gomez and Aguilera (1983) observed increased enzyme susceptibility of corn flour after extrusion due to gelatinization and degradation of starch. Gomez and Aguilera (1984) reported that gelatinization introduced significant ultrastructural changes destroying the tight packing of polymers and permitting their release and therefore increased enzyme susceptibility when compared to raw material. Hagenimana et al. (2006) observed that extrusion cooking, in general, significantly increased starch digestibility of extrudates when compared to the unextruded rice flour, due to physical disruption of the organized granule structure and the degree of starch gelatinization characterized by the increase in viscosity as the crystalline starch molecules are disrupted and also due to the hydration of starch granules. Cheftel (1986) also stated that extrusion probably increased the enzymatic availability of starch by the way of gelatinization, inactivation of endogenous α -amylase inhibitor, disruption of cellular structure, size reduction and increased starch surface, partial separation from bran and protein. Asp and Bjöck (1984) found that high-temperature (170°C), high-shear extrusion of starch produced highly digestible carbohydrate. Wang et al. (1993b) determined higher values of enzyme-digestible starch for extruded whole wheat and wheat bran samples than that of raw samples. On the other hand, Lue et al. (1991) found that extrusion cooking did not change starch digestibility of products from corn meal and sugar beet fiber.

Changes in *in vitro* starch digestibility of barley-pomace blend extrudates as a function of temperature and pomace level are given in Figures 4.48 and 4.49. Starch digestibility of barley flour-tomato pomace extrudates decreased as temperature increased (Figure 4.48). Guha et al. (1997) found that digestibility increased with barrel temperature from 80 to 100°C then decreased. The authors attributed the reduction of digestibility to increasing temperature due to retrogradation or reassociation of gelatinized starch or formation of amylose-lipid complex, starch-protein complex or starch and thus these complexes caused to reduce the susceptibility of starch to enzyme hydrolysis. The reduction in starch digestibility could be attributed to formation of amylose-lipid complex based on the literature data (Asp and Bjöck, 1989; Bjöck et al. 1990). The formation of starch-lipid

complexes provides more stability and higher resistance to enzymatic hydrolysis (Jaisut et al. 2008). Jaisut et al. (2008) observed the lower rate of starch digestion for the thermal treated samples and this was attributed to the effect of amylose-lipid complexes as revealed by DSC. The amylose-lipid complex was observed from DSC measurements at 80.7-88.4°C for barley-tomato pomace extrudates while 89.0-90.1°C for barley-grape pomace extrudates (Table 4.12). Hagenimana et al. (2006) suggested that any lipid-amylose complexes formed during the extrusion process observed at 103°C might have played an important role in delaying starch hydrolysis. The authors also reported that the varying degrees of depolymerization or resistant starch formation resulting from extrusion cooking in addition to the complex formation might be attributed to different digestibility profiles in their study. The trend in starch digestibility as a function of temperature concurs with degree of gelatinization for barley-tomato pomace extrudates. The availability of the starch granules to digestive enzymes increases to different levels with increasing degree of gelatinization. It has been reported that increasing degree of gelatinization causes increased susceptibility to glucoamylase as well as a gradual loss of birefringence, indicating that the highly ordered structure within the granules was destroyed to different extents (Holm et al. 1988). Increasing temperature decreased *in vitro* starch digestibility of barley-grape pomace extrudates up to 155°C extrusion temperature then increased (Figure 4.49). This may be attributed to disruption of amylose-lipid complexes which have been shown to become dissociated at high temperatures (Bjöck et al. 1984) and become more susceptible to enzymic degradation (Bjöck et al. 1984; Mujoo and Ali, 1998). Bryant et al. (2001) found extrusion temperature as a most significant factor affecting digestibility of extruded rice flour. They observed that the flours extruded at 100°C digested slower than those extruded at 125 and 150°C.

Increasing level of both tomato and grape pomace led to reduction in starch digestibility (Figures 4.48 and 4.49). Cleary and Brennan (2006) observed that pastas with 5, 7.5 and 10% fiber fraction generally exhibited a significant decrease in starch digestibility. It was proposed that changes to the microstructure, which results in the entrapment of starch granules within a viscous protein-fiber-starch network, of cereal product in the presence of soluble fiber are responsible for reductions in digestibility (Tudorică et al. 2002; Cleary and Brennan, 2006). Other studies showed that the

limitation of water availability as a consequence of soluble non-starch polysaccharide hydration can restrict gelatinization of starch and hence reduce hydrolysis by α -amylase (Tester and Sommerville, 2003). Other than unique effect of fiber, with increasing level of both grape and tomato pomace levels, an increase in pomace level (Table 4.1), may cause increase in protein and lipid content of extrudates and therefore decreased digestibility of extrudates. Aarathi et al. (2003) reported that the presence of protein bodies around starch granules may restrict granule swelling and starch gelatinization, then, reduces the susceptibility to enzymatic attack. Svihus et al. (2005) reported that it is not possible to assume that there is always a linear relationship between extent of gelatinization of starch and availability of the starch for digestion and other components can interact with starch. It has been reported that processing may increase the amount of amylose-lipid complexes (Jacobs and Delcour, 1998) and thus may reduce digestibility of starch (Svihus et al. 2005). The interaction of temperature and tomato pomace was also significantly ($P < 0.05$) affected *in vitro* starch digestibility of barley-tomato pomace extrudates. High value of starch digestibility was obtained at 140°C and 2% tomato pomace level, whereas extrusion conditions at temperature of 160°C and 10% pomace level gave low value of *in vitro* starch digestibility for barley-tomato pomace extrudates (Figure 4.48).

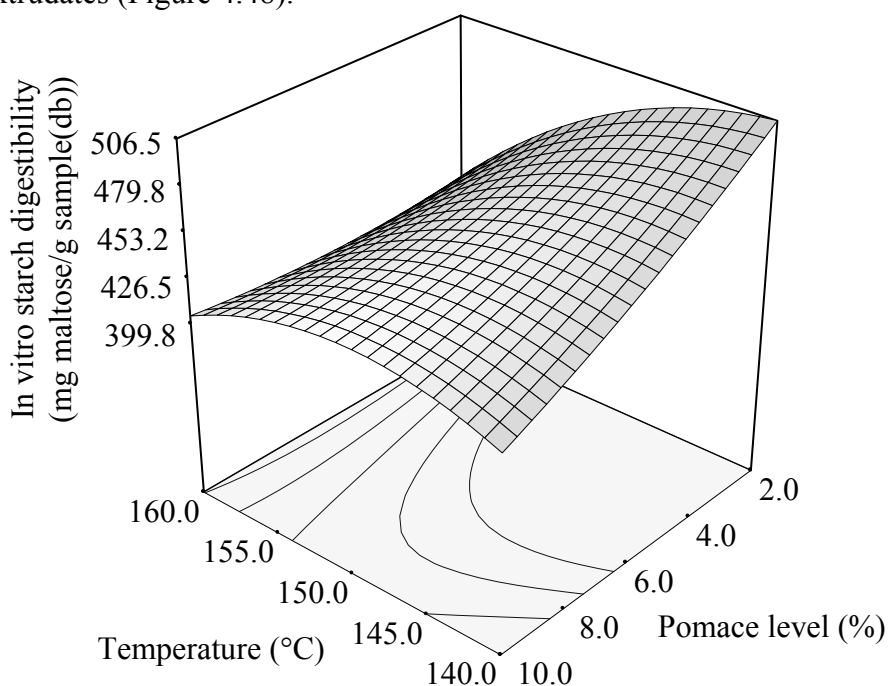


Figure 4.48 Response surface plot for the effect of pomace level and temperature on the *in vitro* starch digestibility of barley-tomato pomace extrudates at a screw speed of 175 rpm

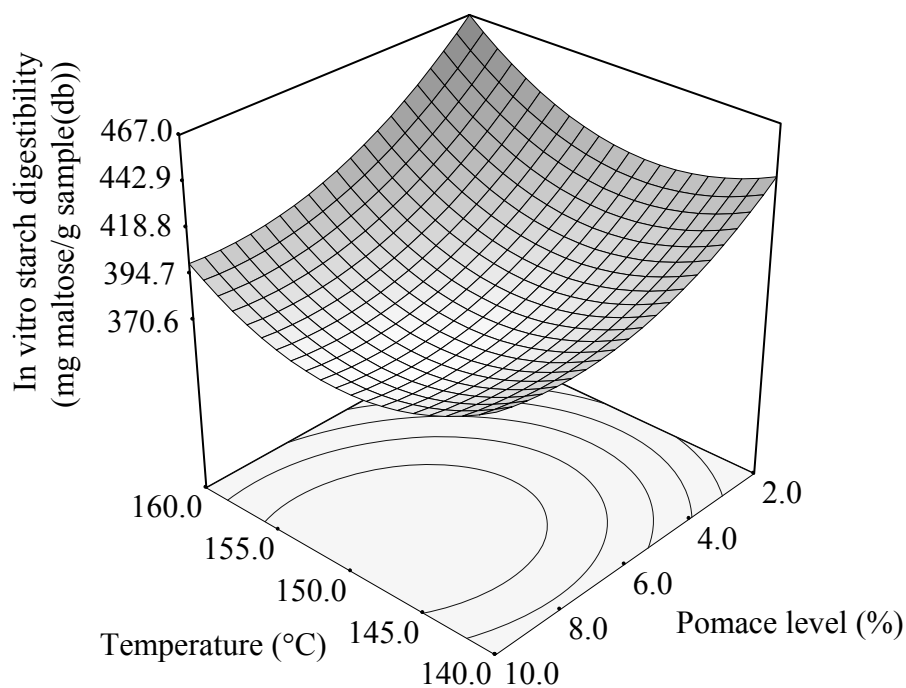


Figure 4.49 Response surface plot for the effect of pomace level and temperature on the *in vitro* starch digestibility of barley flour-grape pomace extrudates at a screw speed of 175 rpm

4.6.4 β -Glucan and dietary fiber contents

Multiple regression equations generated relating to β -glucan contents with coded levels of the variables are shown in Table 4.10 for barley flour and barley-pomace extrudates. Temperature had significant ($P < 0.05$) effect on β -glucan contents of barley flour and barley flour-grape pomace extrudates but β -glucan content of barley flour-tomato pomace extrudates was not significantly ($P > 0.05$) affected from temperature. The interaction term of temperature and screw speed was found to be significant ($P < 0.05$) for barley flour extrudates while β -glucan content of tomato pomace and grape pomace blends extrudates had significantly ($P < 0.05$) influenced from pomace level. The quadratic term of screw speed was significant ($P < 0.05$) for barley-grape pomace extrudates. The significance of models was given in ANOVA table (Table A.13). The lack-of-fit was not significant ($P > 0.05$) for barley flour and tomato pomace extrudates but significant ($P < 0.05$) for grape extrudates. Although the lack-of-fit was significant for grape pomace extrudates, coefficient of variance was found to be low (2.01%). The coefficients of variance for barley and barley-tomato pomace extrudates were 3.06 and 4.95%, respectively. The coefficients of

determination for β -glucan contents of barley flour, tomato pomace and grape pomace extrudates were 0.617, 0.688 and 0.821, respectively.

Changes in β -Glucan contents of barley flour extrudates affected by temperature and screw speed are shown in Figure 4.50. The β -glucan content of barley flour was 3.96% on a dry solid matter. Results showed that the content of β -glucan is higher in barley flour than in extrudates of barley flour and pomace blends. This suggests that extrusion cooking reduced β -glucan contents of barley flour extrudates and pomace blends extrudates. The reduction of barley β -glucan after extrusion is in agreement with the studies of Dudgeon-Bollinger et al. (1997) and Koksel et al. (2004). Dudgeon-Bollinger et al. (1997) investigated the β -glucan contents of whole-meal, pearled ground and high fiber barley and their extruded snacks processed at 300 rpm, cooking zone temperature of 100-150°C and moisture content of 12.6%. According to their results, extruded snacks had lower β -glucan contents when compared to the raw barley flour. This reduction has been attributed to destruction of β -glucan under high shear extrusion conditions. In another study, β -glucan contents of raw mixes and extruded cereals from blends of barley with four different cultivars and rice or wheat flour have been evaluated by Berglund et al. (1994). They found that β -glucan content of some cereals increased over that of the raw mixes but some of them had lower β -glucan content than that of raw mix. The authors couldn't explain the reason for the increase or decrease in β -glucan content from the raw mix to the extruded product. On the other hand, Yao et al. (2006) found that the content of β -glucan in extruded cereals was not affected by extrusion conditions.

