# The Use of Durum Clear Flour in Combination with Hazelnut Cake and Different Pomaces in the Production of Extruded Food

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# ABSTRACT

## THE USE OF DURUM CLEAR FLOUR IN COMBINATION WITH HAZELNUT CAKE AND DIFFERENT POMACES IN THE PRODUCTION OF EXTRUDED FOOD

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The durum clear flour (8-20%), partially defatted hazelnut flour (PDHF) (5-15%), fruit waste blend (3-7%) and rice grit were blended in various ratios and then extruded using single screw extruder. Response surface methodology was used to simulate effects of process variables such as the feed moisture content (12-18%), barrel temperature (150-175$^{o}$C), and screw speed (200-280 rpm) and feed composition on physical and functional properties of the produced extrudates. The product properties of extrudates were mostly affected by changes in PDHF content. Increasing PDHF content caused increase in bulk density, water solubility index (WSI), *L* value, total phenolic content and antioxidant activity but decrease in radial expansion ratio, porosity, hardness, water absorption index (WAI), percent starch gelatinization and starch digestibility values of the extruded products. Changing fruit waste content generally affected the texture, color and functional properties of produced extrudates. Physical and functional properties of extruded products were influenced from extrusion process conditions. Increasing moisture content and decreasing temperature increased expansion ratio and porosity whereas it decreased bulk density for most product formulations. WSI decreased at high temperature and moisture content, whereas WAI increased. Screw speed influenced values of WSI, WAI and hardness. Moisture content and temperature influenced significantly the total phenolic content, antioxidant activity, percent starch gelatinization and starch digestibility values. In extruded samples, positive correlations were determined between antioxidant activity and total phenolic matter and percent starch gelatinization and the starch digestibility values, respectively. It was observed that well expanded extruded products with acceptable sensory attributes were obtained at low PDHF content at the end of sensory evaluation of extruded samples.

**Key words:** extrusion, response surface methodology, partially defatted hazelnut flour, fruit waste, bulk density, textural properties, antioxidant activity

# ÖZET

## İRMİK ALTI UNUNUN FINDIK KÜSPESİ VE FARKLI POSALAR İLE KOMBİNE EDİLEREK EKSTRÜDE GIDA ÜRETİMİNDE KULLANIMI

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İrmik altı unu (% 8-20), az yağlı fındık unu (AYFU) (% 5-15), meyve atığı karışımı (% 3-7) ve pirinç irmiği farklı oranlarda karıştırıldı ve sonra tek vidalı ekstrüder kullanılarak ekstrüde edildi. Besleme nem miktarı (% 12-18), gövde sıcaklığı (150-175$^{o}$C) ve vida hızı (200-280 rpm) gibi proses değişkenlerinin ve besleme kompozisyonunun üretilen ürünlerin fiziksel ve fonksiyonel özellikleri üzerine etkilerinin modellenmesinde yüzey tepki yöntemi kullanıldı. Ekstrüde ürünlerin özellikleri en çok AYFU içeriğindeki değişimden etkilendi. Artan AYFU içeriği ekstrüde ürünlerin bulk yoğunluk, suda çözünme indeksinde (SÇİ), *L* değeri, toplam fenolik madde içeriği ve antioksidan aktivitesinde artmaya sebep olurken, enine genleşme indeksi, gözeneklilik, sertlik, su absorblama indeksi (SAİ), yüzde nişasta jelatinizasyonu ve nişasta sindirilebilirliği değerlerinde azalmaya sebep oldu. Meyve atığı içeriğindeki değişim genellikle üretilen ürünlerin yapısal, renk ve fonksiyonel özelliklerini etkiledi. Ekstrüde ürünlerin fiziksel ve fonksiyonel özellikleri ekstrüzyon proses şartlarından etkilendi. Artan nem miktarı ve azalan sıcaklık bir çok ürün formülasyonu için enine genleşme indeksi ve gözenekliliği artırırken bulk yoğunluğu azalttı. SÇİ yüksek sıcaklık ve nem miktarında azalırken, SAİ arttı. Vida hızı SÇİ, SAİ ve sertlik değerlerini etkiledi. Nem miktarı ve sıcaklık toplam fenolik madde, antioksidan aktivite, yüzde nişasta jelatinizasyonu ve nişasta sindirilebilirliği değerlerini önemli derecede etkiledi. Ekstrüde örneklerde, sırasıyla antioksidan aktivite ile toplam fenolik madde ve yüzde nişasta jelatinizasyonu ile nişasta sindirilebilirliği değerleri arasında pozitif korelasyonlar olduğu tespit edildi. Ekstrüde örneklerin duyusal analizi sonucu, iyi genleşmiş ve kabul edilebilir duyusal özelliklerdeki ekstrüde ürünlerin düşük AYFU miktarlarında elde edildiği gözlendi.

**Anahtar kelimeler:** ekstrüzyon, yüzey tepki metodolojisi, az yağlı fındık unu, meyve atığı, bulk yoğunluk, yapısal özellikler, antioksidan aktivitesi

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## CONTENTS

## LIST OF FIGURES

## LIST OF TABLES

## NOMENCLATURE

| | |
|---|---|
| ANOVA | Analysis of variance |
| $\rho_b$ | Bulk volume |
| $\rho_s$ | Apparent Volume |
| DCF | Durum clear flour |
| FW | Fruit waste |
| $g$ | Acceleration of gravity |
| L-, a- and b- values | Color dimensions |
| $p$ value | probability |
| PDHF | Partially defatted hazelnut flour |
| PRESS | predicted error sum of squares |
| rpm | revolution per minute |
| $r^2$ | coefficient of determination |
| WAI | Water absorption index |
| WSI | Water solubility index |

# CHAPTER I
# INTRODUCTION

## 1.1 Extrusion

Extrusion can be defined as the operation of shaping a plastic or dough-like material by forcing it through high temperature and/or pressure region and then through a restricted opening or die to form the desired shape. It involves compressing a material to form a semisolid mass under a variety of controlled conditions and then forcing it to pass through a hole. The extrusion process was made continuous by substituting the piston in the cylinder of the original design with a helical screw. In this screw extrusion apparatus, material is continuously metered into an inlet hopper and then transported forward by the rotating screw. As the material approaches the die, there is usually increase in pressure and temperature which are sufficient to force extrudate through the die (Rokey, 2000). A food extruder is a device that expedites the shaping and restructuring process for food ingredients. The food extruder is viewed as a continues chemical reactor processing food mixes at high temperatures for relatively short residence times, at high pressures under shear forces, and in most cases, at relatively low water contents (Cheftel, 1986).

Extrusion cooking has become a well established industrial technology with a number of food applications. It has been used to produce wide variety of foods including snacks, ready to eat (RTE) cereals, confectioneries, texturized meat substitutes and extruded crisp breads (Suknark et al., 1997). The food extruder must complete a number of operations in a short time under controlled, continuous and steady-state conditions. The operations may include heating, conveying, mixing, cooking, melting, shearing, puffing, texturing, final shaping and drying in one energy efficient rapid continues process (Harper, 1989). The combination of operations is possible because of multitude of controllable variables such as feed rate, total moisture in barrel, screw speed, barrel temperature, screw profile, and die configuration (Thymi et al., 2005). Several basic components have specific functions common to all extrusion equipment (Huber, 2001).

- A feeding system, which usually consist of a live bottom-holding bin and a variable-speed metering device to feed raw dry-mixed ingredients uniformly and in uninterrupted manner at the desired flow rate.
- A pre-conditioner in which liquids, and/or steam or other vapors, may be uniformly combined with the pre-metered dry formula mix.
- An extruder assembly, whose barrel segments, screws and shearlocks configuration has been pre-selected to properly feed, knead and cook the dry or pre-moistened feedstock.
- A final die to restrict the extruder discharge and shape the product, and a device to cut the extruded profile to the desired length.

In the cooking process, there are generally two main energy inputs to the system. Firstly there is the energy transferred from the rotation of the screws and secondly the energy transferred from the heaters through the barrel walls. The thermal energy that is generated by viscous dissipation and/or transferred through the barrel wall results in an increase in the temperature of the material being extruded. As a result of this, there may be phase changes, such as melting of solid material, and/or the evaporation of moisture (Ainsworth and Ibanoglu, 2006).

During extrusion processing, food materials are generally subjected to a combination of high temperature, high pressure and high shear. The cooking temperature can be as high as 180-190$^{o}$C during extrusion, but residence time is usually only 20-40 seconds. For this reason, the extrusion cooking process can be called a high temperature short time (HTST) process (Riaz, 2001). This process of high temperature short time extrusion bring gelatinization of starch, denaturation of protein, modification of lipid and inactivation of enzymes, microbes and many antinutritional factors (Bahattacharya and Prakash, 1994; Ilo and Berghofer, 1999). According to the extrusion conditions used and products obtained Rokey (2000) classified extrusion processes into three categories:

- Low-shear stress (forming): used to increase density of material that is generally high in moisture (e.g. pasta) using low screw speeds.
- Medium-shear stress: used to process raw materials with lower moisture content with higher energy inputs (e.g. pet foods, aquatic feeds, texturized vegetable protein).
- High-shear stress: where extrusion speeds and energy inputs are high to process low-moisture raw materials in a short length-to-diameter ratio barrel. Highly expanded products are obtained (e.g. snacks, breakfast cereals).

Extrusion of raw food ingredients, containing mainly starches, proteins and lipids lead to many physical and chemical transformations in granules which in turn cause changes in properties of the extruded products (Zheng and Wang, 1994). In the high-shear extrusion process; moistened, starchy, and/or proteinaceous materials, are compressed, sheared and heated to form a melt that is forced through a die opening to the atmosphere. Because the exit temperature can reach as high as 300$^{o}$F (149$^{o}$C), some cooking (gelatinization) of the starches occurs during the extrusion process in addition to the shearing. By the time the material reaches the die opening, it has been heated and pressurized, resulting in a semi liquid mass of gelatinized starch containing superheated water. The pressure instantly drops when the product exits the die opening and the superheated water drop flashes to steam, causing the product to expand (puff) and cool. The faster and greater the pressure drop through the die, the greater the product expansion (Burtea, 2001).

Food extrusion as a process has been known since the late eighteen century. Single screw type extruders used for chopping or mincing soft food by forcing them thorough die plates have been the first screw extruders used in the food industry. In the 1930`s, a single screw extruder was introduced to the pasta industry, to both mix the ingredients (semolina and water) and to shape the resulting dough into macaroni in one continues operation. The principle of these extruders remains the same with recent developments focused on increased capacity and improved control. In the mid-1940s, single screw cooking extruders were firstly applied to make puffed snacks from cereal flours or grits especially cornmeal-based snack. Most likely, this was either an expanded yellow cornmeal or curl that was then coated with some type of seasoning such as cheese and salt. These machines are capable of processing dry granular cereal ingredients (up to 20% moisture content). Further refinement of the food extrusion process and extension of its applications occurred during the 1970s. Examples include use of extruder as a mixer and pasteurizer in the processing of soft-moist pet foods and co-extruded products containing more than one component. The use of twin-screw extruders for food processing started in the 1970s. These machines have improved conveying and mixing capabilities and interchangeable screw profiles. Great interest has been shown in twin screw extruders because of their expanded operational capabilities and extended range of applications (Harper, 1989; Rokey, 2000).

### 1.1.1 Advantages of Extrusion Cooking

Extrusion cooking technology offers several advantages over traditional methods of food processing, including the following (Riaz, 2000 and Guy, 2001).

*Versatility:* Wide range of food products, many of which can not be produced easily by any other process, can be produced by changing the operational settings of extruder and/or the ingredients. As a result, formulations can be altered to lower cost materials and final products are often improved.

*Productivity:* An extruder provides continuous high-throughput processing and can be fully automated. The ability to control quality is maximized because poor quality product is recognized immediately. Data obtained from the pilot plant can be used to scale up the extrusion system for production.

*Product Characteristics:* A variety of shapes, textures, colors and appearances can be produced, which is not easily feasible using the other production methods. They also provide improved product quality over other processes because cooking is done in a very short time and less destruction of heat sensitive in ingredients occurs. Extrusion also permits the inactivation of microbes and several anti-nutritional or toxic factors, of oxidative and other deterioration enzymes.

*Energy Efficiency and Low Cost Process:* The ability of extrusion systems to carry out a series of unit operations simultaneously and continuously gives rise to savings in labor cost, floor space cost and energy cost whilst increasing productivity.

*New Foods:* The advantages of extrusion are evident, especially in the simplification of processing techniques for the manufacture of existing products as well as in the development of novel types of food. Extrusion can modify animal and vegetable proteins, starches, and other food materials to produce wide variety of new and unique snack food products.

*Environmentally-Friendly:* This is very important advantage for the food industry, since new environmental regulations are stringent and costly. Extrusion produces little or no waste streams.

### 1.1.2 Types of Extruded Food Products

Diverse grains, formulated in countless ways and subjected to varying process conditions, can be used to make a wide-spectrum of extruded food products. The textured vegetable proteins, ready-to-eat breakfast cereals, direct expanded and third generation snacks and co-extruded products are the most commonly produced extruded food products.

*The texturized vegetable proteins*

Meat extenders represent the largest portion of textured vegetable food proteins. Cross-linking reactions between aligned protein molecules during extrusion process form a fibrous meat-like structure that can be hydrated and used as a meat substitute in a variety of foods (Harper, 1989). Simulated meat analogs, which are also referred to as just meat analogs, use extrusion cookers to transform vegetable protein sources directly into varieties of simulated meat analogs that are consumed as is and have the appearance, texture, and mouthfeel of meats. Defatted soy flour, flakes, and grits, soy concentrates and isolates, mechanically extracted soy meal, wheat gluten, and other legume/grain sources are raw materials that are used in producing texturized vegetable proteins. As mechanical and thermal energy are applied during extrusion, the macromolecules in the protein portion of the raw material lose their native, organized structure and form a continuous plastic-like mass (Sevatson and Huber, 2000).

*Ready-to-eat breakfast cereals*

Extrusion is one of many techniques used to manufacture ready-to-eat (RTE) cereal. Extrusion has become the preferred processing technology because it accepts both cereal and starch ingredients, creates highly expanded products, produces a wide variety of shapes and textures, cooks and forms in a single processing step, and processes at relatively low moistures (Harper, 1989). The popularity of RTE breakfast cereals really stems from their nutritional content. Such products can simultaneously provide energy (350-400 kcal/100g), nutrients (vitamins and minerals) and health-oriented components (dietary fiber, for instance). These products can also be blended with additives such as dried fruits, nuts, marshmallow pieces, etc. Various forms of RTE breakfast cereals are seen in Figure 1.1. There are two types extrusion cooked breakfast cereals found on the market (Bouvier, 2001).

- Directly expanded extrusion cooked breakfast cereals. Expanded RTE cereals manufactured from mixtures of cereal flour and ingredients at low moisture content (usually below 20%) using combination of preconditioning and extrusion cooking.
- Pellet-to-flakes extrusion cooked breakfast cereals. Cereal flours and ingredients are cooked at a moisture level in the range of 22-26% to form pellets, which are then flaked or sheeted and cut to create the finished product. In such applications, the cooked dough must be cooled before pellet forming to prevent expansion (Harper, 1989).

Figure 1.1 Examples of various ready-to-eat breakfast cereal products (American Extrusion International, South Beloit, USA, 2008)

### *Direct expanded and third generation snacks*

The name snack foods cover a wide range of food products. They are consumed as light meals or a partial replacement for a regular meal. Extrusion has provided a means of manufacturing new and novel products and has revolutionized many conventional snack manufacturing processes. The processing parallels RTE cereal process extensively but is normally performed at lower moistures, so greater fraction of the energy input comes from mechanical sources. To achieve a highly expanded, melt-in-the-mouth texture, production of puffed snacks is done with low moisture (<15%), high shear, and high-temperature extrusion conditions in which significant starch damage occurs (Harper, 1989).

Direct expanded, or second-generation, snacks make up the majority of extruded snacks on the market (Figure 1.2). Expanded snacks, with a variety of attributes, including high fiber, low calorie, and high protein contents, can be produced. They are usually `light`, meaning they have low bulk density, and are seasoned with an array of flavors, oils and salt. These snacks can be finished by frying or baked (Burtea, 2001). The most popular one of these is cornmeal based snacks. The corn meal is exposed to moisture, heat and pressure as it is transported through the extruder into the extruder die. As the material exit the extruder die, it expands due to pressure release and is cut to the proper length with a rotating knife. Extruders for direct expanded snack products are normally short in length (less than 10:1 L/D). Moisture is normally between 8-10% on a wet basis and requires additional drying to produce the desired product crispness. With drying, this moisture is brought down to 1 or 2% (Sevatson and Huber, 2000).

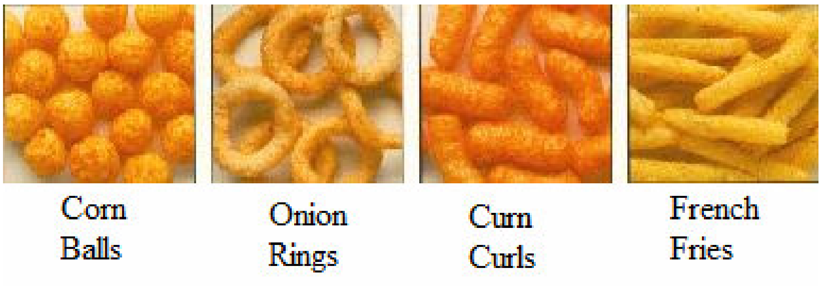


Figure 1.2 Second-generation direct expanded snacks (Wenger manufacturing, Inc., Sabetha, Kansas, 2008)

Third generation snack products are sometimes referred to as `semi-` or `half products`. Following extrusion cooking and forming into dense pellets, the pellets are dried to a stable moisture content to assure stability during storage. Then, they are distributed to processors, where they are puffed or expanded by immersion in hot oil or hot air puffing. They are also sold for frying or puffing by the consumer at home, or in restaurants for immediate consumption (Burtea, 2001). A wide range of raw materials can be selected and blended to produce excellent recipes for many third generation snacks. Generally, the combination of ingredients contains relatively high levels of starch to maximize the expansion of the final product during exposure to hot oil, air, or microwave. Levels of less than 60% total starch in a recipe result in reduced final product expansion, yielding a final product with increased crunchiness and a firmer texture. Shortenings, vegetable oils, salts, and occasionally emulsifiers are included in recipes as processing aids, to reduce stickiness, to control expansion, and to impart a more uniform cellular structure in the final product (Sevatson and Huber, 2000).

### *Co-extruded products*

Co-extruded snacks typically have an extrusion-cooked outer shell with a pumpable, but not free flowing filling. Many variations of this concept may be made with dies and post-extrusion equipment such as rollers, stampers and belt-type cutters (Figure 1.3). In many cases, the shelf lives of the products have been short due to migration of moisture and/or lipids from the filling to the outer shell. However, technological advances in this area are providing possibilities to increase the compatibility of fillings, with a wide range of different moisture and lipid contents (Huber, 2001).

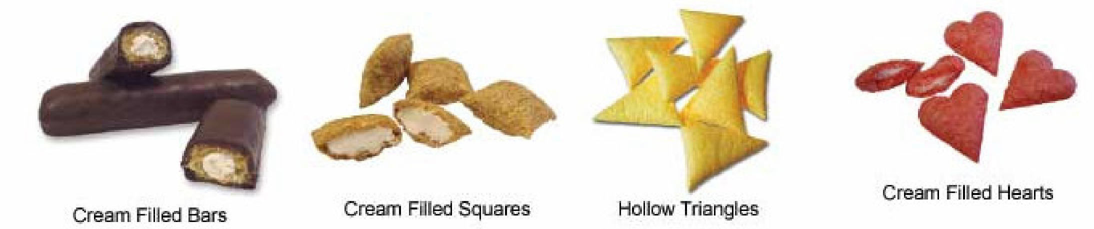


Figure 1.3 Examples of co-extruded products (American Extrusion International, South Beloit, USA, 2008)

### 1.1.3 Types of Extruders

Several designs of extruders are competing in the market place; single-screw and twin-screw extruders are the main types and widely employed in commercial food production.