The β -glucan content of barley flour extrudates decreased when temperature increased at low screw speeds rather than high screw speeds. This may be attributed to increased residence time at lower screw speeds causing destruction in barley β -glucan. Processing can produce substantial fragmentation of β -glucan (Yao et al. 2006) and hence lower the content of β -glucan. Extrusion induced β -glucan fragmentation or breakage of the glycosidic linkages has also been shown by Gaosong and Vasanthan (2000). Huth et al. (2000) reported that the molecular weights of β -glucans isolated from extrudates decreased as temperature increased when compared to that of β -glucan extracted from barley meal. Yao et al. (2006)

examined the impact of extrusion on the β -glucan content of oat. They reported that changing extrusion temperature or moisture content did not affect β -glucan concentration in the products. Our results are not in agreement with this finding. The effects of temperature and tomato pomace ratio on the β -glucan content are given in Figure 4.51. No significant effect of screw speed was observed in barley-tomato pomace extrudates. Increasing pomace level and temperature reduced the content of β -glucan but the reduction by temperature was not significant ($P>0.05$). Figures 4.52 and 4.53 show how processing variables affect the β -glucan content of barley-grape pomace extrudates. Both effect of pomace level and temperature were significant ($P<0.05$) for barley-grape pomace extrudates. Increasing the temperature and pomace level resulted in a decrease in β -glucan content of barley-grape pomace extrudates. The quadratic effect of screw speed dominated on β -glucan content of barley-grape pomace extrudates.

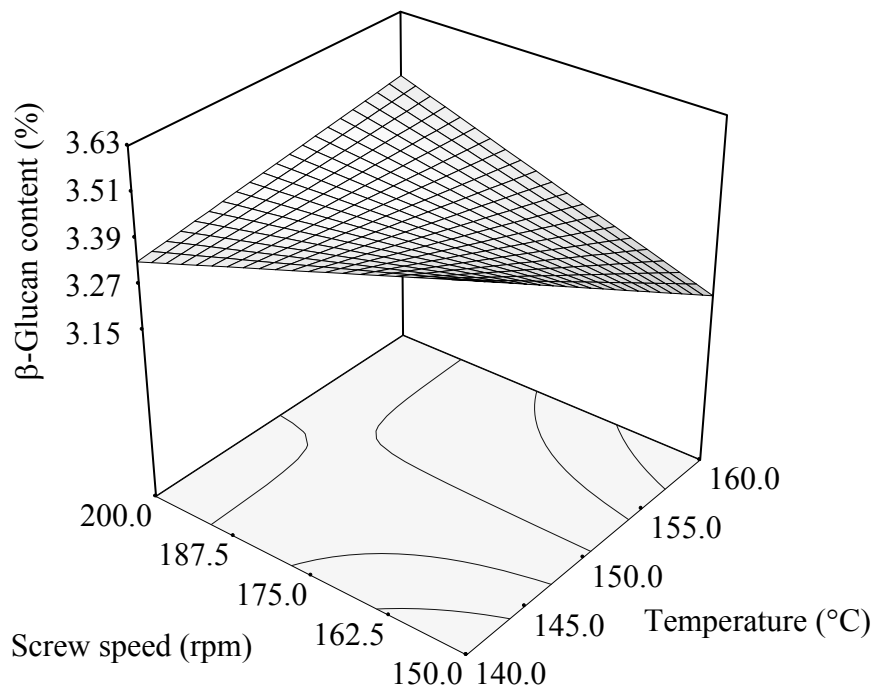


Figure 4.50 Response surface plot for the effect of temperature and screw speed on the β -glucan content of barley flour extrudates

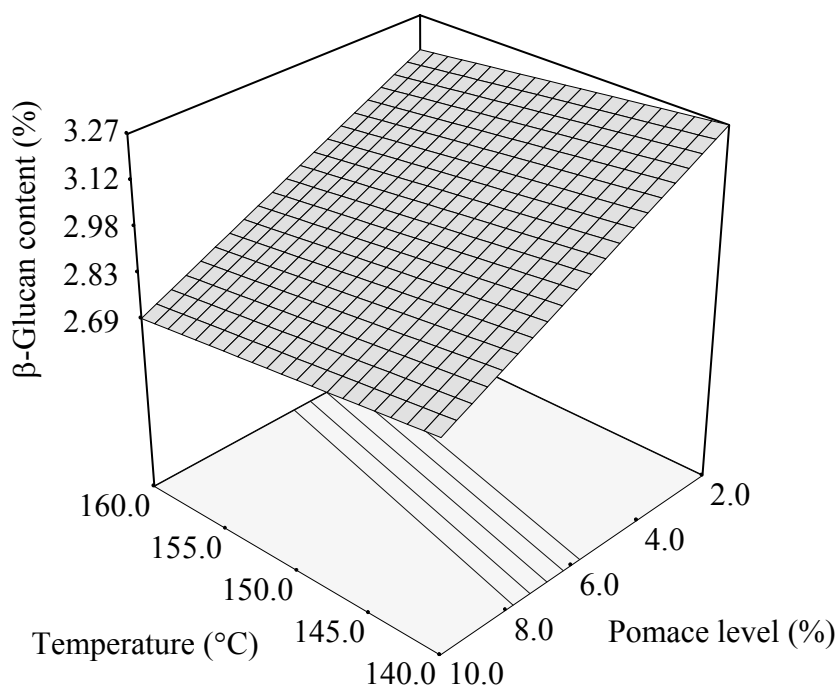


Figure 4.51 Response surface plot for the effect of pomace level and temperature on the β -glucan content of barley-tomato pomace extrudates at a screw speed of 175 rpm

As screw speed increased from 150 to 175 rpm, β -glucan content decreased but it increased when screw speed was increased further to 200 rpm. Both barley-tomato and barley-grape pomace extrudates were lower in β -glucan contents than were the barley extrudates due to the dilution effect of pomace addition. At high pomace level (10%), the barley-tomato pomace extrudates would provide approximately 2.8 g of β -glucan and 3.2 g for barley-grape pomace extrudates per 100 g serving. This value is very close or the same to the amount that determined by the Food and Drug Administration (FDA). In 2006, the FDA broadens the health claim to include whole grain barley and dry milled barley products as an additional source of β -glucan soluble fiber. FDA concluded that consuming whole grain barley and dry milled barley products that provide at least 3 g of β -glucan soluble fiber per day, is effective in lowering blood total and low density lipoprotein (LDL) cholesterol and that the cholesterol-lowering effects of β -glucan soluble fiber in barley products is comparable to that of oat sources of β -glucan fiber (FDA, 2006).

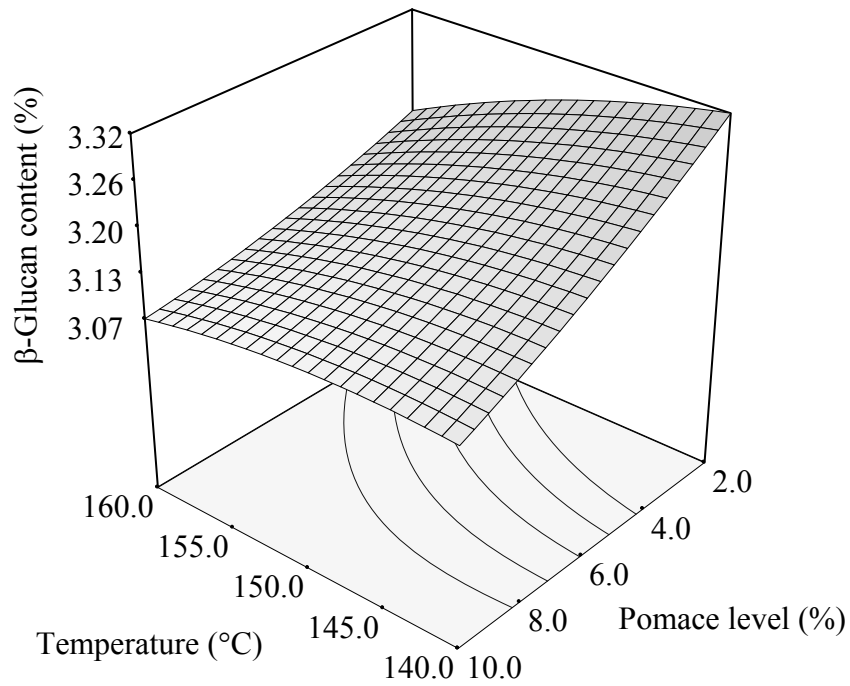


Figure 4.52 Response surface plot for the effect of pomace level and temperature on the β -glucan content of barley-grape pomace extrudates at a screw speed of 175 rpm

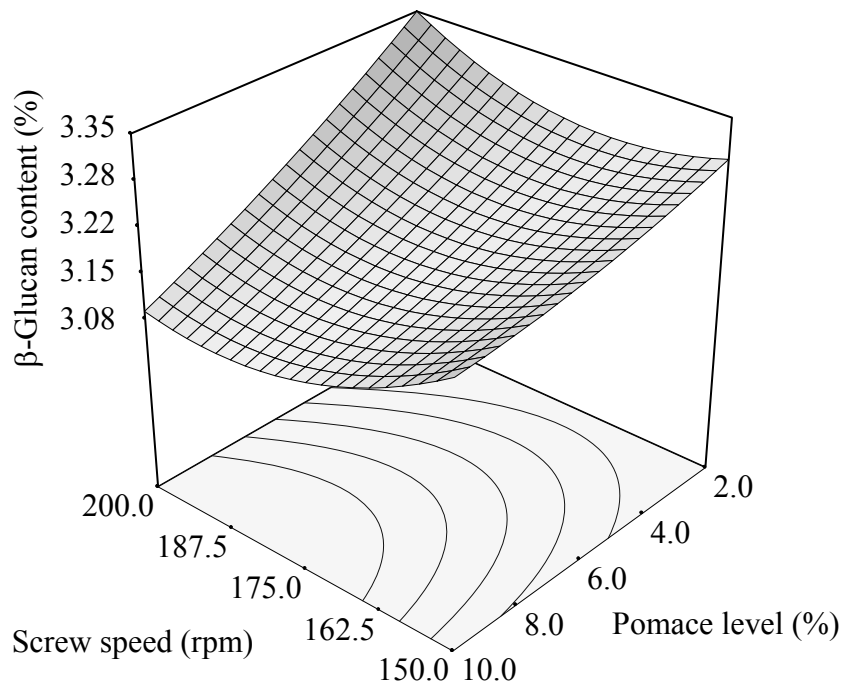


Figure 4.53 Response surface plot for the effect of pomace level and screw speed on the β -glucan content of barley-grape pomace extrudates at a temperature of 150 $^{\circ}$ C

Total dietary fiber content on a dry matter of barley flour, tomato pomace and grape pomace was 16.2, 52.8 and 12.3 %, respectively (Table 4.1). Figure 4.54 presents total dietary fiber content of barley-tomato pomace and barley-grape pomace extrudates at low and high levels of pomace ratio at 160°C. Total dietary fiber content was 18.1 to 20.3% and 15.1 to 15.5% for barley-tomato pomace and barley-grape pomace extrudates. Extrudates produced at 160°C and 2 and 10% of pomace levels gave higher sensory score by panelists with respect to appearance, taste, off-odor, texture and overall acceptability. The dietary fiber of these extrudates that preferred by panelists are 20.3 g and 15.5 g fiber in a 100 g serving (Figure 4.54). There are many studies supporting a role for dietary fiber in prevention and management of cardiovascular disease, diabetes and related conditions. In a study of Wolk et al. (1999) on 68,782 women showed that those with the highest total dietary fiber intake (quintile median 22.9 g/day) had a 47% lower risk for major cardiovascular events than women with the lowest fiber intake (quintile median 11.5 g/day). In another study on 43,757 male health professionals, the incidence of hearth attack and fatal coronary disease was found to be 41 and 55%, respectively, for those with the highest fiber intake (quintile median 28.9 g/day) compared with those with the lowest intake (quintile median 12.4 g/day) (Liu et al. 2002).

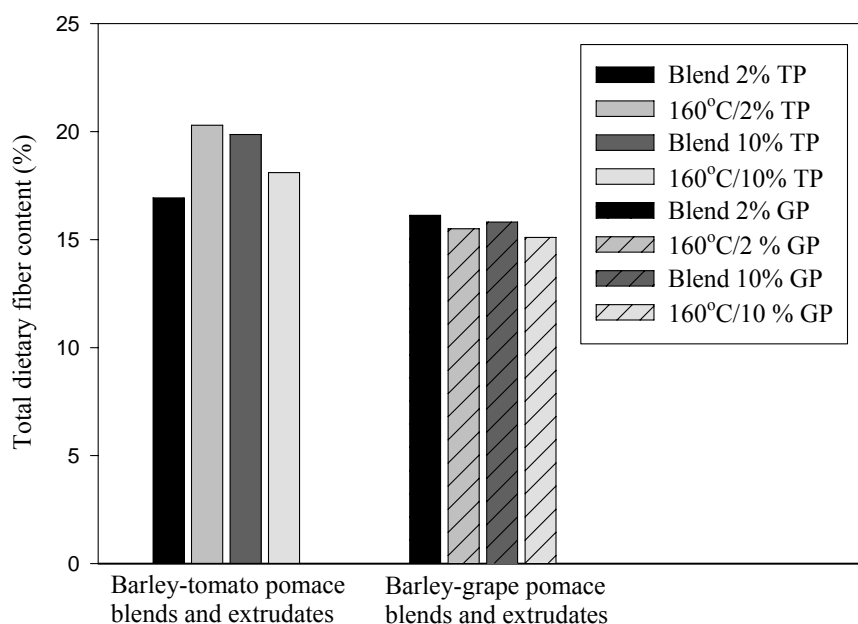


Figure 4.54 Total dietary fiber contents of barley-tomato pomace and barley-grape pomace extrudates processed at different pomace levels for both tomato pomace (TP) and grape pomace (GP) with constant screw speed and temperature of 150 rpm and 160°C

4.7 Optimization

Optimization can be defined as the processing conditions that give the optimum (maximum or minimum) value of a function of certain decided variables subject to constraints that are imposed. Optimization may be the process maximising a desired quantity or minimising an undesired one. The values of the processing variables that produce the desired optimum value are called optimum conditions (Myers and Montgomery, 2002). Product responses such as bulk density, texture and color were the most important major parameters determining quality of extrudates being consumed as snack foods. Therefore, optimum conditions for extrusion of barley flour were determined to obtain minimum bulk density, peak force, slope and distance and maximum Hunter L with minimum Hunter a value. The lower peak force indicates the lower hardness, whereas the lower slope shows the higher crispness of product. The shortest distance would produce the most brittle product. According to the results of sensory analysis of extrudates, numeric limits were selected on the products with higher sensory preference for optimization process. To determine the optimum extrusion conditions, response surface of desirability function was used for numerical optimization.

The desirability function of response surface is shown in Figure 4.55 to obtain optimum conditions in extrusion cooking of barley flour. By applying desirability function method, covering our criteria, one solution was obtained for the optimum conditions to produce barley extrudates. The desirability value obtained was 0.658. The optimum temperature and screw speed estimated were 156°C and 166 rpm, respectively. By applying these optimum conditions, an edible barley extrudate with a bulk density equal to 0.332 g/cm³, peak force (hardness) 4.99 N, slope (crispness) 6.25 N/mm, distance (brittleness) 0.86 mm, Hunter L 76.27, Hunter a 2.56, Hunter b 15.24 and ΔE of 6 could be produced.

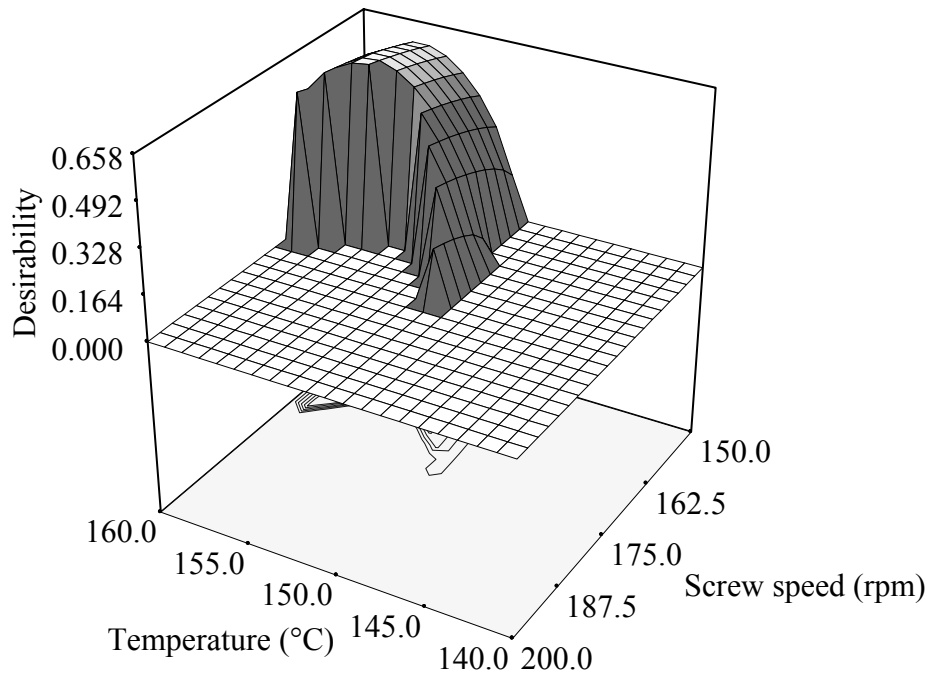


Figure 4.55 Desirability function response surface plot for barley flour extrudates

The main criteria for constraints optimization were bulk density, peak force, slope and distance to be as low as possible; high L and low a values and low ΔE for barley-pomace extrudates. The desirability functions of response surface of barley-tomato pomace extrudate are shown in Figures 4.56 and 4.57. Nine solutions were obtained by applying the methodology of the desirability function for barley-tomato pomace extrudates. The optimal values for considered factors can be one of nine solutions. The final decision of the optimal conditions depends on effects in the sensory characteristics of the product. Therefore, the optimum results were found by the numerical optimization method at the following level of process condition: 157°C for temperature, 188 rpm for screw speed and 4.8% for pomace level in order to obtain bulk density equal 0.449 g/cm³, peak force (hardness) 5.01 N, slope (crispness) 5.82 N/mm, distance (brittleness) 0.97 mm, Hunter L 70.97, Hunter a 8.81, Hunter b 21.83 and ΔE of 8.5. The desired function has a value of 0.651.

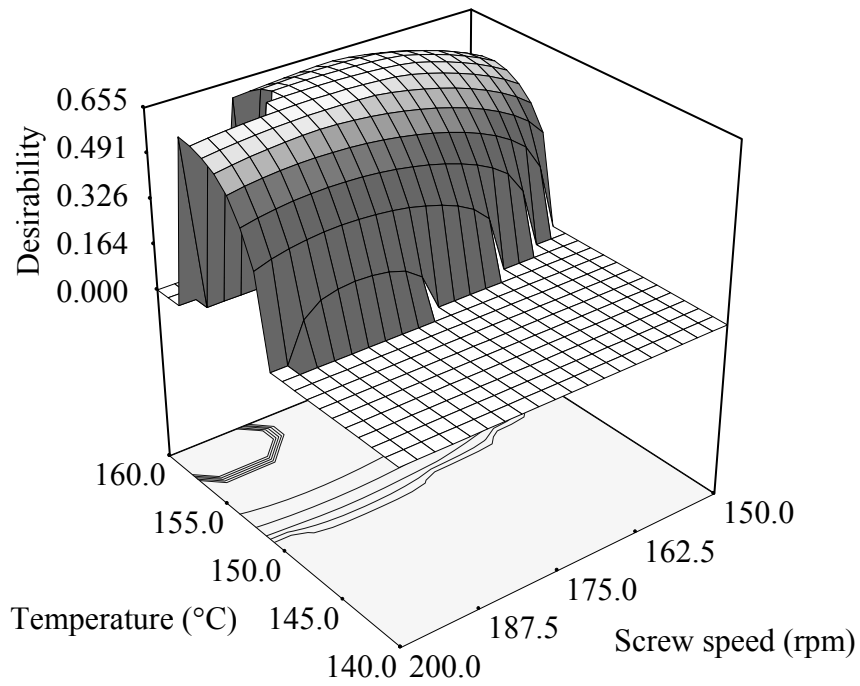


Figure 4.56 Desirability function response surface plot as a function of screw speed and temperature for barley-tomato pomace extrudates at a pomace level of 4.8%

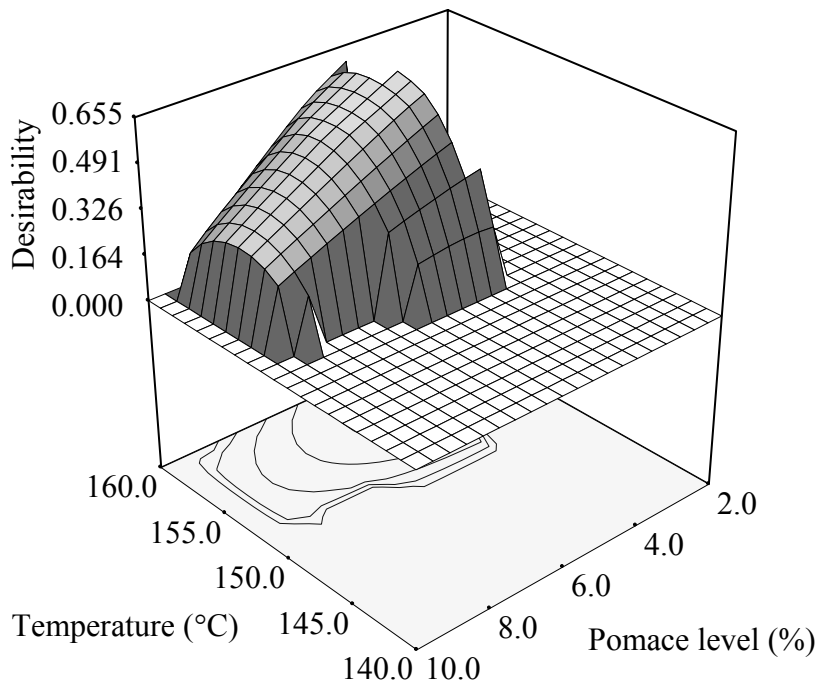


Figure 4.57 Desirability function response surface plot as a function of pomace level and temperature for barley-tomato pomace extrudates at a screw speed of 188 rpm

The desirability functions of response surface of barley-grape pomace extrudate are shown in Figures 4.58 and 4.59. By applying desirability function method, two solutions were obtained for the optimum covering the criteria. The one was 160°C for temperature, 179 rpm for screw speed and 5.4% for pomace level. The second was 160°C for temperature, 159 rpm for screw speed and 4.9% for pomace level. The desirability values of first and second solutions were 0.659 and 0.649, respectively. The results indicated that an increase in bulk density, peak force, slope and Hunter *L* value was observed in second solution. Also, desirability value of the first solution was greater than the second. So, the factor level combinations obtained at the first solution were selected as the optimum. The optimum product properties in terms of bulk density 0.425 g/cm³, peak force (hardness) 2.98 N, slope (crispness) 4.50 N/mm, distance (brittleness) 0.83 mm, Hunter *L* 73.46, Hunter *a* 3.70, Hunter *b* 16.05 and ΔE of 5.5 were obtained at temperature of 160°C, screw speed of 179 rpm and pomace level of 5.4% for barley-grape pomace extrudates.