*Single screw extruders*

Single screw extruders were the first types to be used in the food industry and continue to have a wide use in products like pasta and other cereal products. Their operation is fairly simple. The raw materials are fed into a bin, which has to be adequate size to support extruder operation. The raw materials are fed into the barrel or pre-conditioner of the extruder at a uniform rate controlled by a metering device. Because single screw extruders have relatively poor mixing ability, they are often used with materials that have been either premixed or preconditioned. The pre-conditioner is either an atmospheric or a pressurized chamber in which raw granular food ingredients are uniformly moistened and/or heated by contact with water or live steam. An essential feature of the pre-conditioner is its delivery of uniformly pretreated ingredients to the feed section of the extrusion screw (Harper, 1986).

Single screw extrusion consists of three sections: feed, transition, and metering (Figure 1.4). The extrusion screw sequentially conveys and heats food ingredients and works them into a continuous plasticized mass while rotating in a tightly fitting barrel. The ratio of flow area in the feed section to that in metering section, defined as the compression ratio, ranges from 1:1 to 5:1 in different machines (Harper, 1989). During transport through these extruders, the mechanical energy required to turn the screw is converted to heat, raising the temperature of the mixture rapidly and transforming the mass from granular state into a continuous plasticized mass. In this process, the shear is very high and can cause mechanical damage to the large food molecules (Harper, 1986). The resulting plasticized feed ingredients are then forced through die in which the pressure drop causes puffing to occur (Arhaliass et al., 2003).

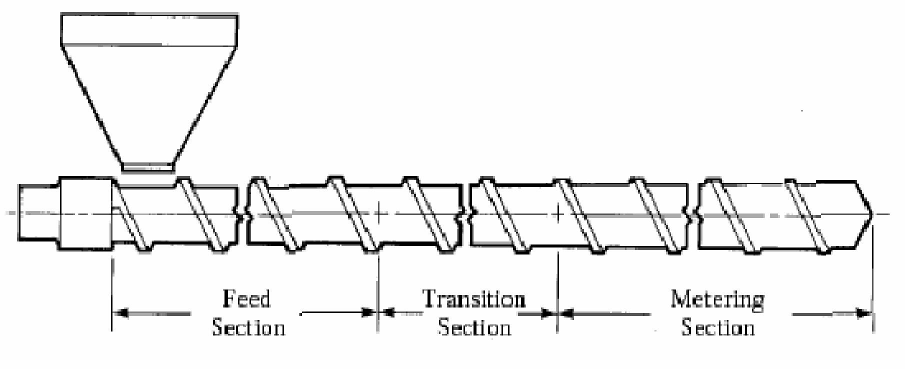


Figure 1.4 Single screw extruder

The single screw extruder relies upon drag flow to convey the feed material through the barrel of the extruder and to develop pressure at the die. The frictional force between the material and the barrel wall is the only force that can keep the material from turning with the screw and hence many single screw machines have grooves cut in the barrel to promote adhesion to the barrel wall (Ainsworth and Ibanoglu, 2006). Since the screw channel is continuous, it also serves as a path for pressure induced flow. This pressure flow is usually opposite in direction to the drag flow because of the comparatively higher pressure behind the die than at the feed throat. The net flow patterns in the extrusion screw are quite complicated and are affected by the magnitude of the pressure flow (Harper, 1986; Harper 1989). Single screw operation depends upon the pressure requirements of the die, the slip at the barrel wall and the degree to which the screw is filled. Screw fill is dictated by feed rate, screw speed, melt characteristics and viscosity. The coupling of these variables limits the operating range and flexibility of single screw extruders (Harper, 1989).

*Twin screw extruders*

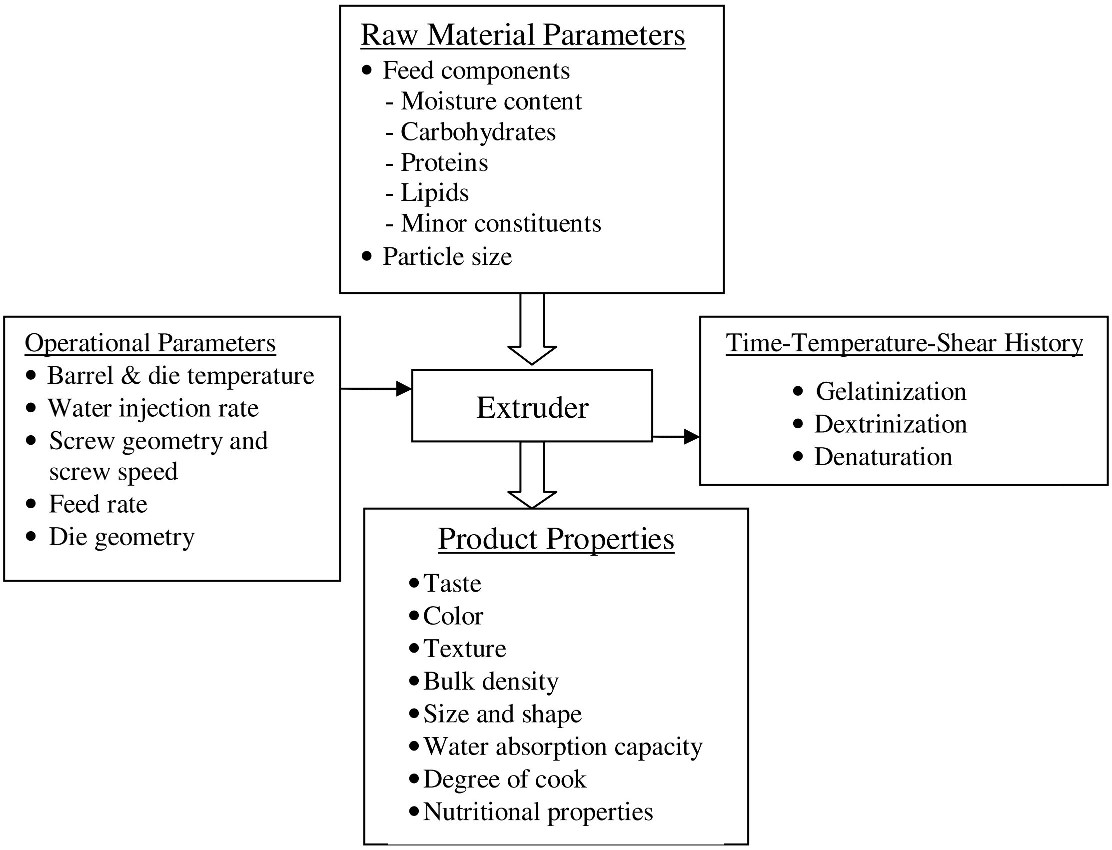
Twin screw extruders consist of two screws of equal length placed inside the same barrel. In addition to manufacturing foods similar to those produced by single screw extruders, twin screw extruders have found a wide application in the food industry due to their better process control and versatility, their flexible design permitting, easy cleaning and rapid product change over and their ability to handle wide variety of formulations (Ainsworth and Ibanoglu, 2006). Twin screws extruders are generally categorized according to the direction of screw rotation (co-rotating and counter-rotating) and to the degree to which the screws intermesh. 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. They are good in processing relatively nonviscous materials requiring low speeds and long residence times such as gum and jelly (Riaz, 2000). In the co-rotating extruders, screws rotate in the same direction. These types of extruders are most commonly used in the food industry especially in the snack food production because of their high capacity and enhanced mixing capability, good control over residence time distribution, self-cleaning mechanism and uniformity of processing (Harper, 1989; Riaz, 2000). Twin screw extruders can also have intermeshing screws in which the flights of one screw engage the other or they can non-intermeshing screws in which the threads of the screws do not engage one another, allowing one screw to turn without interfering with the other. Intermeshing co-rotating extruders are particularly suited to applications where high degree of heat transfer is required but not forced conveyance and thus widely used for the production of expanded products (Ainsworth and Ibanoglu, 2006).

### 1.1.4 Effect of Extrusion on Product Properties

Control of extrusion processes is difficult due to strong interactions between mass, energy and momentum transfer, coupled with complex physicochemical transformations, which govern final product properties. The quality of extruded products is controlled by the formulation, pre-extruder operation (blending and preconditioning), extruder screw configuration, die design, extruder operating conditions (feed rate, screw speed and water injection rate into the barrel) and post extruder operations (drying, toasting and flavor addition) (Chessari and Sellahewa, 2001) . Interactions of raw material parameters, operational parameters and product properties during extrusion cooking are shown in Figure 1.5.

The conditions, under which extrusion systems process a formula, include both independent and dependent variables. Independent operating variables include raw material formulation, feed rate, barrel temperature profile and screw/die configuration which the extruder operator can directly control. Changes in the operating parameters will cause changes in dependent process variables such as die product temperature, die pressure, residence time and viscosity, as well as product quality attributes through change in specific mechanical energy during the products residence in the extruder.

Figure 1.5 Interactions of raw material parameters, operational parameters and product properties during extrusion cooking



### 1.1.4.1 Raw material characteristics

Extruded foods are made from a wide and diverse range of raw materials. 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. Extruded products are formed from the natural biopolymers of raw materials such as cereal or tuber flours that are rich in starch, or oilseed legumes and other protein rich sources. The most commonly used materials are wheat and maize flours, but many other materials are also used such as rice flour, potato, rye, barley, oats, sorghum, cassava, tapioca, buckwheat, pea flour and other related materials (Guy, 2001).

*Particle size*

Particle size greatly affects both processing and extrudate characteristics. It basically alters the extent at which starch is modified inside the extruder. Finer raw materials tend to give products with softer textures and smaller cell structures, while coarse particle size materials produce extrudates with crunchier texture and larger cell structure (Harper, 1989). However, small particles cause problems during extrusion. Small particles tend to segregate in the feeding system and in the inlet portion of the extruder barrel. Small particles absorb water much faster than coarse particles, causing a non-homogenous moisture distribution. This alters product flow and cooking uniformity inside the extruder, producing surging and fluctuation in product quality. This surging can cause the extruder to plug, stopping the production. However, this problem can be diminished with tempering, equilibration and proper granulation of the raw materials.

*Starch*

Starch is the predominant ingredient in extruded snack and ready-to-eat cereals. It provides most of the texture and structure of expanded products made from cereals (Bahattacharya and Hanna, 1987). During the extrusion process, the starch is partially hydrated and subjected to increasing shear while it is mechanically conveyed and heated. Native starch undergoes substantial changes leading to greater molecular disorganization during extrusion. Most importantly from the perspective of finished product texture, the starch losses its native crystallinity, undergoes molecular degradation, and complexes with lipids in the feed mixture (Harper, 1986).

A major difference between extrusion and other forms of food processing is that gelatinization occurs at much lower moisture levels (12-22%). 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., 1992; Onwulata et al., 1998). The loss of crystallinity during extrusion is no longer caused by penetration of water but by mechanical disruption of the molecular bonds by the intense shear fields within extruder (Wen et al., 1990). Therefore, under extrusion at low moisture content, a mixture of small amounts of gelatinized and melted states of starch as well as fragmentation exist simultaneously (Lai and Kokini, 1991).

Starch is made of linear amylose and branched amylopectin. Amylose is less susceptible than amylopectin to mechanical damage in the flow environment within the extruder. Also, higher temperatures are required to increase its solubility (Harper, 1986). It was reported that a high amylopectin content leads to light, elastic, and homogenous expanded textures, while a high amylose content leads to hard, less expanded extrudates (Mercier and Feillet, 1975). Amylopectin-rich starches expand more than amylose-based starches because the linear amylose chains align themselves in the shear field and thus are difficult to pull apart during extrusion (Moraru and Kokini, 2003). During extrusion, the formation of amylose-lipid complex is evident. The extent of amylose-lipid complex formation is dependent upon both starch and lipid type present in a food (Singh et al., 2007a).

*Protein*

The proteins may be present in several forms depending on the ingredient used, as they may be derived from cereals, legumes or animal proteins. These polymers will form separate phases within the continuous starch phase. Typically protein act as a \`filler\` in cereal extrudates and is dispersed in the continuous phase of the extrusion melt, modifying the flow behavior and characteristics of the cooled extrudates (Ainsworth and Ibanoglu, 2006). The addition of proteins to starches increases sites for cross-linking and affects textural quality. Proteins can crosslink in extruder through stronger covalent and ionic bonds, rendering it relatively insoluble and resistant to loss of texture with further processing such as retorting (Harper, 1986).

Proteins have an effect on expansion through their ability to affect water distribution in the matrix and through their macromolecular structure and conformation, which affects the extensional properties of the extruded melts (Madeka and Kokini, 1992). Type and concentration of proteins affected the expansion of extrudates (Faubion and Hoseney, 1982). When soy protein isolate incorporated to wheat starch at levels of 1 to 8%, expansion ratio increased, while wheat gluten addition reduced expansion ratio when used in concentrations of up to 11%. Prinyawiwatkul et al. (1995)

presented that addition of protein to a starchy extrusion system may retard expansion by increased firmness of plasticized extrudates. According to Onwulata et al. (2001) expanded extrudates could be produced with up to 25% whey protein substitution for corn meal, potato and rice flour, while concentrations beyond 25% reduced expansion. Recent research on effect of protein concentration on physical and chemical properties of expanded whey product (Allen et al., 2007) showed that increasing whey protein concentrate level from 16 to 40% reduced extrudate expansion, air cell size, water solubility index and increased extrudate density, and breaking force. In some studies, protein rich raw materials were also added to starchy cereals. Bahattacharya (1997) reported that extrudates produced from rice-green gram blends have lower expansion ratios than the rice alone. Similar finding was observed for the rice-cowpea-groundnut blend, cowpea and groundnut additions to the rice resulted in decrease the expansion ratio (Asare et al., 2004).

### *Lipid*

Lipids are generally used in extrusion process to improve eating quality of extruded products. Lipids have a powerful influence on extrusion cooking processes by acting as lubricants between the particulate matter and the screws of the extruder. They become mixed with the other materials and are rapidly dispersed into small droplets and are trapped in the continuous phase (Guy, 2001). High-fat materials are generally not extruded. Lipids over 5-6% impair extruder performance. Torque is decreased because the lipid reduces slip within the barrel, and often product expansion poor because insufficient pressure is developed during extrusion (Strahm, 2000). The addition of lipid is also found to decrease the degree of starch gelatinization, due to a decrease of the barrel temperature caused by lubricating effect (Lin et al., 1997). Lipid decreases starch conversion during extrusion cooking by preventing severe mechanical breakdown of the starch granules by shear stress and preventing water from being absorbed by starch (Moraru and Kokini, 2003). Lipid reduces the friction between particles in the mix and between the screw surfaces and the fluid so that the forces acting on the starch granules are reduced and they may take longer to disperse in the shear zone (Guy, 2001).

### *Fiber*

Fiber is used as bulking agent, to provide nutritional attributes and to modify the texture of many extruded products. Fiber usage is often limited by its effect on product expansion. Beet pulp, fruit, pea and soy fibers are considered to reduce expansion the least and may be added to starch-based formulas at levels of from 5 to 10% (Huber, 2001). The non-starch polysaccharides in fiber may bind water more tightly during extrusion than do protein and starch. This binding may inhibit water loss at the die and thus reduce expansion (Camire and King, 1991). Hsieh et al. (1989) observed that increasing fiber content in corn meals had positive effect for the axial expansion and the negative effect for radial expansion, and so the net result was increase in the bulk density of both wheat fiber and oat fiber- containing corn meal extrudates. In another study, fiber added to corn extrudates increased expansion and breaking strength and improved physical characteristics of extrudates (Onwulata et al., 2001). Above critical concentration, the fiber molecules disrupt the continuous structure of the melt, impeding its elastic deformation during expansion (Moraru and Kokini, 2003).

### *Other ingredients*

Low molecular weight materials, such as sugars or salts, are commonly added to extruded products for flavoring properties. The materials that are soluble is dissolved in free dough water during the initial mixing stage of processing. Their effects on the extrusion process depend on their concentration and their chemical interaction with starch and protein polymers (Guy, 2001).

Salt is hygroscopic and limits the availability of water for gelatinization of starch if used at high levels (Huber, 2001). It has previously been shown that the addition of sugars has significant effect on expansion of maize extrudates (Fan et al., 1996; Carvalho and Mitchell, 2000). Fan et al. (1996) reported that the inclusion of sucrose and fructose at levels of up to 20% of the dry maize resulted in a substantial decrease in the sectional expansion. The reduced expansion was interpreted as a combined effect of reduction in bubble growth and shrinkage. The addition of sugar has increased the volume and diluted the starch so, the specific mechanical energy input and temperature fall. This cause reduced driving force for bubble growth and reduced bubble wall extension before rupture, caused by less starch conversion at high sugar content. The effect of sucrose on both maize and wheat flour extrudates showed that, in contrast to maize extrudates, sucrose addition had little effect on the degree of starch conversion and sectional expansion of wheat flour extrudates (Carvalho and Mitchell, 2000).

Baking soda (sodium bicarbonate) may be used for creating nucleating sites for steam in both direct expanded products and in snack pellets. In product, numbers of smaller cell are increased and this is generally more desirable to the consumer (Huber, 2001). Berrios et al. (2004) reported two fold increases in expansion ratio of extruded black beans with the addition of 0.5% sodium bicarbonate.

#### 1.1.4.2 Process variables

*Moisture content*

Extrusion cooking 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 basis. At levels of >10% there is sufficient water for the polymers to begin to move and slide across each other and the physical nature of the extrudates changes from a glassy state to a viscous elastic fluid (Guy, 2001). Water content has been found to affect the cellular structure and mechanical properties of extruded products in addition to their overall densities (Harper, 1989). This is particular interest for the many roles water plays: as a diluent or lubricant reducing viscosity; as a heat sink restricting temperature variations; as a reactant, particularly in gelatinization; and as a direct influence on puffing, product moisture, and structure through its effects on elasticity and plasticity (Miller, 1990).

Extrudate expansion and texture depend on the interaction of shear, heat and moisture in the extruder. Moisture during extrusion provides the driving force for expansion and also contributes to the rheological properties of the melt, which in turn affect expansion. It is the main plasticizer of the cereal flours, which enables them to undergo a glass transition during extrusion process and thus facilitates the deformation of the matrix and its expansion (Moraru and Kokini, 2003). Moisture allows the extrudates to expand. The temperature is well above the boiling temperature of water but under pressure. When the product exits through the die the pressure decreases suddenly and the superheated water evaporates, expanding the product. Lower moisture content causes increased viscosity and more mechanical damage. Expansion generally decreases rapidly when moisture content increases. This is due to reduction in viscosity of melt, die pressure and temperature which may affect the extent of product deformation during extrusion process (Miller, 1990). When the extrudate is very moist, it may expand significantly right after the die but collapse before it can cool, solidifying to create a very hard and undesirable texture (Harper, 1986). Many researchers (Ilo et al., 1996; Parsons et al., 1996; Liu et al., 2000; Onwulata et al., 2001) reported that increasing moisture content during extrusion decreases the specific mechanical energy, apparent viscosity, and radial expansion.

*Temperature*

Extrusion temperature plays an important role in changing the rheological properties of the extruded melts (Moraru and Kokini, 2003). The control of temperature has profound effect on the condition of the dough just behind the die and on the final expansion of the product. Temperature determines the vapor pressure of the moisture and thus degree of puffing. Higher temperature lowers the viscosity of the dough mass in the extruder and hence results in higher linear velocity at the die (Hsieh et al, 1989). The vapor pressure of the water in the dough, which is related to the temperature, provides most force which causes expansion on the product is released to ambient pressure (Harper, 1986).