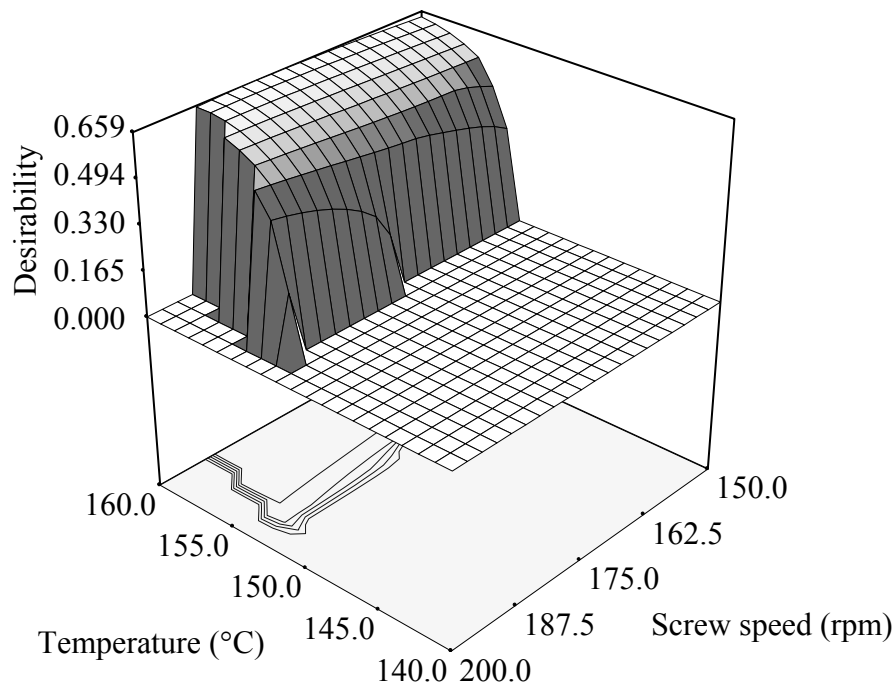


Figure 4.58 Desirability function response surface plot as a function of screw speed and temperature for barley-grape pomace extrudates at a pomace level of 5.4%

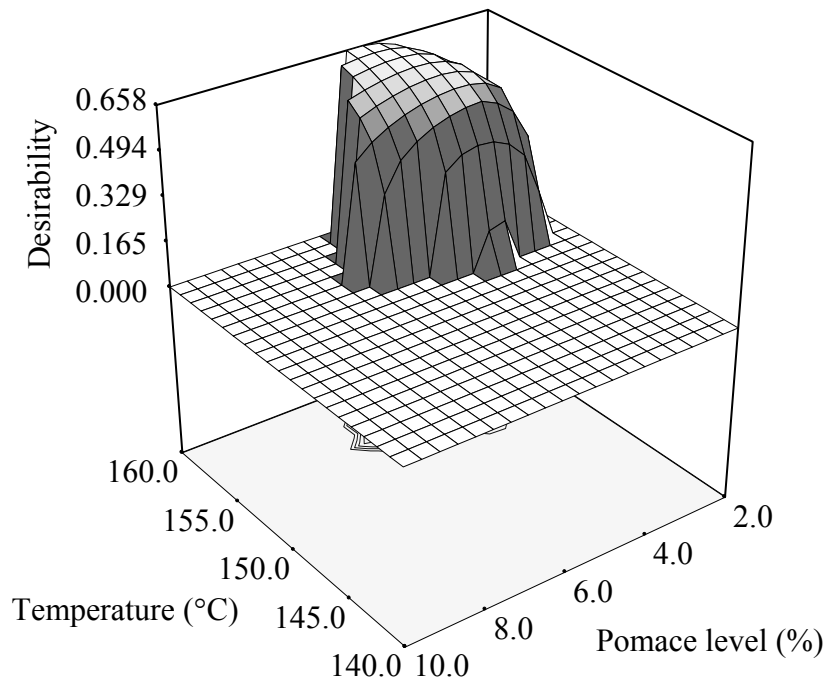


Figure 4.59 Desirability function response surface plot as a function of pomace level and temperature for barley-grape pomace extrudates at a screw speed of 179 rpm

CHAPTER 5

CONCLUSIONS

This research attempted to evaluate the production of extruded barley-based snack foods by incorporation of different type of pomaces. Extrusion of barley flour and barley flour-pomace blends produced extrudates with acceptable physical and sensorial characteristics. The different polynomial models were developed to describe the effect of each independent variable on system parameters and product properties. Correlations between system parameters and product responses, and also textural properties and sensory characteristics allowed better understanding of effect of independent variables on extrudate characteristics. Response surface methodology was used to determine the optimum processing conditions that yield minimum bulk density, peak force (low hardness), slope (high crispness) and distance (high brittleness) and maximum Hunter L with minimum Hunter a , b and ΔE values.

The optimum combination of process variables for the production of acceptable barley extrudates with twin-screw extruder was temperature of 156°C and screw speed of 166 rpm. The optimal conditions for production of barley-tomato pomace extrudates were 157°C, 188 rpm and 4.8% pomace level. Similarly, the optimum processing conditions were obtained as 160°C, 179 rpm and 5.4% pomace level to produce barley-grape pomace extrudates with acceptable properties.

The results suggest that it is possible to produce extruded foods from barley flour and blends of barley flour-pomace. The incorporation of tomato or grape pomace is a suitable means to enrich barley-based extruded snack foods with dietary fiber including β -glucan. This also enables favorable reception of barley by consumers. Considering the consumers current trend towards healthy food, it can be suggested that consumers can be motivated to use more barley in their diets. Increasing barley demand also results in increasing manufacturer's awareness and visibility of barley as a human food.

5.1 Recommendations for Future Work

Expansion is an important physical property for extrudates being consumed as snack food and depends on many factors such as rheological properties. Rheological properties should be significant in terms of controlling extrudate expansion. Thus there is a need to investigate dough flow rheology under typical extrusion conditions and pomace level for complete understanding of system parameters and extrudate characteristics that affected by independent variables.

Microstructure of extrudates should be studied to observe how structure of extrudate affected by temperature, screw speed and incorporation of pomace for better understanding of expansion, bulk density and texture. The examination of possible protein-fiber-starch or starch-protein network during extrusion would be very useful to recognize the effect of those on gelatinization and starch digestibility as well.

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APPENDIX A

Table A.1 Analysis of variance results for fitted models of system parameters for barley, barley-tomato pomace and barley-grape pomace extrudates

Response	Source	df	Sum of squares	Mean squares	F-value	P-value
SME _B	Regression	5	7102.19	1420.44	10.48	0.0038*
	Lack of fit	3	526.19	175.40	1.66	0.3107
	Pure error	4	422.16	105.54		
	Residual	7	948.35	135.48		
	Total	12	8050.54			
SME _T	Regression	9	41266.68	4585.19	11.86	0.0003*
	Lack of fit	5	669.03	133.81	0.21	0.9445
	Pure error	5	3198.53	639.71		
	Residual	10	3867.56	386.76		
	Total	19	45134.24			
SME _G	Regression	3	20596.02	6865.34	9.75	0.0007*
	Lack of fit	11	11063.65	1005.79	24.59	0.0012*
	Pure error	5	204.53	40.91		
	Residual	16	11268.18	704.26		
	Total	19	31864.20			
DP _B	Regression	5	3.435x10 ⁶	6.870x10 ⁵	3.98	0.0497*
	Lack of fit	3	1.158x10 ⁶	3.860x10 ⁶	31.10	0.0031*
	Pure error	4	4.965x10 ⁴	1.241x10 ⁴		
	Residual	7	1.208x10 ⁶	1.725x10 ⁶		
	Total	12	4.642x10 ⁶			
DP _T	Regression	6	3.820x10 ⁷	6.367x10 ⁶	15.07	<0.0001*
	Lack of fit	8	5.201x10 ⁶	6.501x10 ⁵	11.10	0.0084*
	Pure error	5	2.928x10 ⁵	58551.11		
	Residual	13	5.493x10 ⁶	4.226x10 ⁵		
	Total	19	4.370x10 ⁷			
DP _G	Regression	9	6.803x10 ⁶	7.559x10 ⁵	3.15	0.0443*
	Lack of fit	5	1.949x10 ⁶	3.897x10 ⁵	4.30	0.0678
	Pure error	5	4.535x10 ⁵	9.069x10 ⁴		
	Residual	10	2.402x10 ⁶	2.402x10 ⁵		
	Total	19	9.205x10 ⁶			

* significant at P<0.05, df: degrees of freedom, B: barley flour, T: tomato pomace, G: grape pomace

Table A.2 Correlation coefficients between product responses and system parameters for barley extrudates

	SEI	BD	WAI	WSI	L	A	b	ΔE	PF	SME	DP	T
SEI	1	-0.145 ^{ns}	0.093 ^{ns}	0.620 [*]	-0.341 ^{ns}	0.611 [*]	0.663 [*]	0.519 ^{ns}	-0.275 ^{ns}	0.294 ^{ns}	0.809 ^{**}	-0.252 ^{ns}
BD		1	-0.526 ^{ns}	0.076 ^{ns}	0.825 ^{**}	-0.318 ^{ns}	-0.367 ^{ns}	-0.742 ^{**}	0.984 ^{**}	-0.225 ^{ns}	0.203 ^{ns}	-0.868 ^{**}
WAI			1	-0.273 ^{ns}	-0.615 [*]	0.138 ^{ns}	0.173 ^{ns}	0.527 ^{ns}	-0.496 ^{ns}	-0.108 ^{ns}	-0.066 ^{ns}	0.590 [*]
WSI				1	-0.057 ^{ns}	0.383 ^{ns}	0.504 ^{ns}	0.265 ^{ns}	-0.017 ^{ns}	0.804 ^{**}	0.601 [*]	-0.270 ^{ns}
L					1	-0.591 [*]	-0.673 [*]	-0.964 ^{**}	0.815 ^{**}	-0.326 ^{ns}	-0.197 ^{ns}	-0.637 [*]
a						1	0.967 ^{**}	0.701 ^{**}	-0.365 ^{ns}	0.328 ^{ns}	0.503 ^{ns}	0.146 ^{ns}
b							1	0.804 ^{**}	-0.415 ^{ns}	0.467 ^{ns}	0.592 [*]	0.149 ^{ns}
ΔE								1	-0.760 ^{**}	0.435 ^{ns}	0.380 ^{ns}	0.496 ^{ns}
H									1	-0.279 ^{ns}	0.111 ^{ns}	-0.805 ^{**}
SME										1	0.310 ^{ns}	0.132 ^{ns}
DP											1	-0.560 [*]
T												1

SEI: sectional expansion index; BD: bulk density; WAI: water absorption index; ΔE : total color change; PF: peak force; SME: specific mechanical energy; DP: die pressure; T: die melt temperature; * significant at P<0.05, ** significant at P<0.01, ^{ns} not significant

Table A.3 Correlation coefficients between product responses and system parameters for barley-tomato pomace extrudates

	SEI	BD	WAI	WSI	L	A	b	ΔE	PF	SME	DP	T
SEI	1	0.219 ^{ns}	0.149 ^{ns}	0.149 ^{ns}	0.502 [*]	-0.512 [*]	-0.441 ^{ns}	-0.236 ^{ns}	0.282 ^{ns}	0.382 ^{ns}	0.754 ^{**}	-0.494 [*]
BD		1	0.212 ^{ns}	0.542 [*]	-0.106 ^{ns}	0.113 ^{ns}	0.180 ^{ns}	0.376 ^{ns}	0.925 ^{**}	0.152 ^{ns}	0.351 ^{ns}	-0.644 ^{**}
WAI			1	-0.184 ^{ns}	0.508 [*]	-0.548 [*]	-0.543 [*]	-0.269 ^{ns}	0.397 ^{ns}	-0.133 ^{ns}	0.188 ^{ns}	-0.091 ^{ns}
WSI				1	-0.462 [*]	0.433 ^{ns}	0.476 [*]	0.309 ^{ns}	0.467 [*]	0.782 ^{**}	0.488 [*]	-0.618 ^{**}
L					1	-0.988 ^{**}	-0.974 ^{**}	-0.721 ^{**}	-0.031 ^{ns}	-0.271 ^{ns}	-0.089 ^{ns}	0.296 ^{ns}
a						1	0.992 ^{**}	0.722 ^{**}	0.038 ^{ns}	0.212 ^{ns}	0.080 ^{ns}	-0.296 ^{ns}
b							1	0.752 ^{**}	0.110 ^{ns}	0.240 ^{ns}	0.171 ^{ns}	-0.364 ^{ns}
ΔE								1	0.319 ^{ns}	0.131 ^{ns}	0.235 ^{ns}	-0.323 ^{ns}
H									1	0.134 ^{ns}	0.444 ^{ns}	-0.637 ^{**}
SME										1	0.564 ^{**}	-0.533 [*]
DP											1	-0.777 ^{**}
T												1

SEI: sectional expansion index; BD: bulk density; WAI: water absorption index; ΔE : total color change; PF: peak force; SME: specific mechanical energy; DP: die pressure; T: die melt temperature; * significant at P<0.05, ** significant at P<0.01, ^{ns} not significant

Table A.4 Correlation coefficients between product responses and system parameters for barley-grape pomace extrudates

	SEI	BD	WAI	WSI	L	A	b	ΔE	PF	SME	DP	T
SEI	1	0.438 ^{ns}	0.209 ^{ns}	0.333 ^{ns}	0.122 ^{ns}	-0.075 ^{ns}	-0.037 ^{ns}	-0.140 ^{ns}	0.382 ^{ns}	0.407 ^{ns}	0.651 ^{**}	-0.578 ^{**}
BD		1	-0.207 ^{ns}	0.478 [*]	-0.253 ^{ns}	0.196 ^{ns}	0.338 ^{ns}	-0.069 ^{ns}	0.859 ^{**}	0.556 [*]	0.389 ^{ns}	-0.906 ^{**}
WAI			1	-0.511 [*]	0.453 [*]	-0.589 ^{**}	-0.592 ^{**}	0.032 ^{ns}	0.025 ^{ns}	0.085 ^{ns}	-0.113 ^{ns}	-0.009 ^{ns}
WSI				1	-0.572 [*]	0.678 ^{**}	0.701 ^{**}	0.115 ^{ns}	0.226 ^{ns}	0.421 ^{ns}	0.520 [*]	-0.292 ^{ns}
L					1	-0.905 ^{**}	-0.877 ^{**}	-0.657 ^{**}	0.070 ^{ns}	-0.025 ^{ns}	-0.192 ^{ns}	0.029 ^{ns}
a						1	0.964 ^{**}	0.459 [*]	-0.137 ^{ns}	0.048 ^{ns}	0.316 ^{ns}	0.071 ^{ns}
b							1	0.462 [*]	0.055 ^{ns}	0.151 ^{ns}	0.375 ^{ns}	-0.056 ^{ns}
ΔE								1	-0.021 ^{ns}	-0.022 ^{ns}	0.075 ^{ns}	0.122 ^{ns}
H									1	0.545 [*]	0.334 ^{ns}	-0.851 ^{**}
SME										1	0.549 [*]	-0.637 ^{**}
DP											1	-0.522 [*]
T												1

SEI: sectional expansion index; BD: bulk density; WAI: water absorption index; ΔE : total color change; H: hardness; SME: specific mechanical energy; DP: die pressure; T: die melt temperature; * significant at P<0.05, ** significant at P<0.01, ^{ns} not significant

Table A.5 Analysis of variance results for fitted models of sectional expansion index and bulk density for barley, barley-tomato pomace and barley-grape pomace extrudates

Response	Source	df	Sum of squares	Mean squares	<i>F-value</i>	<i>P-value</i>
SEI _B	Regression	5	0.70	0.14	6.42	0.0151*
	Lack of fit	3	0.081	0.027	1.52	0.3395
	Pure error	4	0.071	0.018		
	Residual	7	0.15	0.022		
	Total	12	0.85			
SEI _T	Regression	9	1.71	0.19	23.95	<0.0001*
	Lack of fit	5	0.071	0.014	9.00	0.0154*
	Pure error	5	7.913x10 ⁻³	1.583x10 ⁻³		
	Residual	10	0.079	7.914x10 ⁻³		
	Total	19	1.78			
SEI _G	Regression	9	0.78	0.087	6.14	0.0045*
	Lack of fit	5	0.14	0.028	44.20	0.0040*
	Pure error	5	3.134x10 ⁻³	6.268x10 ⁻⁴		
	Residual	10	0.14	0.014		
	Total	19	0.92			
BD _B	Regression	5	0.83	0.17	60.10	<0.0001*
	Lack of fit	3	0.013	4.174x10 ⁻³	2.43	0.2049
	Pure error	4	6.857x10 ⁻³	1.714x10 ⁻³		
	Residual	7	0.019	2.769x10 ⁻³		
	Total	12	0.85			
BD _T	Regression	9	0.50	0.055	13.58	0.0002*
	Lack of fit	5	0.033	6.611x10 ⁻³	4.29	0.0679
	Pure error	5	7.699x10 ⁻³	1.540x10 ⁻³		
	Residual	10	0.041	4.075x10 ⁻³		
	Total	19	0.54			
BD _G	Regression	9	1.08	0.12	67.10	<0.0001*
	Lack of fit	5	0.015	3.045x10 ⁻³	5.74	0.0389*
	Pure error	5	2.653x10 ⁻³	5.305x10 ⁻⁴		
	Residual	10	0.018	1.788x10 ⁻³		
	Total	19	1.10			

* significant at P<0.05, df: degrees of freedom, B: barley flour, T: tomato pomace, G: grape pomace

Table A.6 Analysis of variance results for fitted models of color parameters for barley, barley-tomato pomace and barley-grape pomace extrudates

Response	Source	df	Sum of squares	Mean squares	F-value	P-value
L _B	Regression	2	4.70	2.35	6.04	0.0191*
	Lack of fit	6	3.73	0.62	14.76	0.0106*
	Pure error	4	0.17	0.042		
	Residual	10	3.90	0.39		
	Total	12	8.60			
L _T	Regression	9	139.17	15.46	36.91	<0.0001*
	Lack of fit	5	1.98	0.40	0.90	0.5468
	Pure error	5	2.21	0.44		
	Residual	10	4.19	0.42		
	Total	19	143.36			
L _G	Regression	9	65.88	7.32	23.32	<0.0001*
	Lack of fit	5	1.64	0.33	1.10	0.4600
	Pure error	5	1.50	0.30		
	Residual	10	3.14	0.31		
	Total	19	69.02			
a _T	Regression	9	138.73	15.41	218.42	<0.0001*
	Lack of fit	5	0.42	0.085	1.50	0.3335
	Pure error	5	0.28	0.056		
	Residual	10	0.71	0.071		
	Total	19	139.44			
a _G	Regression	3	10.87	3.62	29.49	<0.0001*
	Lack of fit	11	1.48	0.13	1.37	0.3833
	Pure error	5	0.49	0.098		
	Residual	16	1.97	0.12		
	Total	19	12.84			
b _T	Regression	9	113.23	12.58	154.03	<0.0001*
	Lack of fit	5	0.68	0.14	5.07	0.0497*
	Pure error	5	0.13	0.027		
	Residual	10	0.82	0.082		
	Total	19	114.05			
b _G	Regression	3	11.47	3.82	28.04	<0.0001*
	Lack of fit	11	1.84	0.17	2.40	0.1721
	Pure error	5	0.35	0.069		
	Residual	16	2.18	0.14		
	Total	19	13.66			
ΔE _B	Regression	5	7.26	1.45	4.17	0.0445*
	Lack of fit	3	1.96	0.65	5.44	0.0677
	Pure error	4	0.48	0.12		
	Residual	7	2.43	0.35		
	Total	12	9.70			
ΔE _T	Regression	9	15.57	1.73	6.85	0.0029*
	Lack of fit	5	1.20	0.24	0.91	0.5409
	Pure error	5	1.32	0.26		
	Residual	10	2.52	0.25		
	Total	19	18.09			
ΔE _G	Regression	9	3.34	0.37	3.08	0.0474*
	Lack of fit	5	1.03	0.21	5.81	0.0380
	Pure error	5	0.18	0.035		
	Residual	10	1.21	0.12		
	Total	19	4.54			

*significant at P<0.05, df: degrees of freedom, B: barley flour, T: tomato pomace, G: grape pomace

Table A.7 Analysis of variance results for fitted models of functional properties for barley, barley-tomato pomace and barley-grape pomace extrudates

Response	Source	df	Sum of squares	Mean squares	<i>F-value</i>	<i>P-value</i>
WAI _T	Regression	3	0.76	0.25	6.70	0.0039*
	Lack of fit	11	0.46	0.041	1.40	0.3741
	Pure error	5	0.15	0.030		
	Residual	16	0.60	0.038		
	Total	19	1.36			
WAI _G	Regression	6	0.82	0.14	6.16	0.0030*
	Lack of fit	8	0.26	0.033	6.82	0.0245*
	Pure error	5	0.024	4.818x10 ⁻³		
	Residual	13	0.29	0.022		
	Total	19	1.10			
WSI _B	Regression	5	17.29	3.46	9.54	0.005*
	Lack of fit	3	0.31	0.10	0.19	0.9002
	Pure error	4	2.23	0.56		
	Residual	7	2.54	0.36		
	Total	12	19.83			
WSI _T	Regression	3	44.94	14.98	26.23	<0.0001*
	Lack of fit	11	8.47	0.77	5.74	0.0333*
	Pure error	5	0.67	0.13		
	Residual	16	9.14	0.57		
	Total	19	54.08			
WSI _G	Regression	9	54.19	6.02	7.11	0.0025*
	Lack of fit	5	2.74	0.55	0.48	0.7813
	Pure error	5	5.73	1.15		
	Residual	10	8.47	0.85		
	Total	19	62.66			

*significant at P<0.05, df: degrees of freedom, B: barley flour, T: tomato pomace, G: grape pomace

Table A.8 Analysis of variance results for fitted models of textural parameters for barley, barley-tomato pomace and barley-grape pomace extrudates

Response	Source	df	Sum of squares	Mean squares	F-value	P-value
PF _B	Regression	5	798.17	159.63	88.43	<0.0001*
	Lack of fit	3	12.52	4.17	140.09	0.0002*
	Pure error	4	0.12	0.030		
	Residual	7	12.64	1.81		
	Total	12	810.81			
PF _T	Regression	9	742.88	82.54	87.91	<0.0001*
	Lack of fit	5	9.34	1.87	176.61	<0.0001*
	Pure error	5	0.053	0.011		
	Residual	10	9.39	0.94		
	Total	19	752.27			
PF _G	Regression	9	992.74	110.30	32.23	<0.0001*
	Lack of fit	5	34.21	6.84	1796.05	<0.0001*
	Pure error	5	0.019	3.809x10 ⁻³		
	Residual	10	34.23	3.42		
	Total	19	1026.97			
S _B	Regression	5	201.53	40.31	65.60	<0.0001*
	Lack of fit	3	4.03	1.34	19.99	0.0072*
	Pure error	4	0.27	0.067		
	Residual	7	4.30	0.61		
	Total	12	205.83			
S _T	Regression	9	210.35	23.37	41.28	<0.0001*
	Lack of fit	5	4.55	0.91	4.10	0.0739
	Pure error	5	1.11	0.22		
	Residual	10	5.66	0.57		
	Total	19	216.01			
S _G	Regression	9	345.49	38.39	23.83	<0.0001*
	Lack of fit	5	15.93	3.19	87.82	<0.0001*
	Pure error	5	0.18	0.036		
	Residual	10	16.11	1.61		
	Total	19	361.60			
D _{iB}	Regression	5	0.36	0.073	7.70	0.0091*
	Lack of fit	3	0.060	0.020	12.69	0.0164*
	Pure error	4	6.306x10 ⁻³	1.576x10 ⁻³		
	Residual	7	0.066	9.474x10 ⁻³		
	Total	12	0.43			
D _{iT}	Regression	9	0.17	0.019	9.89	0.0007*
	Lack of fit	5	0.015	2.903x10 ⁻³	3.22	0.1127
	Pure error	5	4.51x10 ⁻³	9.02x10 ⁻⁴		
	Residual	10	0.019	1.902x10 ⁻³		
	Total	19	0.19			
D _{iG}	Regression	9	0.25	0.028	19.81	<0.0001*
	Lack of fit	5	0.012	2.354x10 ⁻³	5.13	0.0485*
	Pure error	5	2.295x10 ⁻³	4.589x10 ⁻⁴		
	Residual	10	0.014	1.407x10 ⁻³		
	Total	19	0.26			

*significant at P<0.05, df: degrees of freedom, B: barley flour, T: tomato pomace, G: grape pomace

Table A.9 Correlation matrix between sensory attributes and textural properties measured instrumentally for barley extrudates

	Peak force	Slope	Distance	S. Hard.	S. Crisp.	S. Britt.	S. Color	Porosity	Overall acceptability
Peak force	1	0.907*	-0.402 ^{ns}	-0.833*	-0.809 ^{ns}	-0.824*	-0.785 ^{ns}	-0.805 ^{ns}	-0.794 ^{ns}
Slope		1	-0.700 ^{ns}	-0.864*	-0.817*	-0.914*	-0.966**	-0.772 ^{ns}	-0.931**
Distance			1	0.526 ^{ns}	0.562 ^{ns}	0.694 ^{ns}	0.760 ^{ns}	0.391 ^{ns}	0.763 ^{ns}
Sensory Hardness				1	0.972**	0.965**	0.868*	0.969**	0.931**
Sensory Crispness					1	0.943**	0.797 ^{ns}	0.970**	0.924**
Sensory Brittleness						1	0.922**	0.883*	0.952**
Sensory Colour							1	0.755 ^{ns}	0.948**
Porosity								1	0.877*
Overall acceptability									1

*significant at P<0.05, ** significant at P<0.01, ^{ns} not significant

Table A.10 Correlation coefficients between sensory attributes and textural properties measured instrumentally for barley-tomato extrudates

	Peak force	Slope	Distance	S. Hard.	S. Crisp.	S. Britt.	S. Color	Porosity	Overall acceptability
Peak force	1	0.764 ^{ns}	-0.239 ^{ns}	-0.759 ^{ns}	-0.816 ^{ns}	-0.711 ^{ns}	-0.321 ^{ns}	-0.107 ^{ns}	-0.512 ^{ns}
Slope		1	-0.781 ^{ns}	-0.871 ^{ns}	-0.902 [*]	-0.832 ^{ns}	-0.360 ^{ns}	0.583 ^{ns}	-0.546 ^{ns}
Distance			1	0.727 ^{ns}	0.698 ^{ns}	0.738 ^{ns}	0.475 ^{ns}	-0.907 [*]	0.571 ^{ns}
Sensory Hardness				1	0.994 ^{**}	0.993 ^{**}	0.754 ^{ns}	-0.721 ^{ns}	0.884 [*]
Sensory Crispness					1	0.978 ^{**}	0.693 ^{ns}	-0.660 ^{ns}	0.838 ^{ns}
Sensory Brittleness						1	0.788 ^{ns}	-0.750 ^{ns}	0.916 [*]
Sensory Color							1	-0.747 ^{ns}	0.959 [*]
Porosity								1	-0.747 ^{ns}
Overall acceptability									1