Samples do not start to expand until a temperature of about 100°C has been reached. The expansion ratio increases with increasing temperature when water content is lower than about 19.5% (Harper, 1989). This could result either from a viscosity decrease, allowing the melt to expand more readily, or from an increase in vapor pressure. At low extrusion temperatures, radial and longitudinal expansions are reduced because starch is not fully melted. As temperature increases, the degree of expansion of cereal grits reaches a maximum at about 170°C, regardless of the equipment used. The decrease at higher temperatures has been attributed to increased dextrinization and weakening of structure (Harper, 1989). Kokini et al. (1992) observed that there is a temperature range where diametral expansion of starch reaches a maximum. Past a critical temperature, expansion decreases with temperature. They suggested that this was probably due to excessive softening and structural degradation of the starch melt, which becomes unable to withstand the high vapor pressure and therefore collapses.

*Screw speed and Feed rate*

The screw conveys the materials inside the extruder. Screw speed affects the degree of fill within the screw, residence time distribution of product flowing through the extruder, heat transfer rates and mechanical energy input in the extruder, and the shear forces exerted on the materials (Harper, 1986). The higher the specific mechanical energy applied to the product in the form of friction, it is expected to have a higher expansion ratio with increasing screw speed. Screw speed has generally positive effect on extrudate expansion due to increase in shear, and thus decrease in melt viscosity induced by high screw speeds (Kokini et al., 1992).

The effect of screw speed on extrusion and product characteristics is usually complex and is temperature dependent. Increasing screw speed decreased the radial expansion of rice-green gram extrudates at low temperature, such as 100$^{o}$C, and increased the radial expansion at high temperature, such as 175$^{o}$C (Bahattacharya, 1997). It was reported that at high temperature with high screw speed, the product expanded more due to release of superheated steam and shear-induced disruption. On the other hand, if the barrel temperature is low, to achieve a high extent of depolymerization, the screw speed should be low. A low screw speed is associated with high residence time; consequently the food material inside the extruder receives more input of thermal energy in a low shear environment. High input of thermal energy due to high residence time (at low screw speeds) leads to the creation of enhanced level of superheated steam; hence the product will have good expansion which creates flaky and porous structures due to formation of air cells (Bahattacharya, 1997).

Ilo et. al. (1999) found that screw speed influenced the expansion negatively in the radial direction and positively in the longitudinal direction of the rice-amaranth extrudates. They concluded that increased shear stress with increasing screw speed induces the breakdown of the starch molecules that decrease the elasticity of the melted material. This decreased the extrudate expansion in the radial direction, and increases it in the longitudinal direction. On the other hand, some authors have reported that screw speed had no significant effect on expansion ratio (Ding et al., 2005a; Sebio and Chang, 2000).

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 also affect product texture. Lowering the feed rate will reduce the fill of the 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).

*Screw and Die configuration*

A number of different screw segments and locks can be assembled on an extruder shaft, with their effects on the final product ranging from minor to profound. Functionally, some segments convey raw and preconditioned material into the extruder barrel, while other segments compress and degas the feedstock. Where kneading is required, kneading screws may have one or multiple segments (Burtea, 2001). 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 extruded semi-finished products. The construction and shape of the die are important to give uniform flow and correct dimensional size (Sokhey et al., 1997; 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). Die shear rates may be altered dramatically by changing from a single die with one opening to a triple or quadruple die with multiple openings and flow channels. In general, dies with high shear rates cause starch-bearing products to have increased stickiness, increased water absorption and increased solubility (Burtea, 2001).

### 1.1.5 Evaluation of Extruded Products

Extruded products can be characterized according to physical, functional, textural and sensory properties, which are affected from composition of raw materials, extrusion operation conditions and post extruder operations (drying, toasting, flavor addition). These properties are measures of the extruded product quality.

#### 1.1.5.1 Physical properties

*Expansion indices*

This property describes the degree of puffing undergone by the sample as it exits the extruder (Asare et al., 2004). The amount of expansion in food depends on the pressure differential between the die and the atmosphere (Harper, 1989) as well as the ability of the exiting product to sustain expansion. Both of these are related in part to dough viscosity. High food moisture content usually provides less viscosity than lower moisture content. Therefore, the pressure difference is larger for low moisture content foods and consequently gives high expansion product. (Suknark et al., 1997).

The extrudate expansion followed two different directions that are radial and longitudinal expansion. The measurement of radial and longitudinal expansions indices allowed a better description of the cell structure of expanded extrudates (Alvarez-Martinez et al., 1988). More often, expansion is expressed either as the ratio between the cross-sectional area of the rod-shaped product and the area of die (Mercier and Feillet, 1975) or as the ratio between the diameters of the extruded product and the die, which is called the expansion ratio (Faubion and Hoseney, 1982). Longitudinal expansion index is calculated indirectly by doing a mass balance across the extruder.

*Bulk density*

The bulk density is a very important product quality attribute from the view of commercial production of extruded products. Bulk density, expressed as $g.cm^{-3}$, measures expansion. It is measured by a displacement method using sand or glass beads (Colonna et al., 1989). Bulk density also can be measured by weighing known quantity of extrudates required to fill known volume (Baik et al., 2004). Moisture content and process temperature appear to be the most important factors predicting bulk density (Rayas-Duarte et al., 1998). An increase in feed moisture content generally results in extrudates with lower expansion and higher bulk density (Baik et al., 2004). The high dependence of product density on feed moisture reflects its influence on the rheological characteristics of the starch-based material (Ilo et al., 1999). The bulk density of extrudate increases with decreasing expansion ratio (Suknark et al., 1997; Rayas-Duarte et al., 1998).

### *Color*

Color is also an important characteristic of extruded foods. The processing conditions used in extrusion cooking, high temperature and low water content, are known to favor nonenzymic browning reactions (Maillard reaction, sugar caramelisation) in extruded foods. The most characteristic consequence of the nonenzymic browning reactions is the formation of colored compounds which influence the appearance of extruded products. In many instances, it is highly desirable; it gives suitable appearance and a good flavor. However, nonenzymic browning is detrimental to product quality if it is too extensive, undesirable colors and flavors may appear. The changes of color may be used as a measurable symptom of the nonenzymic browning reactions and extent of browning provides a quantitative indicator of the extent of chemical reaction (Ilo and Berghofer, 1999).

### *Texture*

Texture is considered one of the four acceptability factors evaluating food products, the other three being flavor, appearance and nutrition. Upon heating and working during the extrusion process, the macromolecules in food ingredient lose their native, organized tertiary structure and form continues viscous dough. The laminar flow within the channels on the extrusion screw and die aligns the large molecules in the direction of flow, exposing bonding sites which lead to crosslinking and a reformed, expandable structure that creates the crunchy and chewy texture in fabricated foods (Harper, 1986). The rapid flashing of water forms the characteristics texture of extruded products when the starch melt comes out of the extruder die. As the pressure is suddenly reduced from a high pressure in the extruder to atmospheric pressure, water changes from liquid to vapor. 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 (Chessari and Sellahewa, 2001). The texture of the extruded food product is affected by the shear environment in the extruder screw and die, the type of ingredients, and the time/temperature history necessary for chemical crosslinking of the molecules (Harper, 1986).

Expansion and mechanical properties of extrudates contribute to textural attributes, such as crispness, brittleness, and toughness. Crispness of cereals is highly related to their water content or the water activity. When exceeding a critical water activity, water leads to plasticization and softening of the starch-protein matrix, thus altering the strength of the product (Heidenreich et al., 2004). Critical water activities, at which the products lose their typical crispness, are at a water activity level of approximately 0.5 (Katz and Labuza, 1981). Feed moisture and temperature have significant effect on extrudate hardness. The hardness of extrudate increases as the feed moisture content is increased. This might be related with the reduced expansion caused by the increase in moisture content. Increasing temperature slightly increases the crispness of the extrudate (Ding et al., 2005a; Ding at al., 2005b; Liu et al., 2000).

Mechanical properties (shear stress, breaking strength, resistance to rupture or fracture, etc.) of extrudates have been studied by several researchers (Falcone and Phillips, 1988; Bahattacharya and Prakash, 1994; Rayas-Duarte et al., 1998; Ilo et al, 1999; Onwulata et al., 2001). Many authors measured the breaking strength to evaluate the textural properties of the extrudate (Rayas-Duarte et al., 1998; Ilo et al, 1999; Onwulata et al., 2001). Low breaking strength values are usually related to a large number of small cells per unit area with thinner cell walls, resulting in crispy texture and reduced hardness (Rayas-Duarte et al., 1998; Onwulata et al., 2001). The maximum peak force from texture analyzer represents the resistance of extrudate to initial penetration and is believed to be hardness of extrudate, whereas the area under the curve is represented as crispness for the rice-based extruded product (Ding et al., 2005a).

### 1.1.5.2 Functional properties

When extruded starches are dispersed in an excess of water, their main functional properties are water absorption and water solubility. After grinding, extruded products present some solubility, leading to a thickening behavior (Colonna et al., 1989). Water solubility and absorption parameters characterize the extruded product and are often important in predicting how the extruded material may behave if further processed.

*Water absorption index*

Water absorption index measures the volume occupied by the starch after swelling in excess water, which maintains the integrity of starch in aqueous dispersion (Mason and Hoseney, 1986). It is the weight of gel obtained per gram of dry sample (Colonna et al., 1989). 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). Water absorption index can be used as an index of gelatinization (Sacchetti et al., 2004; Ding et al., 2005a; Ding et al., 2005b)

*Water solubility index*

The water solubility index expresses the percentage of dry matter recovered after the supernatant is evaporated from the water adsorption determination (Anderson et al., 1969). Water solubility index 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). Water solubility index, 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., 2005b).

### 1.1.5.3 Sensory properties

The extruded products can be characterized by their sensory properties mainly appearance, color, flavor and mouth feel. The shape and size of extruded products are largely determined by the design of the die, the cutting system and the degree of expansion. The shape and size are also affected by the melt rheology, which in turn is controlled by the formulation and operating conditions (Colonna et al., 1989; Chessari and Sellahewa, 2001).

Extrusion cooking essentially a short time high temperature process and a number of traditional `cooked` flavors are not produced. As in traditional cooking process, flavor and color generated during extrusion cooking by a number of reactions (especially Maillard reaction) which are controlled by the composition, temperature and residence time. However, these reactions are accelerated in extrusion cooking because of shear forces (Chessari and Sellahewa, 2001). Many extruded foods are bland since there is little time for flavor development. Volatile flavors flash off with water vapor when the food exits the extruder at the die (Camire, 2000). Therefore, flavors are usually added after extrusion to make the product more palatable. Extruded foods are generally toasted to improve the color of final product. Some caramelisation and Maillard reactions take place, which introduces flavors as well as dark color (Chessari and Sellahewa, 2001). However, excess reactions are detrimental to product quality if it is too extensive, undesirable colors and flavors may appear (Ilo and Berghofer, 1999).

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. Crunchy products, which are often porous and brittle, constitute a large category of breakfast cereals and snack foods. Many of these products are formed by high-temperature, short-time extrusion, which effectively puffs the material into a cellular and open structure. Texture in these products arises from an incremental and progressive fracturing of cell wall components in response to deformation. During mastication such fracturing gives rise to specific sensory perceptions such as crunchiness (Barrett et al., 1994). It is generally accepted that crispness, which is perceived through a combination of tactile, kinesthetic, visual and auditory sensations, represents the key texture attributes of dry snack products such as breakfast cereals or extruded rice crisps (Szczesniak, 1990).

### 1.1.6 Nutritional Properties of Extruded Products

Maintaining and increasing the nutritional quality of food during processing always important for the food processors. Extrusion cooking is preferable to other food processing techniques in terms of continuous process with high productivity and significant nutrient retention, owing to the high temperature and short time required (Guy, 2001). Extruded foods have been proven to provide nutritious products and combine quality ingredients and nutrients to produce processed foods that contain precise levels of each required nutrient (Cheftel, 1986). Although snack foods were among the first commercially successful extruded foods, today extruders produce many foods of nutritional importance. The ability of extruders to blend diverse ingredients in novel foods can also be exploited in the developing functional foods market. Nutritional concern about extrusion cooking is reached at its highest level when extrusion used specifically to produce nutritionally balanced or enriched foods, like weaning foods, dietectic foods, and meat replacers (Singh et al., 2007a). Functional ingredients such as soy and botanicals that are relatively unpalatable alone can be incorporated into new food items. Traditional foods can be further enhanced by addition of extra dietary fiber during extrusion. Anti nutritive compounds can be reduced during extrusion to provide safer and more nutritious foods (Camire, 2001).

Prevention or reduction of nutrient destruction, together with improvements in starch and protein digestibility, is clearly of importance in most extrusion applications. Five general chemical and physicochemical changes can occur during extrusion cooking: binding, cleavage, loss of native conformation, recombination of fragments, and thermal degradation. Composition of feed materials is altered by physical losses including leakage of oil and evaporation of water and volatile compounds at the die. So, most chemical reactions occur in the portion of the barrel just before the die (Camire, 2000).

*Protein and amino acids*

Protein nutritional value is dependent on the digestibility as well as the level and availability of essential amino acids (Asp and Bjorck, 1989). Protein digestibility value of extrudates is higher than nonextruded products. Most proteins undergo structural unfolding and/or aggregation when subjected to moist heat or shear (Cheftel, 1986). Extrusion improves protein digestibility via denaturation, which exposes enzyme-access sites (Camire, 2001).

At more severe extrusion conditions, the protein digestibility and availability of amino acids may be reduced (Asp and Bjorck, 1989). High barrel temperatures and low moistures promote Maillard reactions during extrusion. Reducing sugars, including those formed during shear of starch and sucrose, can react with lysine thereby lowering protein nutritional value. Lysine is the limiting essential amino acid in cereals, and further depletion of this nutrient can impair growth in children (Camire, 2001). Lysine may thus serve as an indicator of protein damage during processing (Asp and Bjorck, 1989). Cheftel (1986) concluded that, to minimize lysine loss, product temperature should be kept below 180°C, particularly at low moisture content (<15%). An advantage of extrusion cooking is the destruction of antinutritional factors, especially trypsin inhibitors, haemagglutinins, tannins and phytates, all of which inhibit protein digestibility (Singh et al., 2007a). The protein nutritional value of extruded products generally compares favorably with that obtained by alternative methods of processing (Asp and Bjorck, 1989).

*Starch*

Starch changes during extrusion have important nutritional effects. Extrusion conditions and feed materials must be selected carefully to produce desired product. For example, a weaning food should be highly digestible, yet a snack for obese adults should contain little digestible material (Camire, 2001). Humans can not easily digest ungelatinised starch. Extrusion cooking is somewhat unique because gelatinization occurs at much lower moisture levels than is necessary in other forms of food processes (Camire, 2000; Singh et al., 2007a). Extrusion cooking, depending on process conditions and food mix composition, causes swelling and rupture of starch granules, modification of crystalline spectra and DSC peaks, cold water solubility and reduced viscosity of starch and partial to complete release of amylose and amylopectin. It probably increases the enzymatic availability of starch by 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 (Cheftel, 1986). The degree of gelatinization is one important determinant for the rate of enzymatic starch hydrolysis and intestinal absorption.

Extrusion conditions can be manipulated to produce digestion-resistant starch by several mechanisms. As branches are removed from amylopectin molecules, they could react with other carbohydrates in novel linkages that can not be digested by human enzymes (Camire, 2001). Undigestible starch fractions would behave physiologically the same as dietary fiber. Amylose-lipid complex formation can also reduce starch digestibility. The extent of amylose-lipid complex formation is dependent upon both starch and lipid type present in food (Singh et al., 2007a).

### *Lipids*

Extrusion of high-fat materials is generally not advisable, especially in the case of expanded products, as lipid levels over 5-6% impair extruder performance (Camire, 2000). The nutritional value of lipids could be affected during extrusion as a result of oxidation, hydrogenation, isomerization or polymerization (Cheftel, 1986). In general, food lipid content appears to be lower after extrusion. Some lipid may be lost at the die as free oil, but this situation only occurs with high-fat materials such as whole soy. Another explanation for the lower lipid level is formation of complexes with amylose or protein (Camire, 2000). Lipid oxidation is a major cause of loss of nutritional and sensory quality in foods and feeds. However, lipid oxidation does not occur during extrusion due to short residence time, it occurs during storage (Camire, 2001). Extrusion inactivation of lipase and lipoxidase helps protect against oxidation during storage, but the porosity of expanded foods is detrimental with respect to rancidity (Cheftel, 1986).

### *Dietary fiber*

Cereals are important sources of dietary fiber. The behavior of fiber components is therefore of interest when whole or extracted cereal flours are extruded into products. Many extruded products, such as breakfast cereals and flat bread, are good sources of dietary fiber, and extrusion cooking seems to be suitable process for production of fiber-enriched products (Asp and Bjorck, 1989). The American Association of Cereal Chemists (2001) coined the following description of dietary fiber: `Dietary fiber is the edible parts of plants or analogous carbohydrates that are resistant to digestion and absorption in the human small intestine with complete or partial fermentation in the large intestine. It includes polysaccharides, oligosaccharides, lignin, and associated plant substances. Dietary fibers promote beneficial physiological effects including laxation, and/or blood cholesterol attenuation, and/or blood glucose attenuation. `

Soluble forms of fiber such as those found in fruit and gums form gels in the small intestine. The increased viscosity is believed to retard the absorption of glucose, preventing spikes in post-prandial serum glucose levels (Camire, 2000). Insoluble dietary fiber has also health benefits. One possible effect is protection against colorectal cancer by binding dietary carcinogens (Camire, 2001). At mild and moderate conditions, extrusion cooking does not significantly change dietary fiber content but it solubilises some fiber components. At more severe conditions, the dietary fiber content tends to increase, mainly owing to the increases in soluble dietary fiber and enzyme-resistant starch fractions (Singh et al., 2007a).

*Vitamins*

As vitamins differ greatly in chemical structure and composition, their stability during extrusion is also variable. Minimizing temperature and shear within the extruder protects most vitamins (Singh et al., 2007a). Cereals constitute an important source of B-vitamins. Thiamine is the water-soluble vitamin most susceptible to thermal processing. Riboflavin appears to be retained well, whereas thiamine retention seems to be highly dependent on process condition. Niacin, pyridoxine, and folic acid appear to be comparatively stable during extrusion cooking (Asp and Bjorck, 1989). Athar et al. (2006) concluded that short time high-temperature cooking of extruded snacks allows the retention of higher levels of heat labile B vitamins than the longer time and lower temperature cooking methods used in modern snack extruders. They showed that, stability of B vitamins was similar, with riboflavin and niacin having the highest stability during extrusion process. Ascorbic acid (vitamin C) is lost in the presence of heat and oxidation. This vitamin decreased in wheat flour when extruded at high barrel temperatures at fairly low moisture content (10%) (Andersson and Hedlund, 1990). The best storage stability resulted when the vitamin was added after extrusion (Asp and Bjorck, 1989). Among the lipid-soluble vitamins, vitamin D and K are fairly stable. Vitamins A and E and their related compounds, carotenoids and tocopherols, respectively, are not stable in the presence of oxygen and heat (Camire, 2000).

### *Minerals*

The digestibility and bioavailability of Fe, Cu, Zn and Mg is usually low in cereals and in leguminous seeds since these elements may be present as insoluble complexes with dietary fibers, phytate or proteins (Cheftel, 1986). Extrusion can improve absorption of minerals by reducing other factors that inhibit absorption. Extrusion hydrolyses phytate to release phosphate molecules (Singh et al., 2007a). Mineral absorption could be altered by fiber components. Cellulose, lignin and some hemicelluloses affect the mobility of the gastrointestinal tract and interfere with the absorption of minerals (Singh et al., 2007a).