* significant at P<0.05, ** significant at P<0.01, ^{ns} not significant

Table A.11 Correlation coefficients between sensory attributes and textural properties measured instrumentally for barley-grape extrudates

	Peak force	Slope	Distance	S. Hard.	S. Crisp.	S. Britt.	S. Color	Porosity	Overall acceptability
Peak force	1	0.941*	-0.483 ^{ns}	-0.958*	-0.943*	-0.950*	-0.874 ^{ns}	-0.840 ^{ns}	-0.984**
Slope		1	-0.735 ^{ns}	-0.931*	-0.911*	-0.920*	-0.979**	-0.957*	-0.979**
Distance			1	0.557 ^{ns}	0.491 ^{ns}	0.568 ^{ns}	0.791 ^{ns}	0.817 ^{ns}	0.589 ^{ns}
Sensory Hardness				1	0.987**	0.997**	0.836 ^{ns}	0.788 ^{ns}	0.937*
Sensory Crispness					1	0.972**	0.815 ^{ns}	0.753 ^{ns}	0.925**
Sensory Brittleness						1	0.822 ^{ns}	0.780 ^{ns}	0.923*
Sensory Color							1	0.993**	0.944*
Porosity								1	0.917*
Overall acceptability									1

* significant at P<0.05, ** significant at P<0.01, ^{ns} not significant

Table A.12 Analysis of variance results for fitted models of antioxidant activity, total phenolic content and degree of gelatinization for barley, barley-tomato pomace and barley-grape pomace extrudates

Response	Source	df	Sum of squares	Mean squares	<i>F-value</i>	<i>P-value</i>
AA _T	Regression	6	42.57	7.10	3.79	0.0209*
	Lack of fit	8	17.73	2.22	1.69	0.2929
	Pure error	5	6.57	1.31		
	Residual	13	24.31	1.87		
	Total	19	66.88			
AA _G	Regression	9	32.53	3.61	4.05	0.0199*
	Lack of fit	5	4.40	0.88	0.97	0.511
	Pure error	5	4.52	0.90		
	Residual	10	8.92	0.89		
	Total	19	41.45			
TP _G	Regression	3	1.26	0.42	6.90	0.0034*
	Lack of fit	11	0.72	0.065	1.26	0.4250
	Pure error	5	0.26	0.052		
	Residual	16	0.98	0.061		
	Total	19	2.24			
DG _B	Regression	5	358.78	71.76	8.60	0.0067*
	Lack of fit	3	38.84	12.95	2.64	0.1855
	Pure error	4	19.59	4.90		
	Residual	7	58.43	8.35		
	Total	12	417.21			
DG _T	Regression	9	1679.17	186.57	3.94	0.0218*
	Lack of fit	5	333.02	66.60	2.38	0.1820
	Pure error	5	140.16	28.03		
	Residual	10	473.17	47.32		
	Total	19	2152.34			
DG _G	Regression	9	1009.25	112.14	3.35	0.0365*
	Lack of fit	5	310.32	62.06	12.95	0.0069*
	Pure error	5	23.96	4.79		
	Residual	10	334.29	33.43		
	Total	19	1343.54			

*significant at $P < 0.05$, df: degrees of freedom, B: barley flour, T: tomato pomace, G: grape pomace

Table A.13 Analysis of variance results for fitted models of *in vitro* starch digestibility and β -glucan content for barley, barley-tomato pomace and barley-grape pomace extrudates

Response	Source	df	Sum of squares	Mean squares	F-value	P-value
SD _T	Regression	9	28847.01	3205.22	8.38	0.0013*
	Lack of fit	5	3233.53	646.71	5.48	0.0426*
	Pure error	5	589.69	117.94		
	Residual	10	3823.22	382.32		
	Total	19	32670.23			
SD _G	Regression	9	29169.77	3241.09	3.99	0.0210*
	Lack of fit	5	6383.83	1276.77	3.66	0.0906
	Pure error	5	1746.13	349.23		
	Residual	10	8129.97	813.00		
	Total	19	37299.74			
BG _B	Regression	9	0.16	0.052	4.83	0.0285*
	Lack of fit	5	0.084	0.017	5.24	0.0669
	Pure error	5	0.013	3.21x10 ⁻³		
	Residual	10	0.097	0.011		
	Total	19	0.25			
BG _T	Regression	3	0.76	0.25	11.74	0.0003*
	Lack of fit	5	0.31	0.029	4.20	0.0626
	Pure error	4	0.034	6.78x10 ⁻³		
	Residual	9	0.35	0.022		
	Total	12	1.11			
BG _G	Regression	3	0.19	0.021	5.10	0.0089*
	Lack of fit	11	0.041	8.137x10 ⁻³	39.03	0.0005*
	Pure error	5	1.043x10 ⁻³	2.085x10 ⁻⁴		
	Residual	16	0.042	4.173x10 ⁻³		
	Total	19	0.23			

*significant at P<0.05, df: degrees of freedom, B: barley flour, T: tomato pomace, G: grape pomace

APPENDIX B

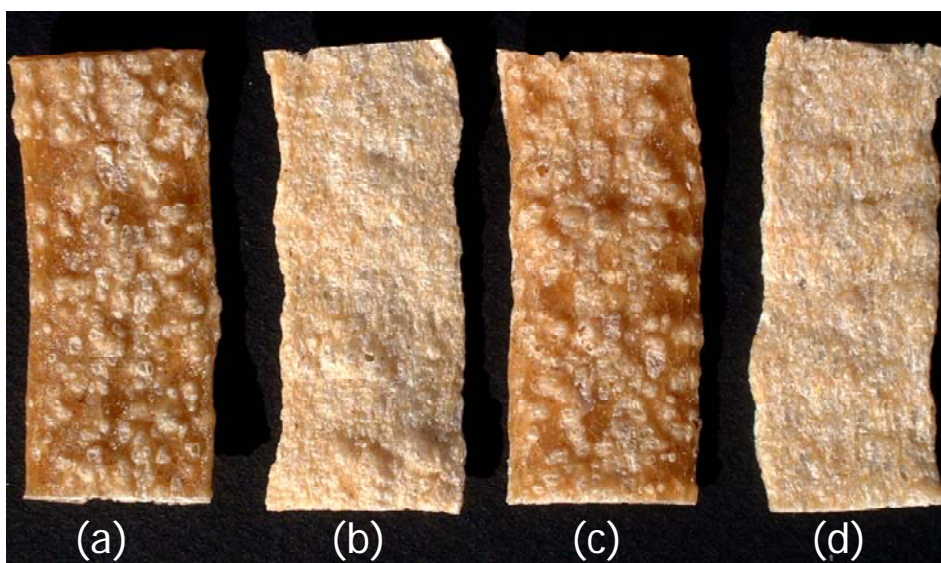


Figure B.1 Images of barley extrudates with following extrusion conditions: (a) 140°C, 150 rpm, (b) 160°C, 150 rpm, (c) 140°C, 200 rpm and (d) 160°C, 200 rpm

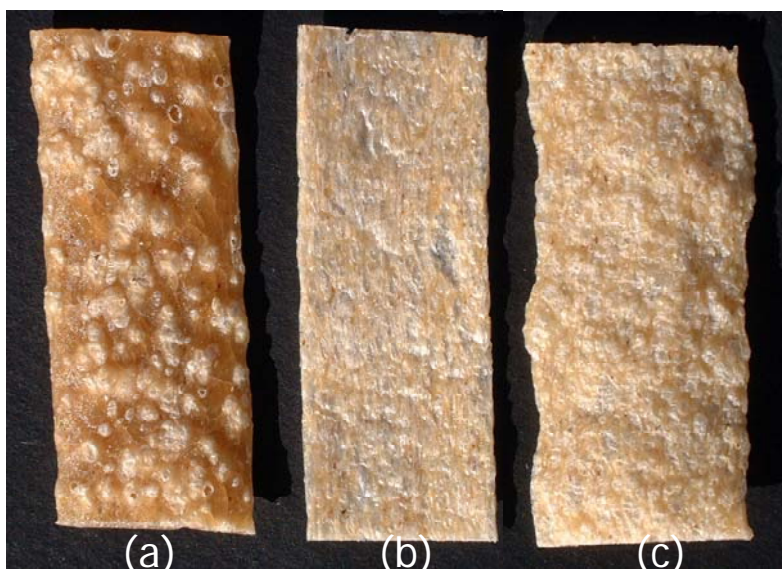


Figure B.2 Images of barley extrudates produced at 175 rpm screw speed and different temperatures: (a) 136°C, (b) 164°C and (c) 150°C

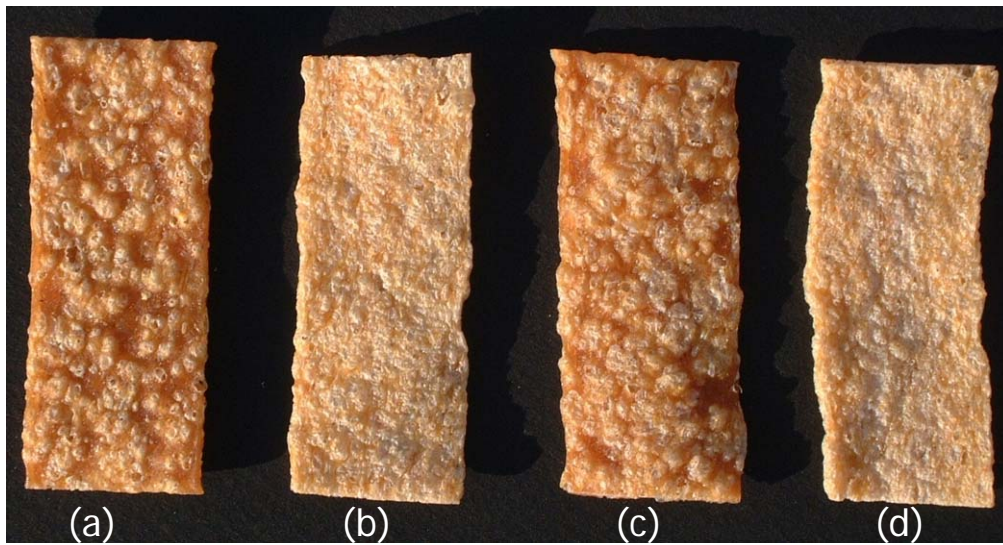


Figure B.3 Images of barley-tomato pomace extrudates produced at 2% pomace level with following extrusion conditions: (a) 140°C, 150 rpm, (b) 160°C, 150 rpm, (c) 140°C, 200 rpm and (d) 160°C, 200 rpm

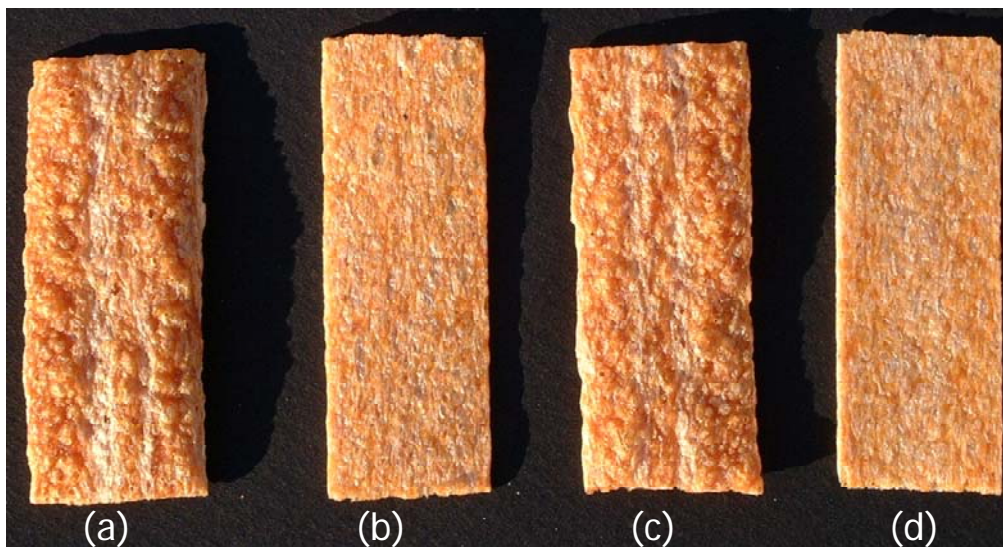


Figure B.4 Images of barley-tomato pomace extrudates produced at 10% pomace level with following extrusion conditions: (a) 140°C, 150 rpm, (b) 160°C, 150 rpm, (c) 140°C, 200 rpm and (d) 160°C, 200 rpm

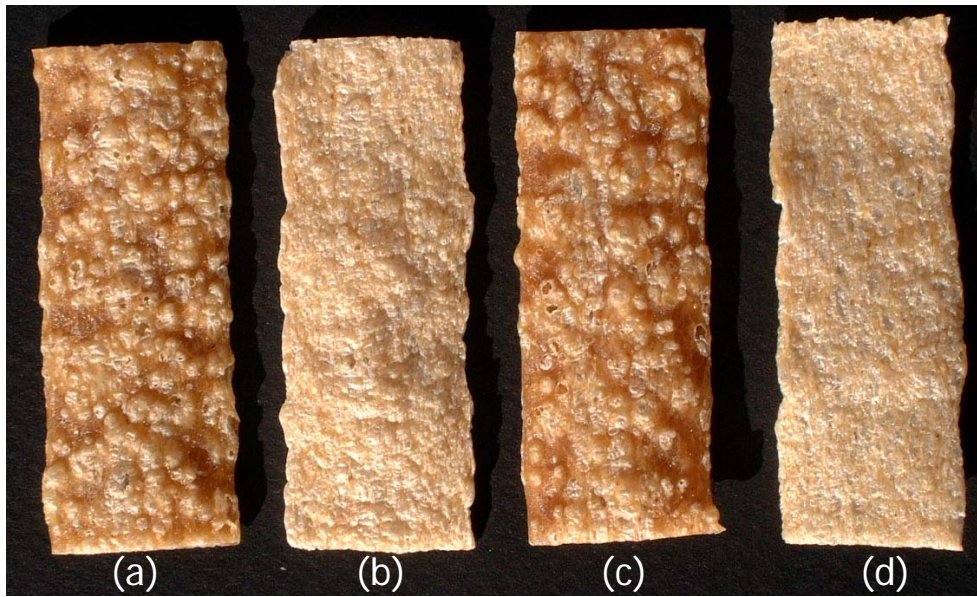


Figure B.5 Images of barley-grape pomace extrudates produced at 2% pomace level with following extrusion conditions: (a) 140°C, 150 rpm, (b) 160°C, 150 rpm, (c) 140°C, 200 rpm and (d) 160°C, 200 rpm

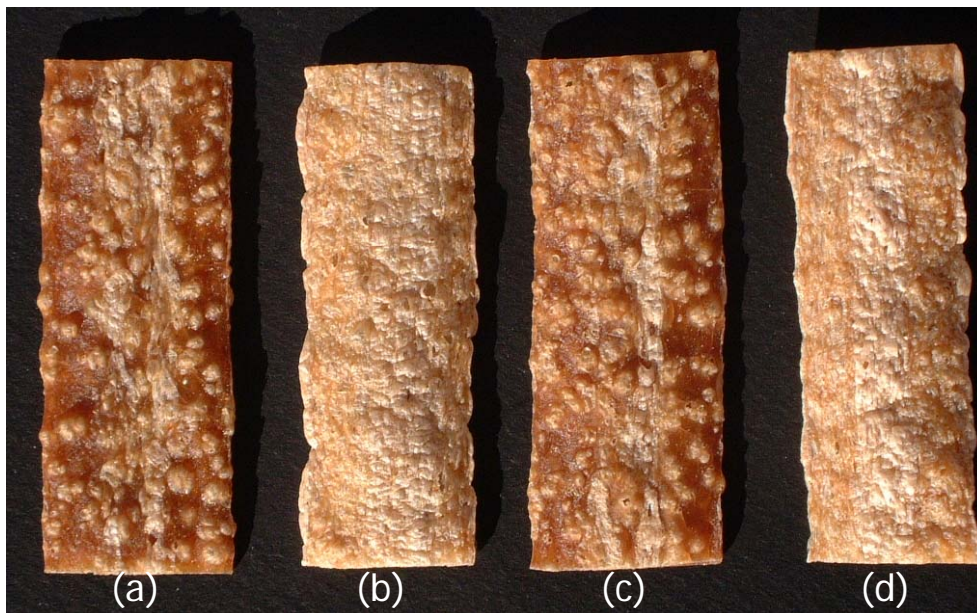


Figure B.6 Images of barley-grape pomace extrudates produced at 10% pomace level with following extrusion conditions: (a) 140°C, 150 rpm, (b) 160°C, 150 rpm, (c) 140°C, 200 rpm and (d) 160°C, 200 rpm

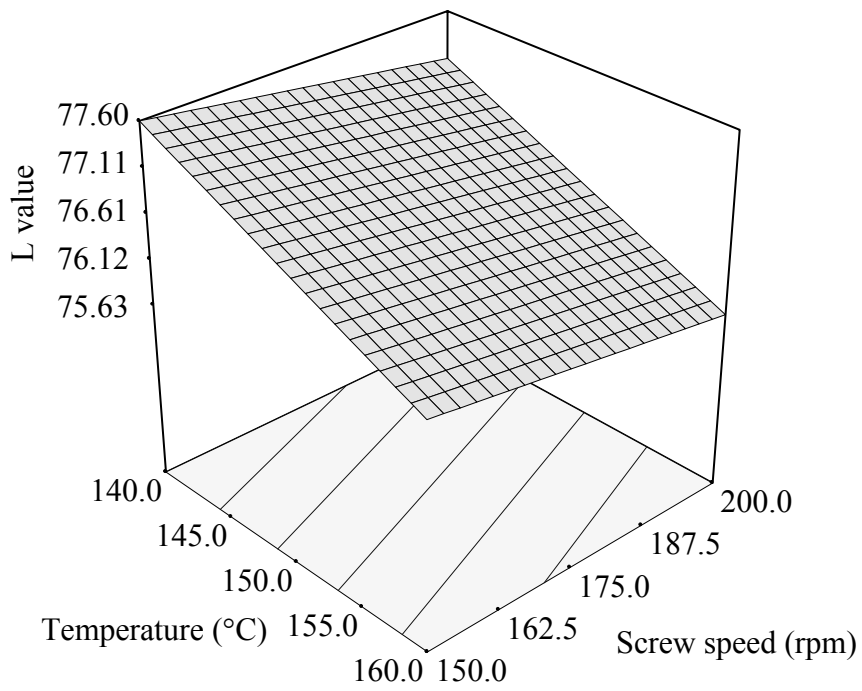


Figure B.7 Response surface plot for the effect of screw speed and temperature on *L* value of barley extrudates

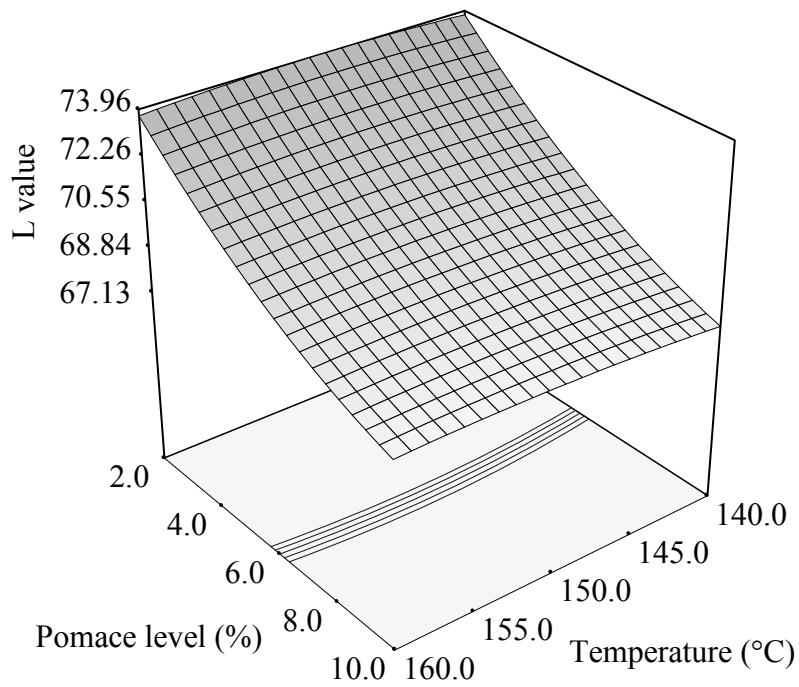


Figure B.8 Response surface plot for the effect of temperature and pomace level on *L* value of barley-tomato pomace extrudates at a screw speed of 175 rpm

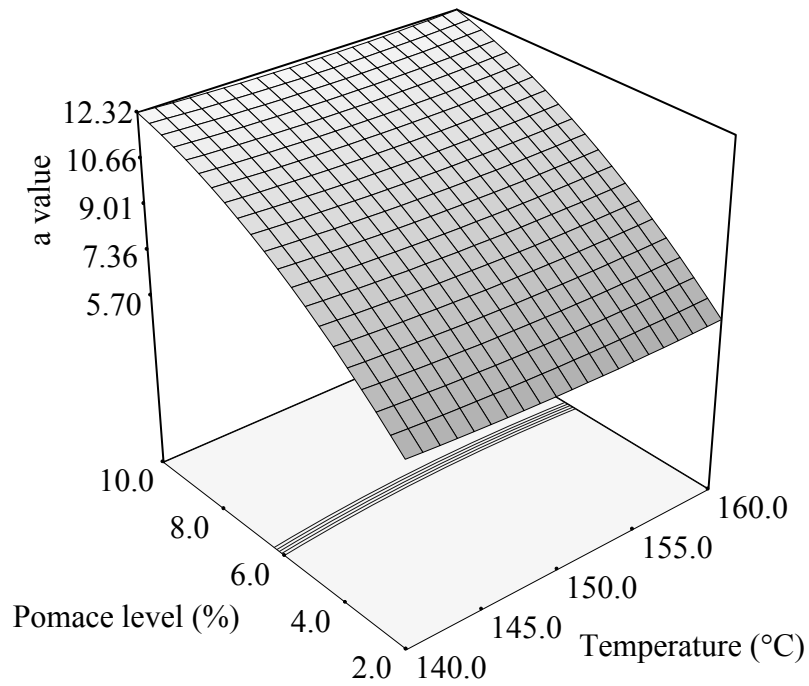


Figure B.9 Response surface plot for the effect of temperature and pomace level on *a* value of barley-tomato pomace extrudates at a screw speed of 175 rpm

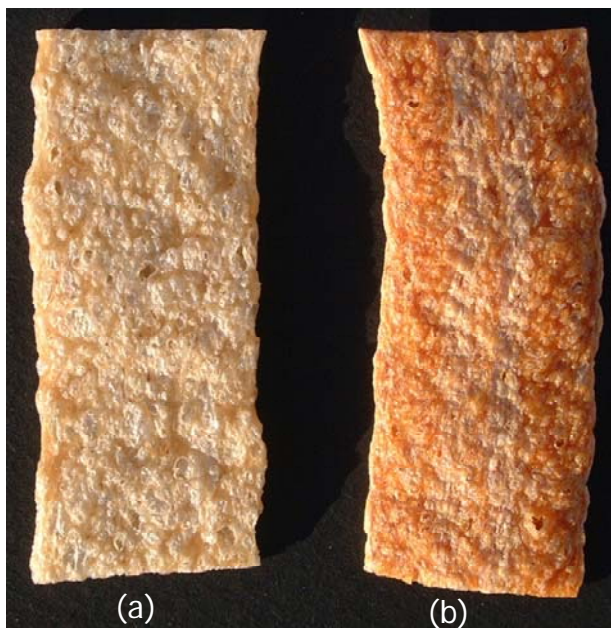


Figure B.10 Images of extrudates produced at 150°C and 175 rpm with different tomato pomace level: (a) 0% and (b) 12.7%