### *Phytochemicals*

Whole grains contain unique phytochemicals that complement those in fruits and vegetables when consumed together. For instance, various classes of phenolic compounds in grains include phenolic acids, anthocyanidins, quinones, flavonols, chalcones, flavones, flavanones, and amino phenolic compounds. Some of these phytochemicals such as ferulic acid and diferulates are predominantly found in grains but are not present in significant quantities in some fruits and vegetables. The most important groups of phytochemicals found in whole grains can be classified as phenolics, carotenoids, vitamin E compounds, lignans, b-glucan, and inulin (Liu, 2007). These biologically active phytochemicals are found to be beneficial in reducing risk of many diseases (Singh et al., 2007a). Phenolic compounds protect against oxidation, disease, and predation. These compounds, including the large flavonoid family, are the focus of numerous studies to elucidate their role in human health. Total free phenolics, primarily chlorogenic acid, decreased significantly, owing to extrusion in potato peels produced by steam peeling. More phenolics are retained with higher barrel temperature and feed moisture. It might be possible that lost phenolics react with themselves or with other compounds to form larger insoluble materials (Camire, 2000). Extrusion caused a reduction in total phenolics of extruded oat cereals by 24-46% (Viscidi et al., 2004). The total antioxidant activity value of samples decreased with an increase in screw speed and decrease in moisture content, while total phenolics values had insignificant changes after extrusion (Özer et al., 2006).

Natural phenolic compounds added to grains prior to extrusion cooking may synergize and protect the endogenous antioxidants. These added antioxidants would be evenly dispersed within the food matrix, resulting in delayed onset of lipid oxidation and thus, longer the shelf life of extruded foods (Viscidi et al., 2004). Enhanced performance of extrusion-created antioxidants compared with synthetic antioxidants may appeal to consumers wanting `natural` foods. Camire et al. (2007) investigated the effects of dehydrated fruit powders as colorants and antioxidants in extruded white cornmeal breakfast cereal. They found that anthocyanins from fruit powders survived extrusion and retain some antioxidant activity.

### 1.1.7 Use of Extrusion Cooking Process for New Product Development

Extrusion system for the production of food products is efficient, economical to run, and produce a final product with built in flexibility. A careful balance of available raw material and final product characteristics is needed to assemble the correct extrusion to system ultimately resulting a successful and profitable final product. In addition to the usual benefits of heat processing, extrusion offers the possibility of modifying the functional properties of food ingredients and/or of texturizing them. The advantages of extrusion are evident, especially in the simplification of processing techniques for the manufacture of existing products as well as in the development of novel types of food.

New bulgur-like products were developed by extruding durum wheat at various levels of feed moisture content, screw speed and feed rate. It was concluded that overall acceptable quality characteristics of bulgur-like product were comparable to those of commercial bulgur and the new process requires less time and seems to be more economical than traditional bulgur production process (Köksel et al., 2003). Effects of extrusion temperature and feed composition on the functional, physical and sensory properties of chestnut and rice flour-based snack-like products were studied by Sacchetti et al. (2004). Chestnut flour was found to be suitable for the extrusion-cooking process adopted if properly mixed with rice flour, with 30% chestnut flour percentage processed at 120°C producing a snack-like product with limited density and browning that was judged good by a sensory panel.

Products with new functional and nutritional properties are a precondition for a higher acceptance of barley in human nutrition. Barley varieties with different contents of β-glucan and amylose were used as primary material for the preparation of extruded products. It was stated that extrusion technology is a suitable method for preparation of products from barley with increased resistant starch content, whilst the β-glucan can be preserved in a macromolecular form (Huth et al., 2000). Baik et al. (2004) investigated the production of extruded ready-to-eat breakfast cereals from different barley flours under different extrusion conditions. Characteristics of barley extrudates produced from different barley granules were found to be influenced by the type of barley as well as milling streams, moisture content, barrel temperature, feed rate, and screw speed.

Cereals are usually fortified with new protein sources such as oilseed and legumes for production of nutritious products. The blending of peas with cereals can complement each other so that protein in the resulting product more nearly resembles that of a complete or balanced protein (Singh et al., 2007b). It was reported that extruded chickpea flours may be considered for the fortification of widely consumed cereal-based food products (Milan-Carrillo et al., 2000). Asare et al. (2004) proposed a model describing the optimal process variables for the extrusion of puffed snack with enhanced product quality characteristics from rice-cowpea-groundnut blend. The model suggested low feed moisture 14-20%, maximum amount of 20% cowpea and 10% groundnut addition for optimum product quality. Partially defatted peanut flour was used as protein source in the production of expanded extrudates formulated from partially defatted peanut flour and different types of starch. The study showed that directly expanded snack-like products could be made from combinations of various starches and up to 30% partially defatted peanut flour (Suknark et al., 1997).

A standardized extrusion cooking process was developed for production of a high protein weaning food based on peanuts, maize and soybeans. The study has established the blend formulation and extrusion cooking for the production of a highly acceptable nutritious weaning food that can adequately replace the existing low quality traditional products in West Africa (Plahar et al., 2003). Bahattacharya and Prakash (1994) studied the extrusion of blends of rice and chick pea flours, containing 20% moisture through single screw extruder. It was observed that acceptable snacks could be produced from blends of rice and chick pea flour. The nutritional quality of extruded unmalted and malted maize fortified with cowpea as complementary food was assessed based on its proximate analysis, amino acid composition. It was reported that the mixture of cowpea and maize, especially the malted mixture, would serve as a good quality complementary food for weaning children in terms of protein and energy adequacy (Obatolu et al., 2000). Pelembe et al. (2002) developed as instant high protein porridge by extrusion of various ratios of sorghum and cowpeas. They found that a serving of 100 g porridge would contribute 28% of the recommended dietary allowance for protein. Nourishing breakfast cereal has been developed by manufacturing black currant seeds, which have significant amounts of essential fatty acids, mineral and dietary fiber, through high temperature extrusion process. A product with more than 40% black currant press residue has found to be hard and was not expand well (Tahvonen et al., 1998).

Whey products including sweet whey solids and whey protein concentrate was incorporated into extruded products to improve the nutrient content of snacks by increasing the protein content (Onwulata et al., 2001). Extrudates with good quality were produced with up to 25% whey protein substitution for corn meal, potato and rice flour. An acceptable snack that incorporates whey proteins such as whey protein concentrate or sweet whey solids could be produced through the extrudates may not be significantly expanded (Onwulata et al., 2001). Mendonça et al. (2000) studied the utilization of corn bran which has high dietary fiber content through the production of expanded snacks from corn meal using the extrusion process. They observed that snacks prepared with 250 g/kg corn bran, higher temperature, lower moisture content and medium level of glycerol monostearate gave the best combination of general acceptability, expansion and fiber. The effects of pectin alone or in combination with wheat fiber on the physical and structural properties of extruded corn starch were studied using a laboratory single screw extruder. Pectin increased porosity and reduced expansion ratio and hardness but there was no apparent effect of pectin on the size and the number of the cells (Yanniotis et al., 2007).

Various tomato powder-starch formulations were extruded using single screw extruder for production of nutritious snacks. The optimum process conditions were determined using response surface methodology. It was concluded that the process can be referred for industrial production, and the extrudates were suitable for extruded snack food with tomato powder (Huang et al., 2006). Brewer`s spent grain, which is the main by-product of the brewing industry, was incorporated into ready-to-eat expanded products using extrusion technology. It was observed that up to 30% of brewer`s spent grain can be added with acceptable physicochemical characteristics, although the addition of 20% brewer`s spent grain was considered better for developing snacks with similar properties to those commercially available (Stojceska et al., 2008). Camire et al. (2007) investigated the effects of dehydrated fruit powders as colorants and antioxidants in extruded white cornmeal breakfast cereals. They concluded that higher levels of fruit powder will increase production costs, but the expense may be off set by the more attractive and functional cereals that result.

## 1.2 Food Processing Wastes

Every industry generates a waste stream. This material is costing the manufacturer more for disposal and may have a significant environmental impact. For many food processing plants, a large fraction of the food processing wastes are produced and most of them is currently disposed or used on a low technological and economical level. In the past, it has been relatively easy to dispose of the waste components of processing operations by dumping them. But now, this is becoming much more expensive, and businesses are examining the options available for them to value-add this material (Laufenberg et al., 2003).

Waste management, placement and disposal, are critical aspects of food processing. The percentage of by-products and wastes in food processing is approximately 30%. The majority of food industry wastes and by-products (of plant origin) is used as animal feed. Only 2-3% is marketed for human use (Monspart-Senyi, 2006). The cost of manufacturing food is becoming increasingly dependent on the ability of the manufacturer to get value from all products of the process. The concept of producing food from waste has become of great interest in the recent years as a result of increasingly frequent food shortages and price rises.

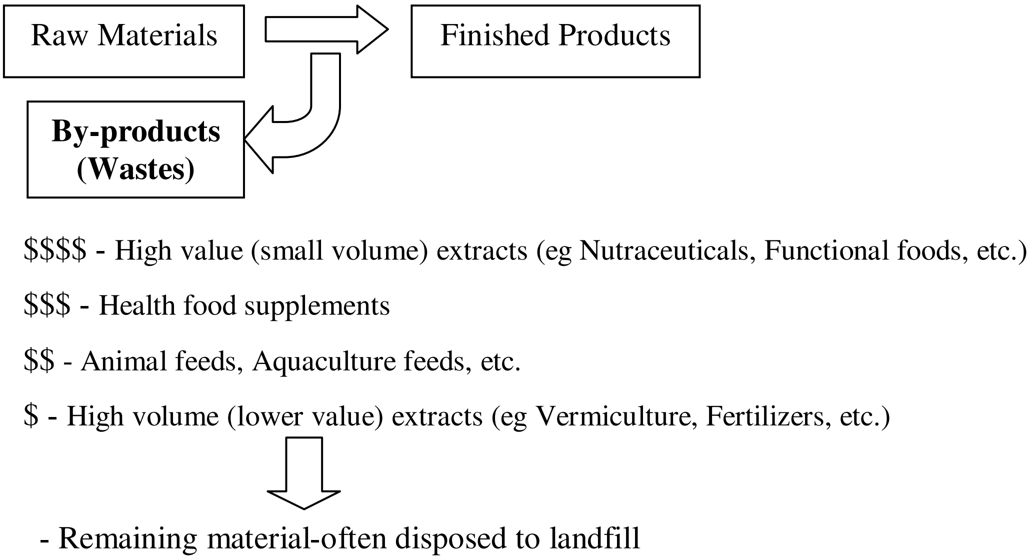
Food processing wastes are promising sources of compounds which may be used because of their favorable technological or nutritional properties. Besides their hazardous or pollution expect, in many cases, food processing wastes might have a potential for recycling raw materials or for conversion into useful products of higher value as a by product, or even as a raw material for other industries, or for the use as food after biological treatment (Laufenberg et al., 2003). They also have health benefits and many functional properties. Food processing wastes include fruit and vegetable peels, seeds and pomaces, cheese whey, blood, bone, grain by-products, process water, waste water treatment sludge and so on. Of all wastes generated by the food industry, fruit and vegetables together account for 93%. Currently nearly all the liquid waste is disposed of in water (stream, lake, ocean) and in sewers (public waste treatment systems), whereas most solid wastes are disposed of by returning them to the land for treatment (Hang, 2004).

### 1.2.1 Environmental and Economic Impacts of Food Processing Wastes

The food industry is not a heavy environmental polluting industry. Some 30% of all production waste are called dangerous waste (mainly of animal origin), which requires specific treatment (Monspart-Senyi, 2006). The food processing waste is generally characterized by its high proportion of organic material and can pollute our environment if not disposed prudently. Due to increasing production, disposal represents a growing problem, especially in the plant wastes. On the other hand, costs of drying, storage and shipment of wastes are economically limiting factors (Schieber et al., 2001). Disposing of this waste can be difficult for the following reasons (Russ and Meyer-Pitroff, 2004):

- Biological stability and the potential growth of pathogens: Many types of waste material either already contain large numbers of microbes and/or will be altered quickly through microbial activity.
- High water content: The water content of meat and vegetable waste lies between 70 and 95% by mass. High water content increases transport costs of the waste.
- Rapid autoxidation: Waste with a high fat content is susceptible to oxidation, which leads to the release of foul-smelling fatty acids.
- Changes due to enzymatic activity: In many types of wastes arising from vegetables and fruits, enzymes are still active, which accelerate or intensify the reactions involved in spoilage.

Environmental legislation has significantly contributed to the introduction of sustainable waste management practices throughout the European Union. By the year 2010, organic waste disposal has to be reduced by 80 % and the utilization rate of food industry waste has to be raised (European Council, 1999). The opportunities exist for the production sector to participate in value adding through utilization of food wastes. Not only will this approach reduce the cost of disposal to processors, but the products are valuable in their own right (Figure 1.6). The process is also influenced by economical conditions since the expense of converting waste products into usable products will happen only if the expenses do not exceed the final selling price (Monspart-Senyi, 2006).



$$$$ - High value (small volume) extracts (eg Nutraceuticals, Functional foods, etc.)

$$$ - Health food supplements

$$ - Animal feeds, Aquaculture feeds, etc.

$ - High volume (lower value) extracts (eg Vermiculture, Fertilizers, etc.)

- Remaining material-often disposed to landfill

Figure 1.6 Values of the food processing by-products

### 1.2.2 Waste Reduction and Recovery Hierarchy

Food processing waste utilization and management hierarchy is the management strategy for producing the greatest benefit to the industry, environment, and society. In the order of descending benefit, food processing residual management options may be grouped as fallows (Magbunua, 2000):

*Source reduction and water conservation:* This management strategy reduces excessive waste generation. Source reduction can be accomplished by reducing material loss, conserving and reusing water, and preventing spills (Magbunua, 2000). Any reductions in the volume of water used in food processing will result in corresponding reductions in the amount of waste water to be treated (Hang, 2004).

*Recovery for human uses:* This management strategy recovers food processing residuals for human ingestion, personal care, home use, or commercial/industrial use. Some examples of food processing residuals recovered for human uses include thermally modified whey proteins used as food additives and starch-based biodegradable packaging materials (Magbunua, 2000). Grape seed extracts may be exploitable for the preservation of food products as well as for health supplements and nutraceuticals (Moure et al., 2001; Jayaprakasha et al., 2001).

*Recovery for animal uses:* Most food processing wastes are presently used for animal feed. They may be fed fresh as silage, or as dried waste solids (Hang, 2004). Examples include pet food and livestock feed from meat, poultry and seafood processing residuals and citrus, pineapple and potato processing residuals.

*Recovery for soil conditioners or fertilizers (land application)*: Food processing wastes can be beneficially used as soil conditioners or fertilizers. This can be accomplished either through composting or through land application. Although ranked relatively low on this hierarchy, composting is typically perceived as the conversion of a waste material into a product of commercial value. Conversely, land application is often viewed as a disposal option. However, properly managed land application programs strive to replenish soil organic matter and nutrients that have been depleted through cropping, with the objective of replacing conventional soil supplements and chemical fertilizers with food processing residuals (Magbunua, 2000).

*Food Processing Waste Treatment and Disposal:* Food processing residuals are wastes at this point at the hierarchy. The goal of this disposal strategy is to efficiently remove the contaminants that are potentially harmful to the environment (Hang, 2004).

### 1.2.3 Important Types of Wastes from Food Industry

Current products from different food processing wastes are listed in Table 1.1, along with intermediate- and long-term product goals for these residuals. Among the food processing industries, the first four industry groups listed (meat, poultry and seafood processing; bakery operations and grain processing; fruit and vegetable processing; and nut and oilseed processing) produce the largest quantities of residuals and would probably deserve the greatest amount of attention and resources with respect to waste reduction efforts (Magbunua, 2000).

Table 1.1 Current and potential products from different food processing residuals (Magbunua, 2000)

| Industry | Wastes | Current products/ Disposal methods | Future Products | |
|---|---|---|---|---|
| | | | Intermediate | Long-term |
| Meat, poultry and seafood processing | Offal, blood, feathers, DAF sludge | Animal feed | Animal feed including lactic acid fermentation | Protein isolates, animal fats, biofuels and insulation materials |
| Bakery operations and grain processing | Waste dough, bread, bakery ingredients, waste grain, brewer's spent, yeast | Animal feed, ingredients in bakery products | Feed ingredients via SCP production or lactic acid fermentation<br>Commodity chemicals | Specialty chemicals |
| Fruit and vegetable processing | Trimmings, culls and pomace | Landfill, land application and animal feed | Feed ingredients via SCP production or lactic acid fermentation, pectin production and biogas production | Biofuel, commodity and specialty chemicals |
| Nut and oilseed processing | Hulls, meals | Compost, animal feed and plastic filler | Fermentation feed stocks, Biofuels and Commodity chemicals | Specialty chemicals |
| Dairy products | Whey | Food and feed in limited quantities | Commodity and specialty chemicals | Specialty chemicals |
| Beverage products | Waste beverage | Municipal sewer | Biofuel | Specialty chemicals |

### 1.2.4 Fruit and Vegetable Wastes

By-products of plant food processing represent a major disposal problem for the industry concerned, but they are also promising sources of compounds which may be used because of their favorable technological or nutritional properties (Schieber et al., 2001). By-products are produced during main steps of the fruit and vegetable production chain. Such by-products may come from (Monspart-Senyi, 2006):

- overstock merchandize
- screenings, tops, stems, pulps, pomace, skins, hulls, peels, meals, seeds, fines, green chop, pressed cake, dried fruit, and fresh fruit waste
- below standard (size, color and texture)
- trimmings
- fruit harvest

Agricultural residues are attractive sources of natural antioxidants. The replacement of synthetic antioxidants by natural ones may have benefits due to health implications and functionality in food system. The natural antioxidants from residual sources may be used for increasing the shelf life of food by preventing lipid peroxidation and protecting from oxidative damage. Oxidative damage to cellular components such as lipids and cell membranes by free radicals and other reactive oxygen species is believed to be associated with the development of a range of degenerative diseases including cancer and atherosclerosis (Moure et al., 2001).

Within the antioxidant literature the number of studied residual sources has been augmented considerably, which is caused by a value adding recycling interest of the agro- and food industry, but also increasing information on the specific location of active compounds and their modification during processing. Studies regarding the fruit and vegetable wastes are mainly focused on the polyphenol content which acts as free radical scavenger. Apple pomace has shown to be a good source of polyphenols which are predominantly localized in the peels and are extracted into the juice to a minor extent. Major compounds isolated and identified include catechins, hyroxycinnamates, phloretin gycosides, querreetin glycosides, and procyanidins. Phenolic constituents of apple pomace have been demonstrated to exhibit strong antioxidant activity (Lu and Yeap Foo, 1997; Lu and Yeap Foo, 2000). Grape skin and seed are a rich source of monomeric phenolic compounds, such as catechin, epicatechin, and these compounds act as antimutagenic and antiviral agents (Moure et al., 2001; Jayaprakasha et al., 2001). Winery wastes, including grape seed, pomace and stems have been reported to be very rich sources of antioxidant polyphenols compared with other agricultural solid wastes (Arvanitoyannis et al., 2005; Makris et al., 2007). The peel and seed fractions of some fruits have higher antioxidant activity than the pulp fractions (Jayaprakasha et al., 2001). Pomegranate peel (Negi et al., 2003), residues from juice and canning industry (red beet, apple, strawberry, tomato and asparagus) (Peschel et al., 2006) and coconut, banana, dragon fruit, rambutan and passion fruit peels (Okonogi et al., 2007) have been indicated as good sources of antioxidants.

Many fruit and vegetable residues i.e. apple pomace, orange peel, potato peel are rich in dietary fiber which preventing and treating many diseases such as intestinal problems, cancer and diet related health problems (Walter et al., 1985; Arora et al., 1993; Camire et al., 1997; Larrea et al., 2005b). An increase in the level of dietary fiber in the daily diet has been recommended (25-30 g/day). Fiber incorporation, in frequently consumed food, could help to overcome the fiber deficit (Figuerola et al., 2005). Different types of dietary fibers, such as pea, apple, sugar beet, soy and citrus fibers, as well as inulin and gums, are now incorporated into foods for their nutritional properties or for their functional and technological properties such as gelling or thickening properties. Dietary fiber fractions of various wastes have been studied in the literature. Nawirska and Kwasniewska (2005) compared the amounts of particular dietary fiber fractions in the apple, cherry, chokeberry, black currant, pear and carrot pomace. The results revealed that pectins occurred in the smallest amounts; however lignin content was very high in each pomace samples. Apple and citrus fruit residues have been found to contain high dietary fiber content with a high proportion of insoluble dietary fiber (Figuerola et al., 2005). Many authors investigated the use of apple pomace as a source of dietary fiber and potential ingredient in the bakery products (Wang and Thomas, 1989; Masoodi and Chauhan, 1998; Carson et al., 1994). Larrea et al. (2005a) studied the change in functional and structural properties of extruded orange pulp and usage of extruded orange pulp as an additive in the cookies. They reported that good technological quality cookies with a good level of acceptance were obtained by means of replacing up to 15 g/100 g of wheat flour with extruded orange pulp.

Many valuable compounds also present in fruit and vegetable wastes. Tomato seeds contain high quality plant proteins that could be supplemented into various food products (Sogi et al., 2005). Tomato seed from tomato cannery waste has been reported to have appreciable amounts of digestible amino acids, methionine, cystine and lysine (Persia et al., 2003). Moreover, most of the lycopene in tomato is associated with the skin and the water insoluble part (Schieber et al., 2001). The anthocyanins from banana bracts, abundant edible residues of banana production, have been investigated as a potential source of natural colorant. It was concluded that the bracts proved to be a good and abundant source of common anthocyanidins which are delphinidin, cyanidin, pelargonidin, peonidin, petunidin and malvidin (Pazmiño-Duràn et al., 2001). Carrot pomace has been evaluated as natural source of α- and β-carotene, which may be recovered and used as functional food ingredient (Stoll et al., 2003).

### *Citrus Processing By-products*

In contrast with other types of fruits, citrus fruits have a small edible portion and large amounts of waste material such as peels and seeds. The traditional citrus by-products of cattle feed pellets and essential oils are still the most important utilization of juice processing residue. Citrus fruit residue remaining from juice extraction amounts to approximately half the wet mass of the whole fruit, with the peel (flavedo and albedo) almost one-fourth the whole fruit mass (Braddock, 1999). Residues of citrus juice production are a source of dried pulp and molasses, fiber-pectin, cold pressed oil, essences, D-limonene, juice pulps and pulp wash, ethanol, seed oil, pectin, ascorbic acid, limonoids and flavonoids (Braddock, 1999). Citrus by-products have a number of potential uses. They could be used as functional ingredients when designing healthy foods, especially non-digestible carbohydrates (dietary fiber) and bioactive compounds (ascorbic acid and flavonoids) (Braddock, 1999; Fernandez-Lopez et al., 2004).

Residues of citrus juice production are composed of principally water, soluble sugars, fiber, organic acids, amino acids and proteins, minerals, oils and lipids, and also contain flavonoids and vitamins (Fernandez-Lopez et al., 2004). All of these components are found in different amounts depending on the fraction of the fruit (juice, albedo, flavedo, rag and pulp, and seeds) (Bradock, 1995). After water removal, soluble and insoluble carbohydrates are the major constituents of citrus peel. The water-soluble fraction contains glucose, fructose, sucrose, and some xylose, while pectin, hemicellulose, and cellulose constitute between 50 and 70% of the insoluble fraction (Braddock, 1999). When the nutritional importance of fiber in the human diet became a popular media item, considerable interest developed in citrus peel use as a fiber source. The main advantage of dietary fiber from citrus fruits when compared to alternative sources of fiber such as cereals is its higher proportion of soluble dietary fiber. The soluble fraction was found to be greater dietary fiber concentrates of fruits and green vegetables (10-14 g/100 g) than in wheat and oat bran (3-4 g/100 g) (Grigelmo-Miguel, 1999).

Particularly high concentrations of the flavanones, hesperidin, and naringin and lesser quantities of many other flavonoids are found in citrus fruits. In mature fruit, flavonoid concentration is highest in the tissue (albedo, 30-50 of total; rag, core, and pulp, 30-50%; flavedo, 10-20%) and lowest in the juice (1-5%) (Braddock, 1999). Citrus seeds and peels were found to possess high antioxidant activity (Bocco et al., 1998). Gorinstein et al. (2001) reported that the content of total polyphenols and ascorbic acid was higher in the peels of citrus than in peeled fruits.

### *Grape Pomace and Seed*

Grape, one of the world's largest fruit crops, with more than 60 million tons produced annually. About 80% of the total crop is used in wine making. Wine making affords grape pomace as a by-product in an estimated amount of 20% by weight of the grapes (Schieber et al., 2001; Amico et al., 2004). Therefore, huge amounts of press residues originating from wine production arise within a few weeks during harvest. Grape pomace is often disposed of as a soil conditioner; however, it is also considered a source of various valuable compounds. The composition of grape pomace major constituents, peels and seeds, has been reported by several authors, with high polyphenolic as well as dietary fibre (DF) contents (Lu and Yeap Foo, 1999; Bonilla et al., 1999; Bartolome et al., 2004; Amico et al., 2004; Kammerer et al., 2005; Llobera and Canellas, 2007).

Grape pomace represents a rich source of various high-value products such as ethanol, tartrates and malates, citric acid, grape seed oil, hydrocolloids and dietary fiber (Arvaniyoyannis et al., 2005). Grape peel and seed are increasingly being used to obtain functional food ingredients, such as natural antioxidants and dietary supplements. From a nutritional point of view, polyphenols are the most important constituents of grape pomace. Grapes contain a large amount of polyphenols which include the phenolic acids, flavonoids, anthocyanins and proanthocyanidins (Lu and Yeap Foo, 1999). Functional ingredients of grape seeds include several flavonoids with a phenolic nature such as monomeric flavanols (catechin and epicatechin), dimeric, trimeric and polymeric procyanidins, and phenolic acids (gallic acid and ellagic acid). Catechins, together with other polyphenols, are potent free radical-scavengers. Grape seed oil contains vitamin E (80-120 mg per 100g), vitamin C, beta-carotene unsaponifiables rich in tocopherols, steroids (campesterol, beta-sitosterol and stigmasterol) and several fatty acids (Arvaniyoyannis et al., 2005). The association between a diet rich in polyphenols and the decrease in the risk of suffering cardiovascular diseases and certain types of cancer (Yilmaz and Toledo, 2004; Llobera and Canellas, 2007). By-products from grape have a high antioxidant capability and as a consequence of this, they are endowed with potential health benefits.

### *Tomato Pomace*

Tomatoes constitute an important agricultural crop and an integral part of the human diet worldwide. Its high nutritive value and multiple uses in culinary preparations have made it a most important vegetable. The majority of tomatoes for processing are made into products such as juices, ketchup, sauce, paste, puree and powder. The solid waste, remaining after the juice/pulp extraction process, consists of skin, seeds, fibrous matter, trimmings, cores and cull tomato. This waste is dumped, flushed into sewage, streams and rivers or to some extent used as fertilizers (Sogi et al., 2005).

Tomato pomace consists mainly of peel and seeds and fiber is the major compound of tomato pomace on a dry matter basis, at 25.4–50.0%. Other components ranged between 15.4% and 23.7% for total protein, 5.4% and 20.5% total fat, and 4.4% and 6.8% mineral content (Del Valle et al., 2006). The seeds account for approximately

10% of the fruit and 60% of the total waste, respectively, and are source of protein (35%) and fat (25%) (Schieber et al., 2001).Tomato seed from tomato cannery waste has been reported to have appreciable amounts of digestible amino acids, methionine, cystine and lysine (Persia et al., 2003). The tomato peel contains many carotenoids but lycopene is the most important responsible for red color. Lycopene exhibits the highest antioxidant activity and singlet oxygen quenching ability of all dietary carotenoids (Kaur et al., 2006). The ability of lycopene to act as a potent antioxidant is thought to be responsible for protecting cells against oxidative damage, thereby decreasing the risk of chronic diseases (Huang et al., 2006). Tomato pomace is rich in nutrients and could be used as a potential source of fiber, protein or fat. Tomato pomace utilization could provide extra income for the tomato industry and, at the same time, reduce the waste disposal problem. Tomato pomace components could improve the nutritional quality of the final products in tomato industry and could be added to different traditional foods as flours or other cereal products (Del Valle et al., 2006).

### 1.2.5 Partially Defatted Hazelnut Flour

Wild species of hazelnut, genus *Corylus*, are distributed in nearly all parts of temperate zones of the northern hemisphere while the major producers are Turkey, Italy, Spain, and USA (Özdemir, 1997). Besides their economic value, hazelnuts also add flavor and texture to many food products and play a major role in human nutrition and health (Alphan et al., 1996; Ozdemir and Akinci, 2004). Hazelnuts are marketed in shell or as kernels. The kernels are consumed as natural, roasted, sliced, chopped, diced, flour, or as roasted paste. 80 % of the hazelnut kernels are used in chocolate manufacture, 15 % in confectionery, biscuit, and pastry manufacture, and the remaining 5 % is consumed without any further processing (Özdemir, 1997). They are used to provide a sweet, delicate, yet definite flavor in the food products such as dairy, bakery, confectionery products or are used to enhance flavor of chocolate, ice cream, desserts, snack bars and side dishes (Özdemir, 1997; Ozdemir and Akinci, 2004). The principal flavor component of the hazelnut is filbertone which is formed during roasting (Buckholz et al., 1980).

Among the nut species, the hazelnut has great importance because of its special nutritional composition of proteins (15-19 %), carbohydrates (15-17 %), fat (60 %)

and vitamins. Hazelnuts are rich in protein, complex carbohydrates, dietary fiber iron, calcium, potassium and vitamin E. Its protein quality (66.6 %) is also high in comparison to many proteins of plant origin. Glutamic acid, followed by arginine and aspartic acid are present in greatest concentrations in Turkish hazelnut varieties. Hazelnut contains all essential amino acids and digestibility of protein from hazelnut is about 80-90 % (Özdemir, 1997). It appeared to be one of the best sources of plant origin for iron (5.8 mg/100g), calcium (160.0 mg/100 g), and zinc (2.2 mg/100g), which are the most important minerals for growth and development. Hazelnuts were also found to be rich in potassium (655 mg/100g), which is necessary for nerve stimulation and functioning of muscle tissue. Hazelnuts were found to be good sources for vitamins B1 (0.33 mg/100g), and B2 (0.12 mg/100g), and very good sources for vitamin B6 (0.24 mg/100g) and Vitamin E (31.4 mg/100g) (Alphan et al., 1996).

Partially defatted hazelnut flour (PDHF) is residual of hazelnut after extraction of oil parts from the main fruit. It mainly consists of defatted hazelnut kernel, whole kernel skin and non-extracted fat. It is high in protein (35-41 %), dietary fiber (10 %), and other nutritional constituents (Villarroel et al., 1989). Shahidi et al. (2007) suggested that hazelnut by-products could potentially be considered as an excellent and readily available source of natural antioxidants. They evaluated antioxidant efficacies of ethanol extracts of defatted raw hazelnut kernel and hazelnut by-products (skin, hard shell, green leafy cover, and tree leaf) by monitoring total antioxidant activity and free-radical scavenging activity tests, together with antioxidant activity in a beta-carotene-linoleate model system, inhibition of oxidation of human low-density lipoprotein (LDL) cholesterol, and inhibition of strand breaking of supercoiled deoxyribonucleic acid (DNA). It was reported that extracts of hazelnut skin, in general, showed superior antioxidative efficacy and higher phenolic content as compared to other by-products. Five phenolic acids (gallic acid, caffeic acid, p-coumaric acid, ferulic acid, and sinapic acid) were identified and quantified in these by-products. PDHF is generally used as an additive in chocolate, and biscuits manufacturing due to its low cost and adding flavoring properties to the final product. The PDHF can be used in combination with cereal flours for production of extruded food products due to its valuable characteristics.

### 1.2.6 Durum Clear Flour

The technological advances in the processing of durum wheat allow separating different fractions indicated as durum wheat by-products. These by-products can be used to improve the technological performance and/or to integrate foods with healthy compounds. Some of by-products mainly containing insoluble dietary fiber can be used to hold free water, while others, containing high amount of soluble dietary fiber, are great of nutritional value (Esposito et al., 2005). Durum clear flour is a by-product which is obtained during the semolina production by collecting the line fractions of certain streams and the flour which is extracted during grinding (Kılıç, 1999). Clear flour is the lowest quality of all commercial grades of flour. It is milled from the outer part of the endosperm made from flour streams that remain after patent flour is produced (Figoni, 2003), and composed approximately 13-16 % of the milled durum wheat (Kılıç, 1999).

Durum clear flour is relatively high in bran, high in protein (14-16 %), ash (1.5 -2 %) and starch (about 65 %) and slightly gray in color (Kılıç, 1999). Durum clear flour is less expensive than patent flour. While it is higher in total protein quality than patent flour, the gluten formed from clear flour is typically of lesser quality than that from patent flour (Figoni, 2003). The durum clear flour is higher in ash, sugar and pentosan contents than the bread wheat flour. Clear flour contains a greater amount of the outer layers of the kernel which is higher in sugar, pentosans and ash contents than the endosperm portion (Kılıç, 1999). Many authors showed that the concentration of bioactive constituents was greater in the outer layers of the grain; thus the bran fraction demonstrates higher antioxidant activity than other milling fractions (Beta et al., 2005; Esposito et al., 2005; Liyana-Pathirana and Shahidi, 2007). Esposito et al. (2005) investigated the soluble and insoluble dietary fiber content and antioxidant activity of durum wheat bran by-products. They observed that soluble fiber content ranged between 0.9-4.1%; while insoluble fiber content was between 21 and 64%. They also reported that the antioxidant activity is higher for the internal bran fractions and it increases in fractions having reduced granulometry. As phenolic compounds are found to be concentrated in the outermost layers, the bran fractions obtained as milling by-products may be used as a natural source of antioxidants and as a value-added product in the preparation of functional food ingredients and/or for enrichment of certain products (Liyana-Pathirana and Shahidi, 2007). Durum clear flour is generally utilized as additives for improving gluten strength of low gluten grains such as rye and some bakery products such as bread. The utilization of durum clear flour for production of extruded food products may increase its commercial value and usage. Durum clear flour contains significant amount of starch (about 65 %), which may give adequate expansion during extrusion cooking. The gluten in clear flour provides nutritional value, crispness and desired texture to the final extruded products (Harper, 1989).

## 1.3 The Aim of Study

In recent years, researches on evaluation of food wastes are rapidly expanding around world. Food processing wastes cause loss of several valuable constituents which are important for nutrition such as dietary fiber, antioxidants, pectin, essential fatty acids. Due to this, collection and use food wastes for production of new foods are important from the point of human health and country's economy. Extrusion system for the production of food products is efficient, economical to run, and produce a final product with built in flexibility. The advantages of extrusion are evident, especially in the simplification of processing techniques for the manufacture of existing products as well as in the development of novel types of food. Thus, the objectives of this study are:

1. To provide alternative technology for utilization of food processing wastes for development of useful, value-added food product. The primary objective of this study is particularly to develop the nourishing extruded food product (easy to consume) from durum clear flour in combination with PDHF and different pomaces (orange, grape and tomato) by using extrusion cooking technology.
2. To investigate the effects of extrusion conditions such as screw speed, feed moisture content, barrel temperature and change in feed composition on physical (radial expansion, bulk density, porosity, color, texture, water solubility and absorption indices), sensory and functional (total phenolic content and antioxidant activity, starch digestibility & gelatinization) properties of extrudates.

# CHAPTER 2

# MATERIALS AND METHODS

## 2.1 Raw Materials

Durum clear flour was obtained from Tat Macaroni Industry and Trading Corporation (Gaziantep, Turkey). Partially defatted hazelnut flour (PDHF) was obtained from Ordu Oil Industry Inc. (Ordu, Turkey) and rice grit was obtained from Üçel Food Industry and Trade Corporation (Gaziantep, Turkey). Sucrose (Helin, İstanbul, Turkey) was purchased from the market. Orange peel, grape seed and tomato pomace were obtained from Namsan Food Industry and Trade Corporation (Bursa, Turkey). They were sun-dried by spreading the samples on cloth for 3 days under sun light and milled into powder for extrusion cooking process. They were all stored in polyethylene bags at 4°C for further usage in experiments.

## 2.2 Chemical Analysis of Raw Materials

Moisture, ash, protein, fat analysis of raw materials was carried out using standard procedures of AOAC (1990). Carbohydrates were calculated by difference. All analyses are expressed as the mean (±SD) of triplicate analyses.

## 2.3 Blend Preparation

Milled orange peel (80.0%, dry basis (d.b.)), grape seed (10.0%, d.b.) and tomato pomace (10.0%, d.b.) were mixed in laboratory mixer (Kitchen Aid, USA). This blend was used as fruit waste blend in the production of extruded food. Rice grit (67%), sucrose (3%), durum clear flour, PDHF, and fruit waste blend were blended in 5 kg batch according to design of experiment. Using the mixer, blends were conditioned by adding sprayed water while tumbling in a rotating drum and mixed for 30 min at the high speed to ensure homogeneity of the feeding material before extrusion. Moisture content of prepared blends was determined by infrared moisture analyzer (Sartorius, Goettingen, Germany) at 105°C.

## 2.4 Extrusion

American Extrusion International 300 B Bake Type (South Beloit, Illinois, USA), single screw industrial scale extruder was employed for this study. The extruder was equipped with 304.8 mm barrel, feed screw, standard 12-hole die and standard baked knife blades. The extruder has one heating zone with electrical resistance heaters and thermocouple sensor to monitor the temperature. The screw was 75 mm in diameter and 290 mm long. The screw had constant pitches and gradual decrease in flight depth from 4.5 mm to 1 mm through the exit of the die. The die consisted of a 12-hole die with each hole being 2 mm in diameter. Before the extrudate was collected, care was taken to ensure that the flights at the feed section were kept full throughout the extrusion run. During each extruder run, the machine was allowed to equilibrate for 5-10 min until a stable motor current was achieved. Extrudates were collected on metal screens to allow excess steam flash off. Once cool, all samples were transferred to polyethylene bags and stored at cool and dry place.

Extrusion process variables such as feed moisture content, barrel temperature, screw speed have been studied to find the desired product by using various compositions of the blend. The blend having 67% rice grit (d.b.), 3% sucrose (d.b.), various durum clear flour (8-20%, d.b.), partially defatted hazelnut flour (PDHF) (5-15%, d.b.) and fruit waste (3-7%, d.b.) contents were extruded at different extrusion conditions. The independent extrusion operating variables considered varied in the following ranges: feed moisture content from 12 to 18%, barrel temperature from 150 to 175°C, and screw speed from 200 to 280 rpm. The steps of processes for the production of extruded samples were given in the following flow diagram (Figure 2.1).