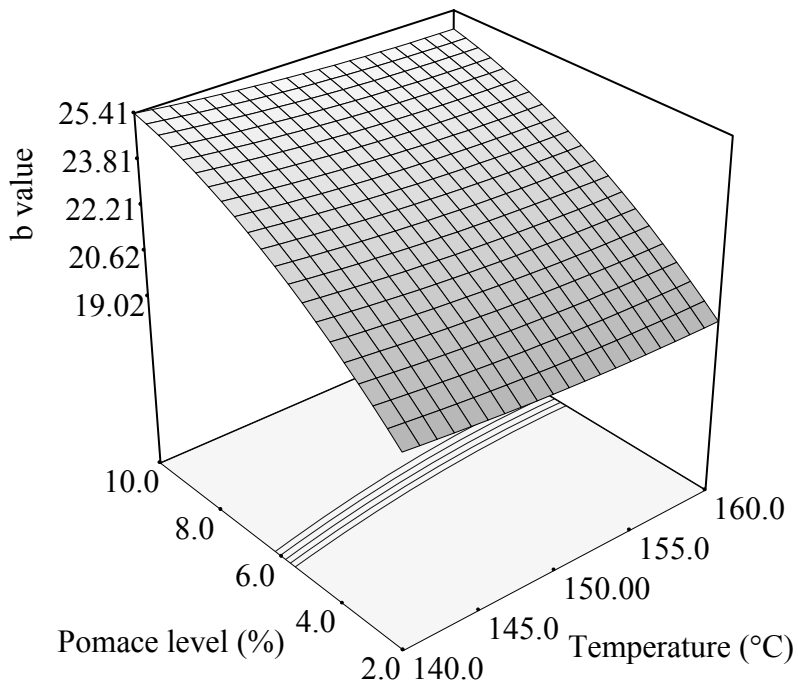


Figure B.11 Response surface plot for the effect of temperature and pomace level on *b* value of barley-tomato pomace extrudates at a screw speed of 175 rpm

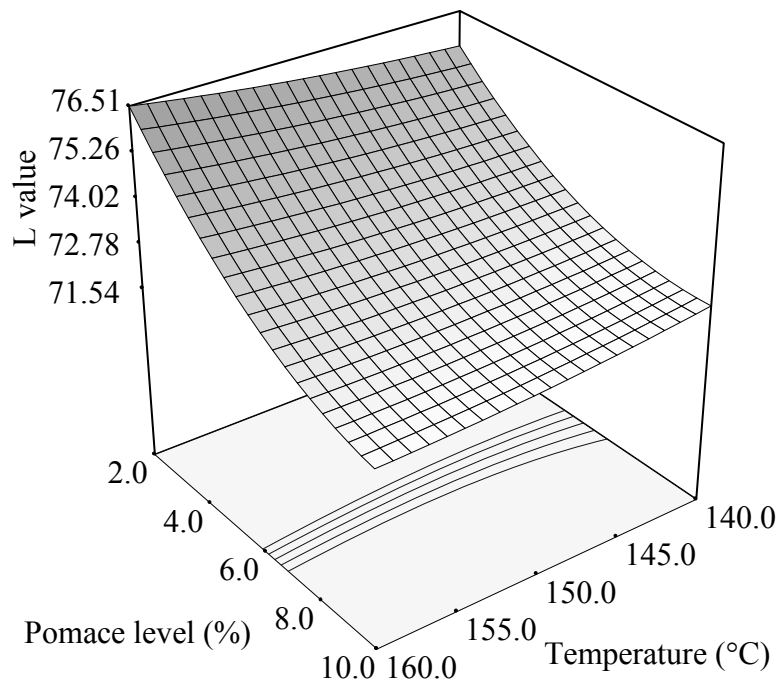


Figure B.12 Response surface plot for the effect of temperature and pomace level on *L* value of barley-grape pomace extrudates at a screw speed of 175 rpm

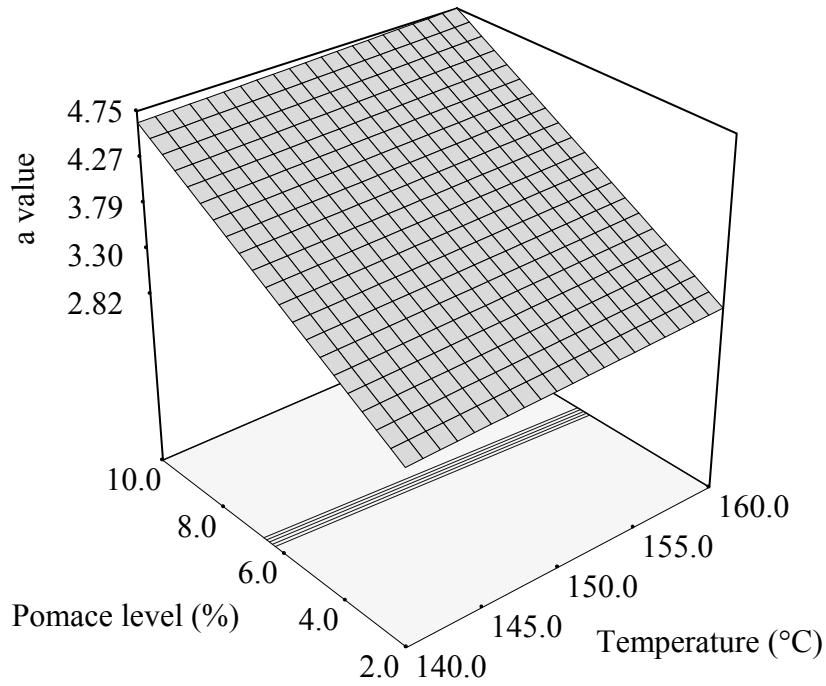


Figure B.13 Response surface plot for the effect of temperature and pomace level on *a* value of barley-grape pomace extrudates at a screw speed of 175 rpm

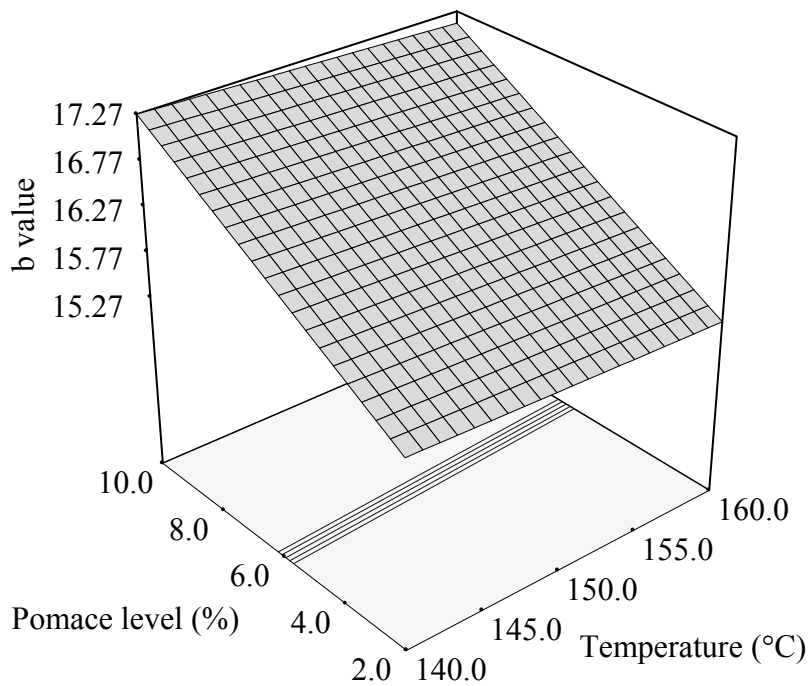


Figure B.14 Response surface plot for the effect of temperature and pomace level on *b* value of barley-grape pomace extrudates at a screw speed of 175 rpm



Figure B.15 Images of extrudates produced at 150°C and 175 rpm with different grape pomace level: (a) 0% and (b) 12.7%

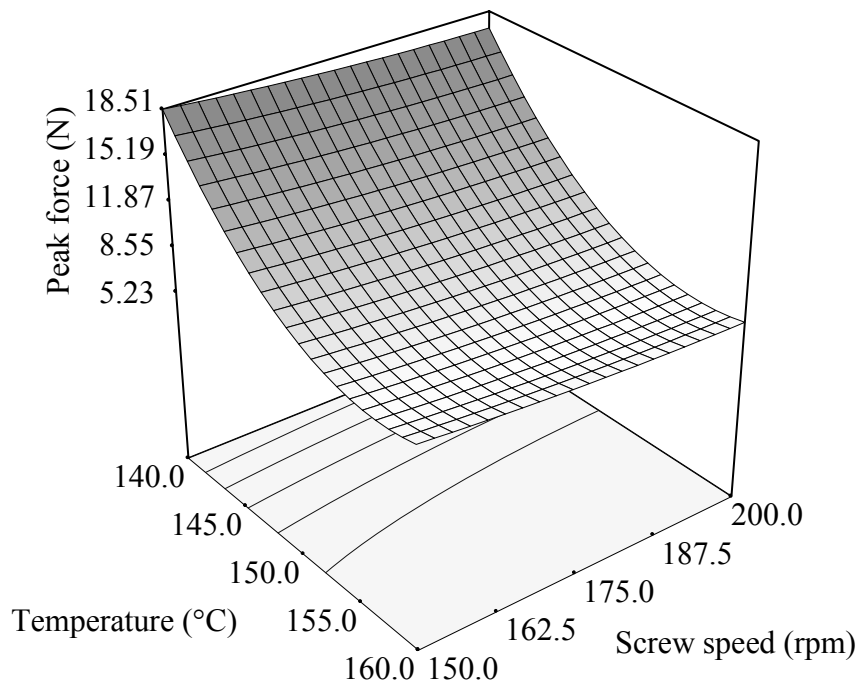


Figure B.16 Response surface plot for the effect of screw speed and temperature on peak force of barley-tomato pomace extrudates at a pomace level of 6%

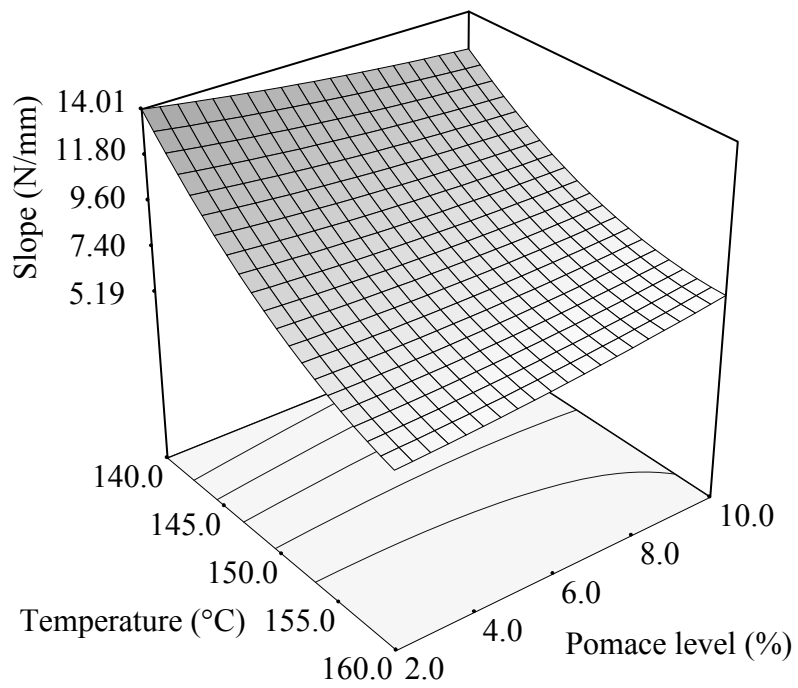


Figure B.17 Response surface plot for the effect of pomace level and temperature on slope of barley-tomato pomace extrudates at a screw speed of 175 rpm

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FOREIGN LANGUAGES

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PUBLICATIONS

Articles in Peer-Reviewed Journals

Altan, A. McCarthy, K.L. and Maskan, M. (2008). Extrusion Cooking of Barley Flour and Process Parameter Optimization by Using Response Surface Methodology. *Journal of the Science of Food and Agriculture*, 88, 1648-1659.

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Altan, A. McCarthy, K. L. and Maskan, M. Production of Healthy Extruded Snacks from Composite of Barley and Fruit By-products, ICC International Conference Bosphorus 2008, April 24-26, 2008, Istanbul, Turkey. (**Best Poster Award**)

Vandeven, J.C., **Altan, A.**, Maskan, M. and McCarthy, K.L. Value-Added Extruded Products: Barley and Fruit Pomace Blends, IFT Annual Meeting, Institute of Food Technologists, July 28-Aug. 1, 2007, Abstract No. 008-19, Chicago, IL.

Altan, A., McCarthy, K. L. and Maskan, M. Generation of New Snack Foods by Extrusion of Barley and Grape pomace, 2nd International Congress on Food and Nutrition, 24-26 October, 2007, Abstract No. P260, Istanbul, Turkey. **(3rd Place Poster Award)**

Altan, A., McCarthy K.L. and Maskan, M. The Effect of Screw Configuration on Extrudate Properties of Barley Products. Cereal Products Technology Congress and Exhibition, 7-8 September, 2006, pp.270-279, Gaziantep, Turkey.

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