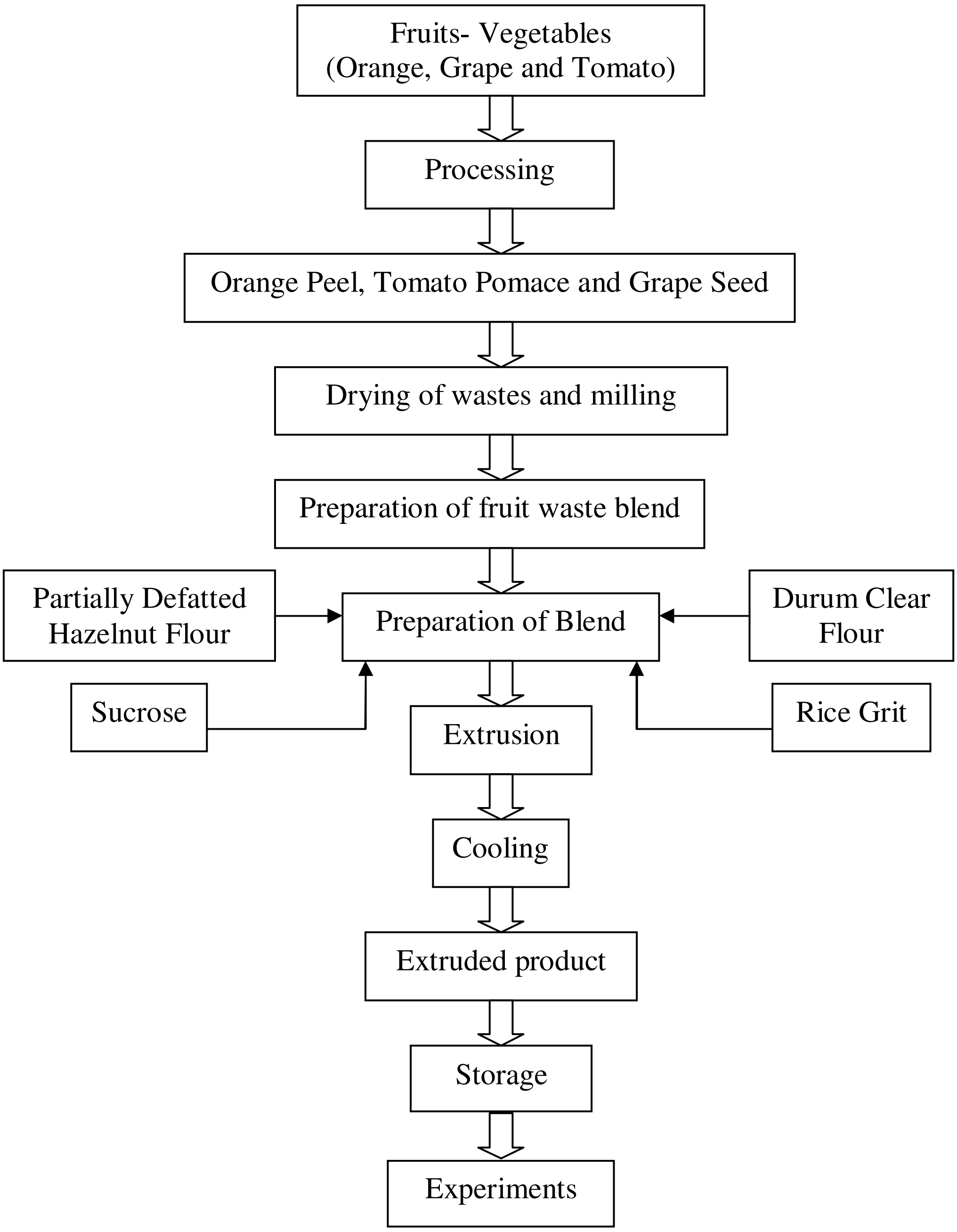


Figure 2.1 Production flow chart of extruded sample

### 2.5 Experimental Design and Statistical Analysis

Combined design was used to investigate the relationships between the process variables (screw speed, moisture content, and barrel temperature), mixture components (durum clear flour, partially defatted hazelnut flour and fruit waste blend) and response variables used in this study. This design combines process variables, mixture components and categorical factors in one design. When there are constraints on the mixture components and the number of mixture components and process variables increase, the D-optimal criterion is often used for generating a design (Myers and Montgomery, 2002).

A D-optimal design was used for design of experiment with three independent variables including feed moisture content (12-18%), temperature (150-175°C), screw speed (200-280 rpm) and three dependent mixture components having different durum clear flour (8-20%, d.b.), partially defatted hazelnut flour (5-15%, d.b.) and fruit waste contents (3-7%, d.b.) using a commercial statistical package, Design-Expert version 7.0 (Statease, Minneapolis, USA). The levels of each variable were established according to preliminary trials. The quadratic model was used as a design model which included the candidate points of the vertices, the edge centers, the axial check blends, interior check blends and the overall centroid. The three levels of independent variables were coded as -1, 0 and 1. Coded levels for process variables are given in Table 2.1. Totally seventy different combinations were studied using response surface methodology to investigate the effect of these process and component variables on response variables.

Table 2.1 Coded levels for the independent variables

| | Coded Level | | |
|---|---|---|---|
| Variables | -1 | 0 | +1 |
| Barrel Temperature (°C) | 150 | 162.5 | 175 |
| Feed Moisture Content (%) | 12 | 15 | 18 |
| Screw Speed (rpm) | 200 | 240 | 280 |

The outline of the experimental design is presented in Table 2.2. The experimental data were evaluated using response surface methodology. Data were modeled by multiple regression analysis adopting backward stepwise analysis and only the variables significant at $p<0.01$, $p<0.05$ and $p<0.1$ levels were selected for the model construction. The goodness-of-fit of the models was evaluated using the adjusted $r^2$, approximate $r^2$ for prediction values based on PRESS (predicted error sum of squares) statistic and analysis of the residual plots. Statistical significance of the terms in the regression equation was examined by analysis of variance (ANOVA) for each response. A Pearson's correlation matrix on responses was carried out using SPSS 11.0 (SPSS Inc., Chicago, IL, USA) in order to determine correlation coefficients between parameters.

Table 2.2 The outline of the experimental design

| Run | Durum Clear Flour Content (%) | Hazelnut Cake Content (%) | Waste Content (%) | Barrel Temperature (°C) | Feed Moisture Content (%) | Screw Speed (rpm) |
|---|---|---|---|---|---|---|
| 1 | 12 | 15 | 3 | -1 | -1 | +1 |
| 2 | 18 | 5 | 7 | -1 | +1 | +1 |
| 3 | 20 | 5 | 5 | +1 | -1 | -1 |
| 4 | 18 | 5 | 7 | +1 | -1 | +1 |
| 5 | 16 | 11 | 3 | +1 | 0 | -1 |
| 6 | 18 | 5 | 7 | -1 | -1 | 0 |
| 7 | 8 | 15 | 7 | 0 | 0 | +1 |
| 8 | 8 | 15 | 7 | -1 | 0 | -1 |
| 9 | 13 | 10 | 7 | 0 | -1 | 0 |
| 10 | 12 | 15 | 3 | +1 | +1 | -1 |
| 11 | 12 | 15 | 3 | +1 | -1 | +1 |
| 12 | 18 | 5 | 7 | +1 | -1 | -1 |
| 13 | 18 | 5 | 7 | -1 | -1 | -1 |
| 14 | 12 | 15 | 3 | -1 | 0 | -1 |
| 15 | 14.3 | 9.7 | 6 | +1 | +1 | -1 |
| 16 | 8 | 15 | 7 | -1 | -1 | -1 |
| 17 | 8 | 15 | 7 | +1 | 0 | 0 |
| 18 | 20 | 7 | 3 | 0 | -1 | -1 |
| 19 | 20 | 7 | 3 | -1 | -1 | +1 |
| 20 | 16 | 11 | 3 | +1 | -1 | 0 |
| 21 | 20 | 7 | 3 | -1 | +1 | -1 |
| 22 | 20 | 7 | 3 | +1 | +1 | -1 |
| 23 | 14.3 | 9.7 | 6 | -1 | +1 | +1 |
| 24 | 14.3 | 9.7 | 6 | -1 | -1 | +1 |
| 25 | 20 | 7 | 3 | +1 | -1 | -1 |
| 26 | 18 | 5 | 7 | -1 | 0 | -1 |
| 27 | 18 | 5 | 7 | -1 | +1 | -1 |
| 28 | 20 | 5 | 5 | +1 | +1 | -1 |
| 29 | 18 | 5 | 7 | -1 | -1 | +1 |
| 30 | 13 | 10 | 7 | 0 | -1 | -1 |
| 31 | 15.6 | 9.4 | 5 | 0 | 0 | 0 |
| 32 | 20 | 5 | 5 | -1 | -1 | +1 |
| 33 | 14.3 | 9.7 | 6 | +1 | -1 | +1 |
| 34 | 12 | 15 | 3 | -1 | -1 | -1 |
| 35 | 20 | 7 | 3 | -1 | +1 | +1 |
| 36 | 20 | 5 | 5 | -1 | -1 | 0 |
| 37 | 8 | 15 | 7 | +1 | +1 | -1 |
| 38 | 8 | 15 | 7 | +1 | -1 | -1 |
| 39 | 18 | 5 | 7 | 0 | -1 | -1 |
| 40 | 12 | 15 | 3 | -1 | +1 | -1 |
| 41 | 20 | 7 | 3 | 0 | -1 | -1 |
| 42 | 20 | 7 | 3 | +1 | -1 | +1 |
| 43 | 20 | 7 | 3 | -1 | -1 | 0 |
| 44 | 20 | 5 | 5 | 0 | 0 | 0 |
| 45 | 13 | 10 | 7 | -1 | -1 | 0 |

Table 2.2 continued

| | | | | | | |
|---|---|---|---|---|---|---|
| 46 | 8 | 15 | 7 | +1 | -1 | +1 |
| 47 | 12.8 | 12.2 | 5 | +1 | +1 | +1 |
| 48 | 15.6 | 9.4 | 5 | 0 | 0 | 0 |
| 49 | 8 | 15 | 7 | -1 | -1 | +1 |
| 50 | 20 | 5 | 5 | -1 | +1 | +1 |
| 51 | 8 | 15 | 7 | -1 | +1 | +1 |
| 52 | 13 | 10 | 7 | +1 | +1 | +1 |
| 53 | 8 | 15 | 7 | -1 | +1 | -1 |
| 54 | 20 | 7 | 3 | -1 | 0 | -1 |
| 55 | 8 | 15 | 7 | 0 | +1 | 0 |
| 56 | 12 | 15 | 3 | 0 | -1 | -1 |
| 57 | 12 | 15 | 3 | -1 | -1 | 0 |
| 58 | 14.3 | 9.7 | 6 | -1 | +1 | -1 |
| 59 | 12 | 15 | 3 | +1 | -1 | -1 |
| 60 | 20 | 7 | 3 | -1 | -1 | -1 |
| 61 | 20 | 5 | 5 | +1 | -1 | +1 |
| 62 | 8 | 15 | 7 | -1 | -1 | 0 |
| 63 | 18 | 5 | 7 | +1 | +1 | -1 |
| 64 | 20 | 5 | 5 | -1 | 0 | -1 |
| 65 | 13 | 10 | 7 | -1 | 0 | -1 |
| 66 | 12 | 15 | 3 | -1 | +1 | +1 |
| 67 | 20 | 5 | 5 | 0 | -1 | -1 |
| 68 | 18 | 5 | 7 | 0 | -1 | -1 |
| 69 | 8 | 15 | 7 | 0 | -1 | -1 |
| 70 | 12 | 15 | 3 | 0 | -1 | -1 |

## 2.6 Radial Expansion Ratio

Radial expansion of the extruded samples was calculated by dividing cross-sectional area of the extrudates to the cross-sectional area of the die orifice (Thymi et al., 2005). Caliper was used to measure the diameters of extrudates. Ten samples were used for each extrudate to calculate the average.

## 2.7 Bulk Density

The bulk density was calculated by measuring the actual dimensions of the extrudates (Thymi et al., 2005; Asare et al., 2004). The diameter and length of the extrudates were measured using caliper. The weight per unit length of extrudate was determined by weighing measured lengths (about 1 cm). The bulk density was then calculated using the following formula, assuming a cylindrical shape of extrudate.

$$\rho_b = 4 / \pi d^2 l \tag{2.1}$$

where $\rho_b$ is bulk density (g.cm$^{-3}$), $d$ is diameter of the extrudate (cm), and $l$ is the length per gram of the extrudate (cm.g$^{-1}$). Five pieces of extrudate were randomly selected and average was taken.

## 2.8 Apparent Density

The extrudates were milled and sieved through 500 µm sieve. A 5 ml graduated measuring cylinder was tared and gently filled with extrudate. The bottom of the cylinder was repeatedly tapped gently until there was no further reduction of sample volume and it was weighed (Onyango et al., 2004). The apparent density ( $\rho_s$ ) of the extruded samples was calculated as mass per unit volume (g.cm$^{-3}$). Three measurements were performed to calculate the average.

## 2.9 Porosity

The porosity of extrudates was determined from the bulk and apparent volumes. Porosity was calculated using the equation:

$$Porosity = \frac{Bulk\ Volume - Apparent\ Volume}{Bulk\ Volume} \tag{2.2}$$

where $Bulk\ Volume = 1/\rho_b$ (2.3)

and $Apparent\ Volume = 1/\rho_s$ (2.4)

## 2.10 Water Absorption and Solubility Indices

Water solubility (WSI) and water absorption (WAI) indices of extruded products were determined by a modification of the method of Anderson et al. (1969). The extrudate samples were ground and sieved through 500 µm sieve. Distilled water (10 ml) at 25$^{o}$C was placed in a tared centrifuge tube and 0.5 g of extrudate was dispersed in the water. Care was taken to avoid lumping in order to produce smooth dispersion. After standing for 30 min (with intermittent shaking every 5 min), the sample was centrifuged (Roto Silenta II, Hettich, Tuttlingen, Germany) at 1800×*g* for 15 min. The supernatant was decanted into a tared aluminum pan and dried at 105°C until constant weight. The weight of the gel remaining in the centrifuge tube was noted. The results were expressed as the average of two measurements.

$$WAI(g/g) = \frac{Weight\ Gain\ of\ Gel}{Dry\ Weight\ of\ Extrudate} \tag{2.5}$$

$$WSI(\%) = \frac{Weight\ of\ Dry\ Solids\ in\ Supernatant}{Dry\ Weight\ of\ Extrudate} \times 100 \tag{2.6}$$

### 2.11 Color

The color measurement was done using HunterLab ColorFlex (A60-1010-615 Model Colorimeter, Hunter lab, Reston VA). The extrudate was milled with a laboratory mill. The color values were expressed as L (lightness or darkness), a (redness or greenness), b (yellowness or blueness). The colorimeter was calibrated against a standard white plate (L = 91.08, a = -1.12, b = 1.25). Three readings were averaged.

### 2.12 Textural Properties

The textural characteristics of extrudate were measured according to method of Veillard et al. (2003) with a TA-XT2i Texture Analyzer (Texture Technologies Corp, scarsdale, NY/Stable Micro Systems, Godalming, Survey, UK) and the software texture Expert (version 2.03). The instrument was fitted to the standard 25 kg load cell supplied with the texture analyzer. A rectangular probe (38 mm width and 51 mm length) attached to the arm of analyzer was used to compress the sample at a constant speed of 1 mm/s against the flat plate fixed on the loading frame. The sample was compressed to 70% of the sample's original height, and the force-deformation displacement was recorded. The trigger force of the analyzer was 5 g. All sample sizes were 10 mm in length. Five replicates were conducted for samples from each treatment. Texture parameters such as hardness and area (energy required for given displacement) were calculated from the force-deformation curve. The peak force represents the resistance of extrudate to initial penetration and is believed to be hardness of extrudate; whereas the area under the curve is used to determine the energy required to bite or chew the products (Ding et al., 2005a; Ding et al., 2005b).

### 2.13 Sensory Characteristics

A preliminary product selection was made in order to reduce the number of samples to be submitted to the panel. First of all, extrudates having maximum expansion ratio were selected from each product formulation. Then, the products were selected according to fruit waste and PDHF content so that extrudates with minimal and maximal PDHF content at each fruit waste percentage were selected. In total six samples were used to perform sensory evaluation. Nine point hedonic scales were adopted and the categories were rated from 1(absent/dislike extremely) to 9 (very high/like extremely) in order to evaluate the extrudate characteristics which are bitterness (absent to very high), off-flavor (absent to very high), orange and hazelnut flavor (absent to very high), color (dislike extremely to like extremely), air cell homogeneity (dislike extremely to like extremely), texture (hardness, crispness and breakability) (dislike extremely to like extremely) and overall acceptability (dislike extremely to like extremely) (Appendix 1). A test panel consisted of 46 semi-trained panelists (20-45 year old males and females) who are students and staff members of the Department of Food Engineering at Gaziantep University. Panelists were selected in preliminary sessions and experienced with the products and terminology (Appendix 1). Duncan's multiple range test was used to differentiate the sensory data.

## 2.14 Total Phenolic Content

Ground extrudates and raw materials including rice grit, partially defatted hazelnut flour, durum clear flour and fruit waste blend were analysed for total phenolics with Folin-Ciocalteau method which based on colorimetric oxidation/reduction reaction. Procedures were adapted from Gao et al. (2002) and Beta et al. (2005). Samples were ground to pass through a 355 μm screen and samples remaining under the screen were used in analyses. Two hundred milligrams were extracted with 4 ml acidified methanol (HCl/methanol/water, 1:80:10, v/v) at room temperature for 2 h on a magnetic mixer. The mixture was centrifuged (Roto Silenta II, Hettich, Tuttlingen, Germany) at 1000×*g* rpm for 10 min. A 0.2 ml of supernatant was added to 1.5 ml of freshly diluted 10-fold Folin-Ciocalteau reagent (Merck, Darmstadt, Germany). The mixture was allowed to equilibrate for 5 min and was then mixed with 1.5 ml of sodium carbonate solution (60 g/L). After incubation at room temperature for 90 min, the absorbance of the mixture at 725 nm was read on UV/VIS Lambda 25 Spectrometer (Perkin Elmer, Shelton, USA). Acidified methanol was used as the blank. Ferulic acid (Merck, Hohenbrunn, Germany) was used as the standard. A standard curve was prepared using solutions containing concentrations of ferulic acid (50-500 mg/ml; $r^2$=0.998). The results were expressed as ferulic acid (mg/g dry sample) equivalents. All tests were duplicated.

## 2.15 Antioxidant Activity

The scavenging activity on 2,2-diphenyl-1-picrylhydrazyl (DPPH) (Sigma, Steinhein, Germany) radical of all extracts of extrudates and raw materials including rice grit, partially defatted hazelnut flour, durum clear flour and fruit waste blend, was determined by modifying the methods of Brand-Williams et al. (1995) and Beta et al. (2005). A 1 g of ground and sieved (through 355 μm screen) sample was extracted with 10 ml of methanol at room temperature for 2 h on a magnetic mixer. Sample was centrifuged (Roto Silenta II, Hettich, Tuttlingen, Germany) at 1000×*g* for 10 min. A 0.1 ml of extract was reacted with 3.9 ml of a $6 \times 10^{-5}$ mol/L of DPPH solution (1.2 mg of DPPH in 50 ml of methanol). The mixture was left to stand for 30 min in the dark at room temperature. The mixture of methanol and DPPH solution was used as the control. Absorbance ($A$) at 515 nm was determined using UV/VIS Lambda 25 Spectrometer (Perkin Elmer, Shelton, USA) at 0 and 30 min. Methanol was the blank. Antioxidant activity was calculated as % of inhibition from the following equation.

$$\%\ Inhibition = \left[ 1 - \frac{Absorbance\ of\ sample_{t=30\ \mathrm{min}}}{Absorbance\ of\ control_{t=0\ \mathrm{min}}} \right] \times 100 \qquad (2.7)$$

## 2.16 Differential Scanning Calorimetry (DSC)

DSC analyses were conducted on raw materials including durum clear flour, partially defatted hazelnut flour and rice grit, selected unextruded blend and extrudate to study extent of starch gelatinization in these samples. A Perkin-Elmer Pyris 6 DSC differential scanning calorimeter (Perkin Elmer, Netherland) equipped with Perkin-Elmer intracooler was used to analyze the samples. The DSC was first calibrated high purity indium metal as a standard and purged with nitrogen gas at 40 $ml.min^{-1}$. About 4 mg (d.b.) of ground and sieved (through 355 μm screen) sample was placed in 70 μl aluminum pan and distilled water was added to obtain water to dry sample ratio of 3:1 (w/w). Sealed pans were kept at 4°C for 24 h in order to ensure homogenous hydration. For each run, an empty sample pan was used as reference. The slow heating rate of 5°C $min^{-1}$ was used to minimize any temperature lag due to the large mass of the steel pan. The pans were heated from 5 to 150°C at a constant rate of 5°C $min^{-1}$, hold for 1 min at 150°C and cooled to 5°C at a constant rate of 5°C $min^{-1}$. The onset, peak and end temperatures were recorded and enthalpy of gelatinization required to melt starch crystallite was measured from area of endothermic peak in DSC thermogram and expressed as $J.g^{-1}$ sample on dry basis.

## 2.17 Polarized Light Microscopy

Selected extrudates and raw materials including rice grit and durum clear flour were observed using a polarized light microscope. About 2 mg of ground sample (passing through 53 µm mesh sieve) were placed in slides covered by a droplet of glycerol/water (20:20) solution with a cover slip and immediately observed at a magnification of 10X under a polarized light microscope (Model BX51, Olympus Corp., Tokyo, Japan) equipped with a 100-W halogen light source. The microscope images were acquired with a Pixera camera (Model PVC 100C, Los Gatos, CA, USA). Each experiment was replicated two times.

## 2.18 Percent Starch Gelatinization

The percent starch gelatinization in each extruded product was determined by a modified method of Wootton and Chaudhry (1980), which is based on the formation of a blue iodine complex by amylose released during gelatinization. Ground sample (sample amount referring to 2 g of dry sample) was macerated with water (100 ml) at room temperature for 3 min on a magnetic mixer. The suspension was then centrifuged at 1500×*g* for 10 min (Roto Silenta II, Hettich, Tuttlingen, Germany). Triplicate aliquots of 1 ml were mixed with 1 ml iodine solution (4% KI, 1% $I_2$) and the mixture was diluted to 10 ml with distilled water. Absorbance ($A_1$) at 600 nm was then measured against blank solution containing all reagents except the extruded sample using UV/VIS Lambda 25 Spectrometer (Perkin Elmer, Shelton, USA). In a separate analysis, sample amount referring to 2 g of dry unextruded blend of each extrudate was mixed with 100 ml of distilled water and the suspension was boiled for 5 min with continuous stirring and then cooled. This prepared suspension was subjected to the same procedure as above to measure the intensity of the blue color formed spectrophotometrically ($A_2$). Percent starch gelatinization in extruded samples was calculated using equation (2.8) by assuming the starch in boiled unextruded blend sample was 100% gelatinized (Ibanoglu et al., 1996).

$$\%\ Starch\ Gelatinization = \left[\frac{A_1}{A_2}\right] \times 100 \qquad (2.8)$$

## 2.19 Starch Digestibility

In vitro digestibility of starch in the extruded sample and raw materials was estimated according to the method of Onyango et al. (2004) using α-amylase (from porcine pancreas; Sigma-Aldrich Co. A-6255; activity 1122 units/mg protein). 5 mg sample was dissolved in 1 ml of 0.2 M phosphate buffer (pH 6.9). A 0.5 ml of α-amylase suspension (0.4 $mg.ml^{-1}$ of 0.2 M phosphate buffer, pH 6.9) was added to the sample suspension and incubated at 37°C for 2 h. At the end of incubation period, 1 ml of 3,5-dinitrosalicylic acid reagent was quickly added and the mixture heated for 5 min in a boiling water bath. After cooling, the solution was made to 25 ml with distilled water and filtered prior to measurement of absorbance at 510 nm using UV/VIS Lambda 25 Spectrometer (Perkin Elmer, Shelton, USA). A blank for each sample was prepared by incubating the sample first and 3,5-dinitrosalicylic acid reagent was added before addition of the enzyme solution. A standard curve was prepared using solutions containing concentrations of maltose monohydrate in the range of 0.01-0.1 $mg.ml^{-1}$. The amount of starch digestion was determined by comparing the absorbance of each sample to a standard maltose calibration curve ($r^2$=0.987). The values were expressed as mg maltose/g dry sample for duplicate measurements.

# CHAPTER 3

# RESULTS AND DISCUSSIONS

## 3.1 Physical Properties of Extruded Foods

In this study, ready to-eat, functional, puffed extruded foods were produced by extrusion method. For this purpose fruit wastes including orange peel, grape seed and tomato pomace which have many nutritional and functional properties (Braddock 1999; Bocco et al., 1998; Jayaprakasha et al., 2001; Schieber et al., 2001) were combined with the valuable side products, durum clear flour, PDHF, orange peel, grape seed and tomato pomace. The proximate compositions of the raw materials used in the formulations of extruded products are shown in Table 3.1.

Table 3.1 Proximate compositions (g/100 g) of durum clear flour, PDHF, fruit waste blend and rice grit (results are average of three replicates ± standard deviation)

| Component (%) | Durum Clear Flour | PDHF | Fruit Waste Blend | Rice Grit |
|---|---|---|---|---|
| Moisture | 7.39 ± 0.08 | 2.90 ± 0.06 | 8.27± 0.08 | 11.45 ± 0.04 |
| Fat | 2.93 ± 0.05 | 17.26 ± 0.06 | 2.01± 0.02 | 0.79 ± 0.04 |
| Protein | 13.38 ± 0.01 | 33.51 ± 0.03 | 4.99±0.05 | 5.92 ± 0.03 |
| Ash | 1.93 ± 0.04 | 6.61 ± 0.02 | 3.64±0.03 | 0.41 ± 0.02 |
| Carbohydrate* | 74.37 | 39.72 | 81.09 | 81.43 |

*determined by difference

The results obtained from preliminary trials confirmed that durum clear flour and PDHF are suitable for extrusion cooking process only if properly blended with other cereal flours due to their smaller size and relatively high protein (Table 3.1), which has limiting effects on both gelatinization and expansion. Rice grit was used as a principal cereal and the percentage of rice grit in blends was kept as 70%. Preliminary trials revealed that composition of durum clear flour, PDHF and fruit waste blend higher than 30% in whole blend causing irregular flow in the extruder barrel and gave poor expansions in the final extruded product.

Physical properties and expansion characteristics of extruded products has an important role in the acceptability of the final product. Extrudate of widely different physical structure were obtained in the experiments with various formulations of the blends. Extrusion of blends of durum clear flour, PDHF and fruit waste in combination with rice grit gave acceptable extruded products in terms of physical properties. Extrusion of most combinations of durum clear flour, PDHF and fruit waste produced expanded extrudates at most extrusion conditions; however blends having 15% PDHF usually failed to extrudate into products with consistent shapes and degrees of expansion.

In this study, response surface methodology was used to model physical properties of extruded foods with respect to component and process variables. Tables A.1 and A.2 show the coefficients of equations obtained by fitting of experimental data. The coefficient of determinations of regression equations changed from 0.583 to 0.862 with significant probability values ($p<0.0001$) and non-significant lack of fit values (Table A.1 and Table A.2). These models could be adequately used as predictor models, regardless of low coefficient of determinations. Only coefficients making a significant contribution to the model are included in the model. Furthermore, non-significant lack of fit in the models makes them as predictive models such that the lack of fit error significantly larger than the pure error indicates something remain in the residuals can be removed by an appropriate model. If a model has a significant lack of fit, it is not a good indicator of the response and should not be used for prediction (Myers and Montgomery, 2002). Hence, it can be concluded that the proposed models approximate the response surfaces and can be used suitably for prediction at any values of the parameters within experimental range.

### 3.1.1 Radial Expansion Ratio

The expansion ratio of the extrudates seeks to describe the degree of puffing undergone by the dough as it exits the extruder. The stored energy was released in the expansion process, increasing the radial expansion ratio (Thymi et al., 2005). In this study, response surface methodology was used to model physical properties of extruded foods with respect to component and process variables. Radial expansion ratio of extrudates varied in complex manner and depended on extrusion process parameters and composition of the initial blends. The coefficients of multiple regression model for predicting the expansion ratio is given in Table A.1. The analytical results indicated the model were acceptable ($p<0.0001$; non-significant lack of fit) and could be used to predict values for radial expansion ratio. The radial expansion ratio was significantly affected ($p<0.001$) by linear effects of PDHF, durum clear flour, waste contents. Durum clear flour and waste content had significant effect and positive correlation with radial expansion ratio. On the other hand, radial expansion ratio was negatively affected by the linear coefficient of PDHF. The effect of PDHF content was stronger than that of both durum clear flour and fruit waste contents (also evident by comparing the coefficients of $x_1$, $x_2$ and $x_3$ in Table A.1).

The radial expansion ratios measured for all the extruded samples ranged between 1.24±0.24 and 10.05±0.17. Expansion ratios of extruded samples were similar to published values of rice-based extrudates (Ilo et al., 1999; Asare et al., 2004; Ding et al., 2005a). Response surface plot for the radial expansion ratio as a function of components are shown in Figure 3.1. As presented, when PDHF content increased, the radial expansion ratio decreased. The steady decrease in expansion may be due to dilution of total starch available for expansion with addition of PDHF. The addition of PDHF may affect the extent of starch gelatinization, and the rheological properties of the melted material in the extruder due to its relatively high protein and fiber content. Prinyawiwatkul et al. (1995) presented that addition of protein to a starchy extrusion system may retard expansion by increased firmness of plasticized extrudates. Badrie and Mellowes (1992) reported a negative correlation of expansion with protein and fiber content. The nonstarch polysaccharides in fiber may bind water more tightly during extrusion than do protein and starch. This binding may inhibit water loss at the die and thus reduce expansion (Camire and King, 1991). Bahattacharya (1997) reported that extrudates produced from rice-green gram blends have lower expansion ratios than the rice alone. Similar finding was observed for the rice-cowpea-groundnut blend, cowpea and groundnut additions to the rice resulted in decrease the expansion ratio (Asare et al., 2004). Expansion ratio of extrudates may also be reduced by the addition of lipid in PDHF to the blend at increased PDHF content. Addition of lipid in extrusion is generally found to retard the degree of gelatinization and affect dough rheology in the barrel (Schweizer et al., 1986).

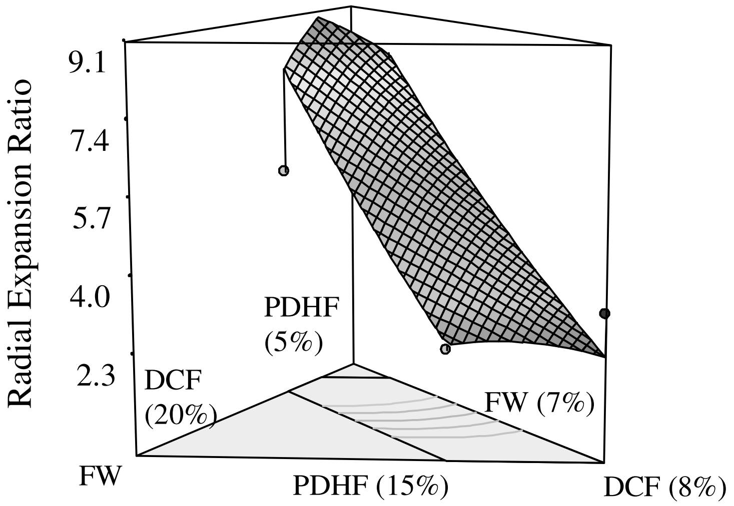


Figure 3.1 Response surface plot for radial expansion ratio as a function of components at 12% moisture content, 200 rpm screw speed and 150°C barrel temperature (DCF, durum clear flour; PDHF, partially defatted hazelnut flour; FW, fruit waste)

Volumetric expansion of extruded starch based materials is consequence of extensive flash-off of internal moisture and flow properties of molten mass. The latter depends on the degree of gelatinization, which is determined by processing conditions and raw material composition (Jin et al., 1994; Mendonça et al., 2000). Moisture content had significant interaction ($p$<0.05) with durum clear flour, PDHF and waste content (Table A.1). Increasing moisture content from 12 to 18% caused an increase in expansion ratio for most compositions. Similar trend was observed for sorghum extrudates, increasing moisture content from 13 to 18% increased radial expansion (Falcone and Phillips 1988). The effect of moisture content is mostly depending on composition. At high PDHF content, such as 12.5%, increasing moisture content increases expansion of extrudates, while at low PDHF content, such as 5%, elevation of moisture content had no effect on expansion (Figure 3.2). The moisture and PDHF interaction showed positive interaction which may suggest that increasing the amount of PDHF added in the blend required a corresponding increase in moisture content in order to achieve same level of radial expansion ratio. The elevated moisture requirement was probably due to increase in protein content, which needs more moisture to hydrate (Li et al., 2005).

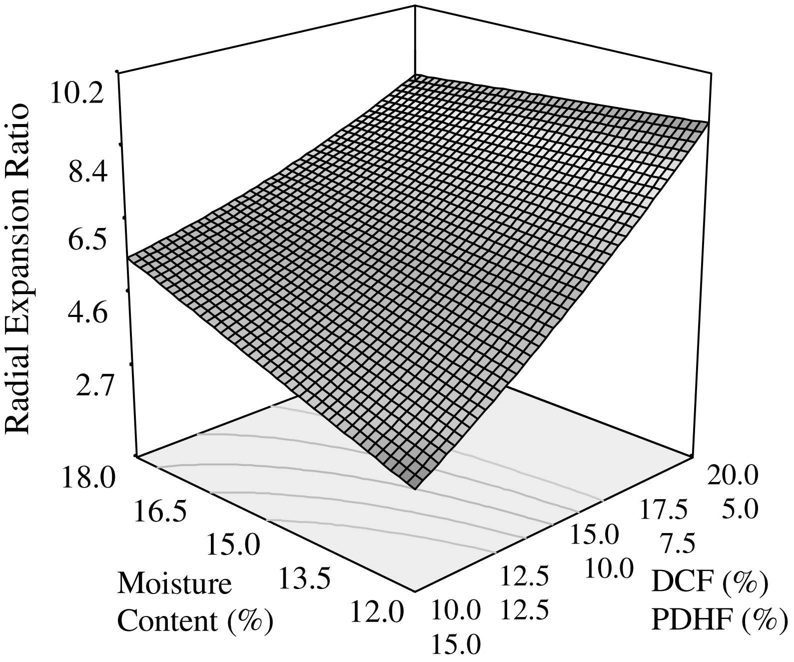


Figure 3.2 Response surface plots for radial expansion ratio as a function of moisture content & durum clear flour & PDHF at 5% fruit waste content, 200 rpm screw speed and 150°C barrel temperature (DCF, durum clear flour; PDHF, partially defatted hazelnut flour)

The interactions between screw speed and durum clear flour, PDHF and waste content did not show significant influences on expansion at $p<0.05$ level. Temperature affected expansion with significant interaction ($p<0.05$) with PDHF. Temperature generally had negative effect on expansion for most compositions (Figure 3.3). Expansion decrease at higher extruder temperatures can be attributed to increased dextrinization and weakening of structure (Mendonça et al., 2000).

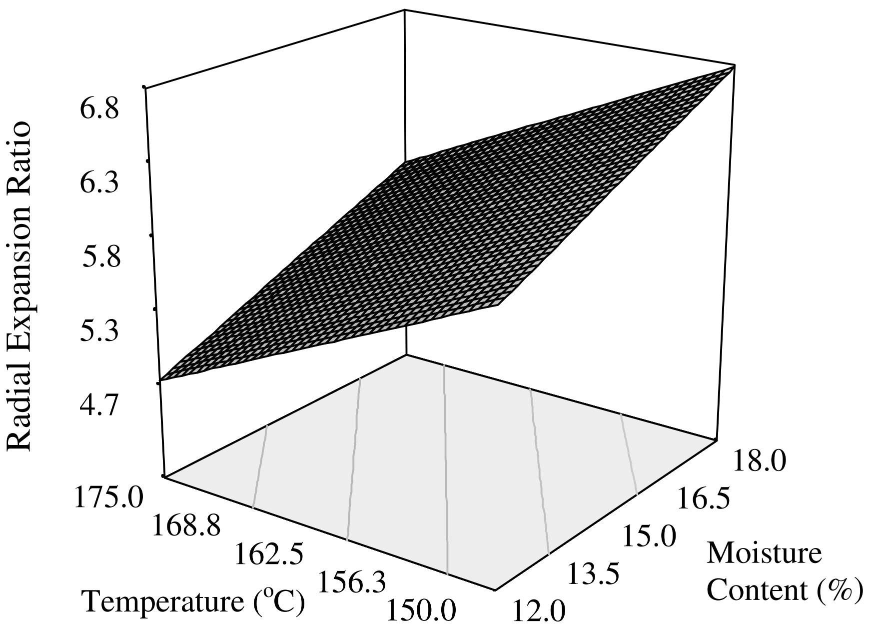


Figure 3.3 Response surface plots for radial expansion ratio as a function of moisture content & temperature at 15.6% durum clear flour, 9.4% PDHF, 5% fruit waste contents and 200 rpm screw speed

### 3.1.2 Bulk Density

Bulk density has been linked with the expansion ratio in describing the degree of puffing in extrudates (Asare et al., 2004). The bulk density of the extrudates varied between 0.117±0.007 and 0.254±0.061 g.cm$^{-3}$. Table A.1 shows the coefficients of equation which is obtained by fitting of the response data employing a backward elimination procedure. The multiple regression model for predicting the bulk density could explain 80.2% of the observed variations. The component variables and linear interaction between component variables and process variables showed significant influence ($p<0.01$ and $p<0.05$) on bulk density of the extrudates. The quadratic effect of feed moisture content which had interaction with durum clear flour and PDHF was also found to significantly influence the bulk density. Durum clear flour and PDHF content had significant effect and positive correlation with bulk density. However, it was negatively affected by the linear coefficient of fruit waste content.

The effect of component variables on the bulk density of extrudates is presented in Figure 3.4. The bulk density was found to be the most dependent on PDHF content rather than fruit waste content. Higher PDHF and lower fruit waste addition resulted higher bulk density in the studied experimental range. The steady increase in bulk density with increasing PDHF could be due to the addition of increasing amounts of protein to the blend which may affect the extent of starch gelatinization and thus the rheological properties of the melted material in the extruder. An increased bulk density with addition of PDHF agrees with the results of Hsieh et al. (1989). In their study, they found that increasing fiber content in corn meals had positive effect for the axial expansion and the negative effect for radial expansion, and so the net result was increase in the bulk density of both wheat fiber and oat fiber- containing corn meal extrudates. Similar to our study, Falcone and Phillips (1988) obtained more dense extrudates through the extrusion of cowpea-starch blends rather than the extrusion of starch alone. Remarkable observations have been reported by Suknark et al. (1997) who studied the physical properties of extrudates formulated from the partially defatted peanut flour (PDPF) and different starches. They reported that the bulk density first decreased when 15-30% PDPF was substituted and then increased when more PDPF was substituted to the starches. Bahattacharya and Hanna (1988) reported that higher lipid concentration also decreased the puff ratio of product and hence it gave a denser extrudate.

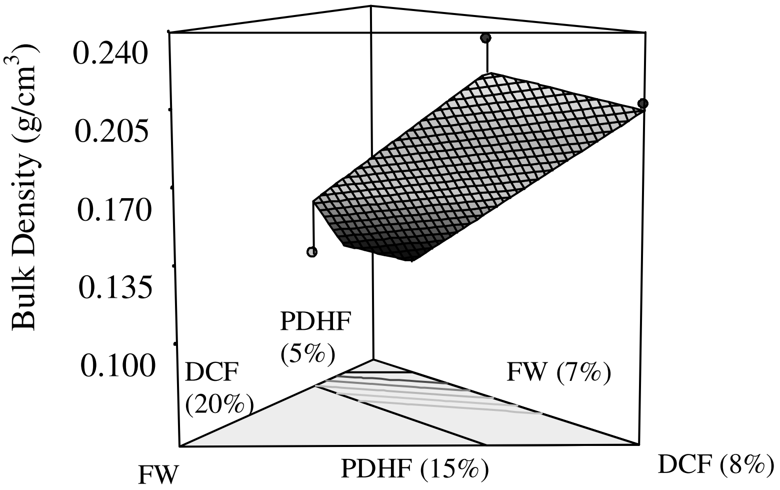


Figure 3.4 Response surface plots for bulk density as a function of components at 12% moisture content, 200 rpm screw speed and 150°C barrel temperature (DCF, durum clear flour; PDHF, partially defatted hazelnut flour; FW, fruit waste)

The bulk density of extrudates slightly decreased due to the addition of fruit waste. Fruit waste was mainly composed of orange pulp in which pectin is predominantly found (Larrea et al., 2005b). Yanniotis et al. (2007) observed increasing porosity of corn starch extrudates with addition of the pectin. They concluded that pectin reduces the fracture of cell walls by increasing their extensibility and so the porosity increases slightly with pectin content.

Extrudate density has been reported to inversely relate with the overall expansion (Ilo et al., 1999). Correlation of experimental data of radial expansion with that of bulk density suggested that there was no significant correlation at $p<0.05$ (Table A.3) with the bulk density and radial expansion ratio. This result seems to be inconsistent. Although expansion generally has been found to inversely relate with the bulk density of the extrudates (Suknark et al., 1997; Rayas-Duarte et al., 1998), it was noted that density is more highly correlated with longitudinal expansion rather than the radial expansion (Mendonça et al., 2000). Falcone and Phillips (1988) explained that expansion ratio considers expansion only in the direction perpendicular to extrudate flow, while unit bulk density considers expansion in all directions. They observed poor correlation ($r^2=0.4$) between the radial expansion ratio and bulk density data as observed in this study. Similarly, Hsieh et al. (1989) and observed increase in bulk density and axial expansion in wheat fiber and oat fiber extrudates with increasing fiber content.

The influence of feed moisture content on the bulk density was significantly dependent ($p<0.05$) on the composition of the extrudates. The quadratic term of feed moisture content in the surface model of bulk density was significant ($p<0.05$, Table A.1). The bulk density of the extrudate increased with increasing feed moisture at high levels of fruit waste and low levels of PDHF additions (Figure 3.5A). On the other hand, bulk density was lower for extrudates with low levels of fruit waste (3%) and high levels of PDHF percentages (14%) at intermediate feed moisture content.

Temperature significantly affected the bulk density in interaction with durum clear flour and PDHF contents ($p<0.05$) and also triple interaction with component and process variables ($p<0.05$, Table A.1). At high temperature, the bulk densities of extrudates slightly increased for most of the extrudate formulations except for the extrudates having high PDHF and low fruit waste contents (Figure 3.5B). The temperature and PDHF relation showed negative interaction ($p<0.01$) which indicates increasing temperature caused reduced values of bulk density at the high percentage of PDHF (Table A.1).

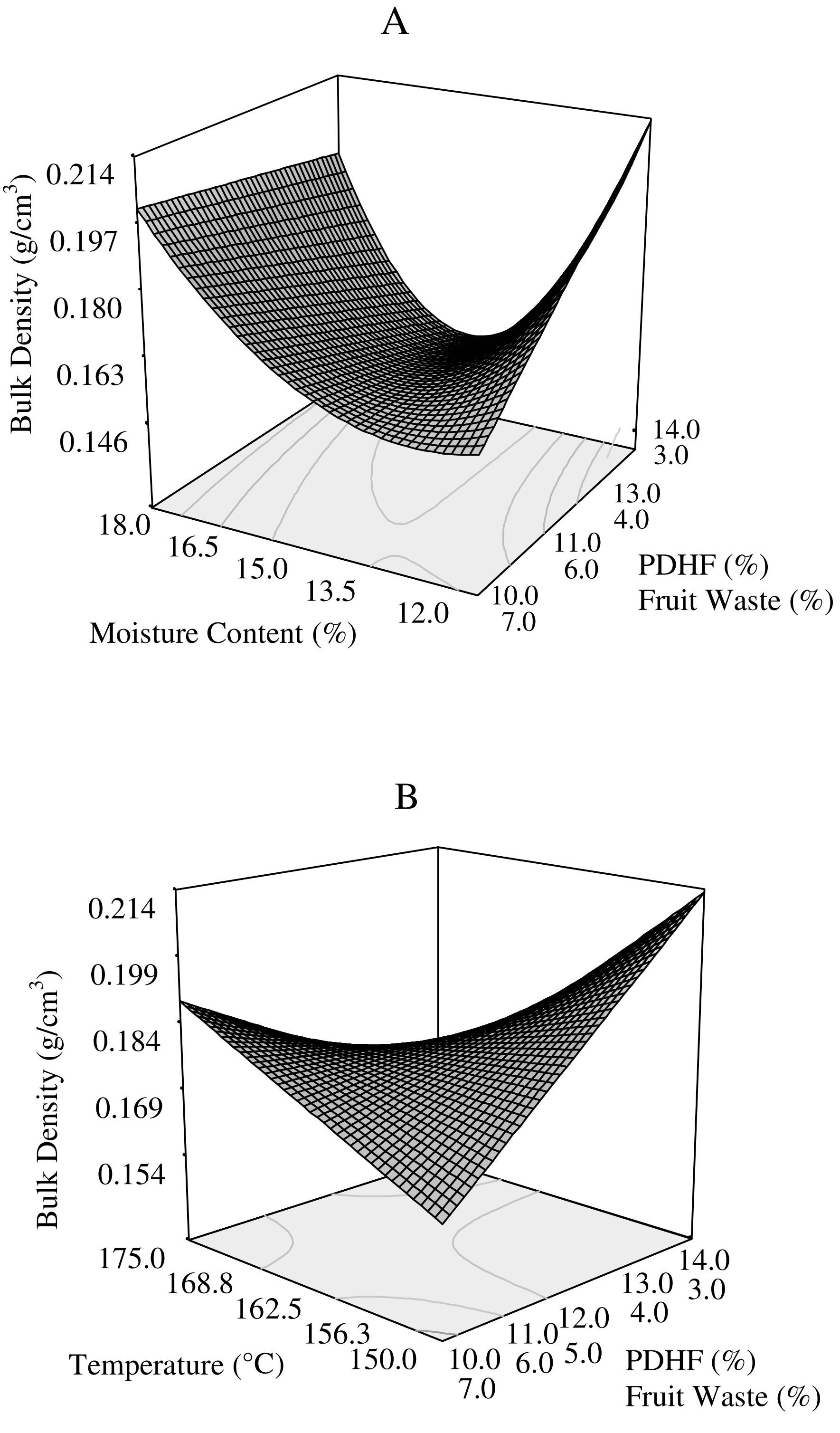


Figure 3.5 Response surface plots for bulk density. A: Moisture Content & PDHF & Fruit Waste Content at 150°C barrel temperature and B: Temperature & PDHF & Fruit Waste Content at 12% moisture content (The extrusion variables studied at 13% durum clear flour content and 200 rpm screw speed)

The reducing effect of temperature on bulk density may be due to combined effect of higher temperature and relatively higher fiber content of the blend, which probably increase the pressure inside the extruder barrel and viscosity of the melt, respectively. Increased pressure at high shear environment was able to puff the extrudates, thus likely reduce the bulk density of extrudates. Temperature determines the vapor pressure of the moisture and thus degree of puffing. Higher temperature lowered the viscosity of the dough mass in the extruder and hence resulted in higher linear velocity at the die. On the other hand, presence of insoluble fiber also reduces the elasticity and plasticity of dough (Hsieh et al., 1989). In this study, temperature increase leads to lower porosity and expansion ratio values, resulting higher bulk densities for most formulations. Similar results were observed for the fiber-added cornmeal extrudate (Mendonça et al., 2000). Bahattacharya (1997) stated decrease in density of rice-green gram extrudates in temperature range of 100-140°C, however above 140°C, the density generally stayed constant.

The effects of temperature and screw speed were found to be dependent on each other. Bulk density stayed fairly constant at low screw speed values, whereas decreased as the temperature increased at high screw speeds (Figure 3.6). This was probably due to intensified effect of temperature on extrudate melt under increased shear environment (high screw speed) which may increase the extent of gelatinization process and so it gave lighter extrudate. Increasing screw speed increased the bulk density at most of the extrudate composition (Figure 3.6). The results agree with the results in literature for different type of the extruded products (Onyango et al., 2004; Li et al., 2005). A low screw speed is associated with high residence time; consequently the food material inside the extruder receives more input of thermal energy in a low shear environment. High input of thermal energy due to high residence time (at low screw speeds) leads to the creation of enhanced level of superheated steam, hence the product will have good expansion which creates flaky and porous structures due to formation of air cells (Bahattacharya, 1997) and hence the products become lighter.

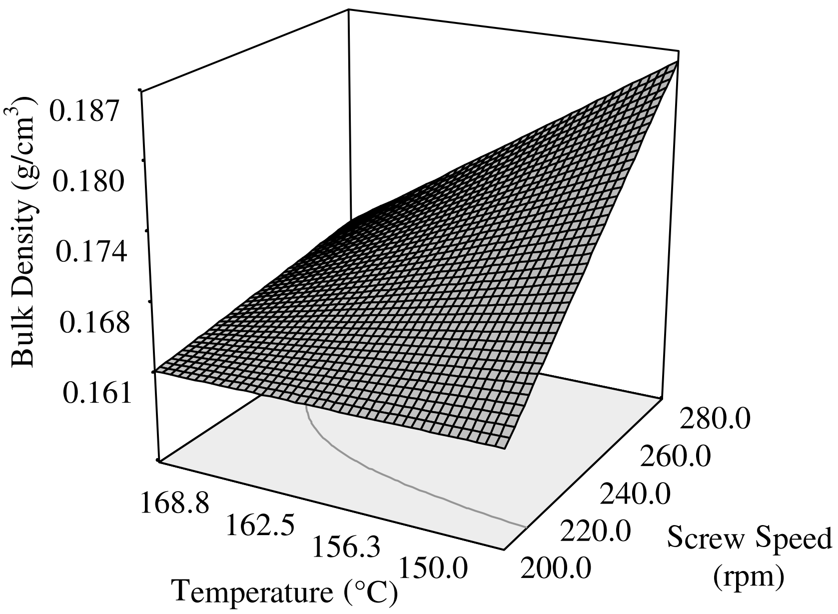


Figure 3.6 Response surface plots for bulk density as a function of temperature and screw speed at 12% moisture content (The extrusion variables studied at 15.6% durum clear flour, 9.4% PDHF and 5% fruit waste contents)

### 3.1.3 Porosity

Air cells were created during extrusion giving expanded products with variable pore size and the number. A porous, expanded, sponge-like structure is formed inside extrudates due to many tiny steam bubbles produced by the rapid release of pressure after exiting the die (Suknark et al., 1997). Porosity created during extrusion can be used to describe the expansion properties of the extruded product (Thymi et al., 2005; Yanniotis et al., 2007). The parameters of regression equations obtained by fitting of porosity data are given in Table A.1.

The porosity decreased considerably as the PDHF content increased, whereas it slightly increased with increasing content of fruit waste (Figure 3.7), showing the opposite effect than the bulk density. Decrease in porosity may be due to dilution of total starch available for expansion with addition of PDHF. A high bulk density is associated with a low expansion index (Suknark et al., 1997; Rayas-Duarte et al., 1998) because more compact material is obtained after milling a less expanded product (Onyango et al., 2004). Statistical analysis revealed that bulk density was negatively correlated with porosity which is indication of expanded structure (r=-0.692, *p*<0.01) (Table A.3). This result is in agreement also with the works of other researchers (Camire and King, 1991; Suknark et al., 1997; Rayas-Duarte et al., 1998; Ilo et al., 1999). The preferred higher porosity values occurred nearly in the same regions as the low predicted values for bulk density and high predicted values for radial expansion ratio.

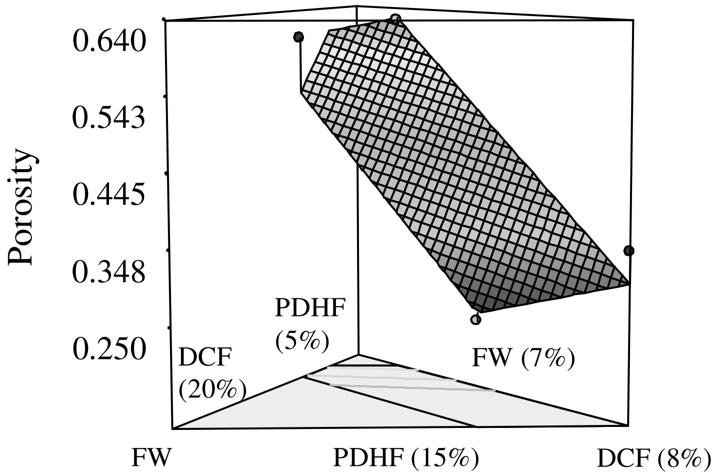


Figure 3.7 Response surface plot for porosity as a function of components at 12% moisture content, 200 rpm screw speed and 150°C barrel temperature (DCF, durum clear flour; PDHF, partially defatted hazelnut flour; FW, fruit waste)

The effect of processing conditions on porosity showed similar behavior as in radial expansion ratio. Increasing feed moisture content from 12 to 18% caused an increase in porosity values for most compositions, while temperature increase seems to have the opposite effect, resulted a considerable porosity decrease (Figure 3.8). Falcone and Phillips (1988) found that increasing moisture content from 13 to 18% increased expansion of sorghum extrudates but further increase caused a decrease in expansion. Porosity decrease at higher extruder temperatures can be attributed to increased dextrinization and weakening of structure and was observed for temperatures higher than 150°C (Mendonça et al., 2000), which was used in this study. The interactions between screw speed and durum clear flour, PDHF and waste content did not show significant influences on porosity at *p*<0.05 level.

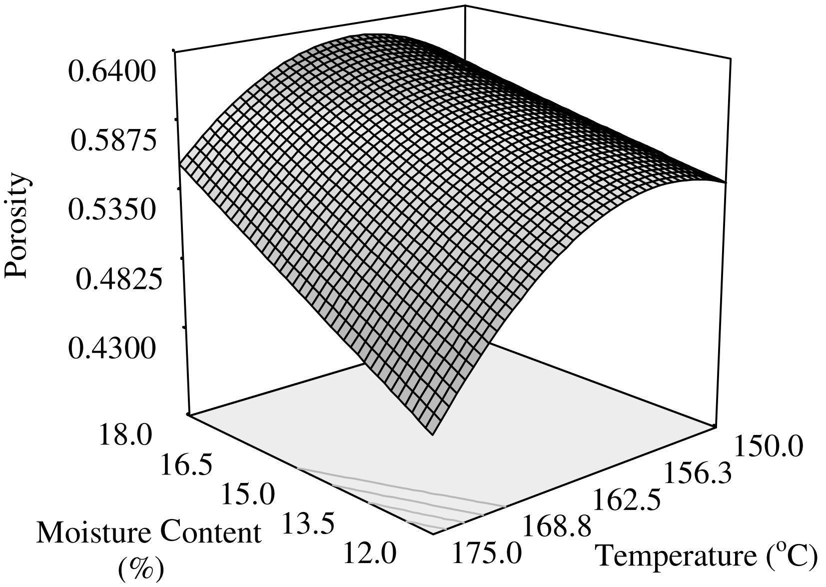


Figure 3.8 Response surface plot for porosity as a function of moisture content and temperature at 200 rpm screw speed (The extrusion variables studied at 15.6% durum clear flour, 9.4% PDHF and 5% fruit waste contents)

### 3.1.4 Water Absorption and Water Solubility Indices

Water absorption has been generally attributed to the dispersion of starch in excess water, and the dispersion increases 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). Water solubility index, 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., 2005b). The values of the WAI and WSI of extrudates as a function of component variables are shown in Figure 3.9. The values for WAI ranged between 3.65± 0.06 and 5.59±0.05 g water/g dry sample and the WSI varied from 15.73±1.11 to 37.19±0.38 g/g dry sample. The general regression equations for water absorption and water solubility indices are reported in Table A.1. Both indices showed first order relation with component variables used in this study. Durum clear flour, PDHF and waste content had significant effect ($p<0.01$) and positive correlation with WAI and WSI.

WAI increased considerably as the percentage of PDHF decreased, while WSI decreased. Increase in the value of WAI may probably be caused by uncovering of hydrophilic groups in extrudates and greater availability and easier penetration of structures by water molecules (Colonna et al., 1989; Rayas-Duarte et al., 1998). Increasing the PDHF content decreased the WAI suggesting that PDHF decreased the starch molecular degradation. Relative decrease in starch content with addition of PDHF may affect the extent of starch gelatinization in barrel and caused reduced water absorption. Singh et al. (2007b) reported similar trend in WAI with addition of pea grits in extrusion of rice was due to the dilution of starch in rice pea blends. In similar to our study, water absorption decreased with increasing lipid content (Bahattacharya and Hanna,1988) and addition of flaxseed (Ahmed,1999).

Comparison of the radial expansion ratio and porosity change plots to the corresponding water absorption plots suggested that the higher values of porosity were associated with high water absorption. WAI was positively correlated with porosity of extrudates (r=0.677, $p<0.01$) whereas it negatively correlated with bulk density (r=-0.252, $p<0.05$) (Table A.3). The amount of water absorbed by the ground extrudate has been used as indirect estimation of the porosity of the material (Colonna et al., 1989). As the porosity of the extrudate material increases, the water absorption would also increase (Rayas-Duarte et al., 1998). There was no distinctive effect of fruit waste addition on the WAI, but WSI index decreased slightly.

WSI increased as a function of PDHF content (Figure 3.9). WSI is a parameter that reflects the degradation suffered by the components of the fiber (Larrea et al., 2005a). The increased WSI found in extruded products can be related to the lower molecular weight components, which can be separated quite easily from each other when the processing conditions are more severe (Colonna et al., 1989). PDHF is relatively high in fiber, protein and lipid content. Above critical concentration, the fiber molecules disrupt continues structure of the melt in extruder, impeding elastic deformation during extrusion (Moraru and Kokini, 2003). So, the greatest WSI values may be due to disintegration of starch granules and low molecular compounds from extrudate melt during extrusion process. This may cause in an increase in soluble material. The WSI increased with an increase in wheat germ oil, which was reported for wheat starch and meal (Singh and Smith, 1997). Badrie and Mellowes

(1992) reported a maximum WSI in cassava flour at 4% addition of soybean oil. WSI was not only due to starch content but also water soluble proteins (Pelembe et al., 2002). Pelembe et al. (2002) reported increasing WSI as the percentage of cowpeas increased in the blend and suggested that increasing WSI was probably due to water soluble cowpea protein.

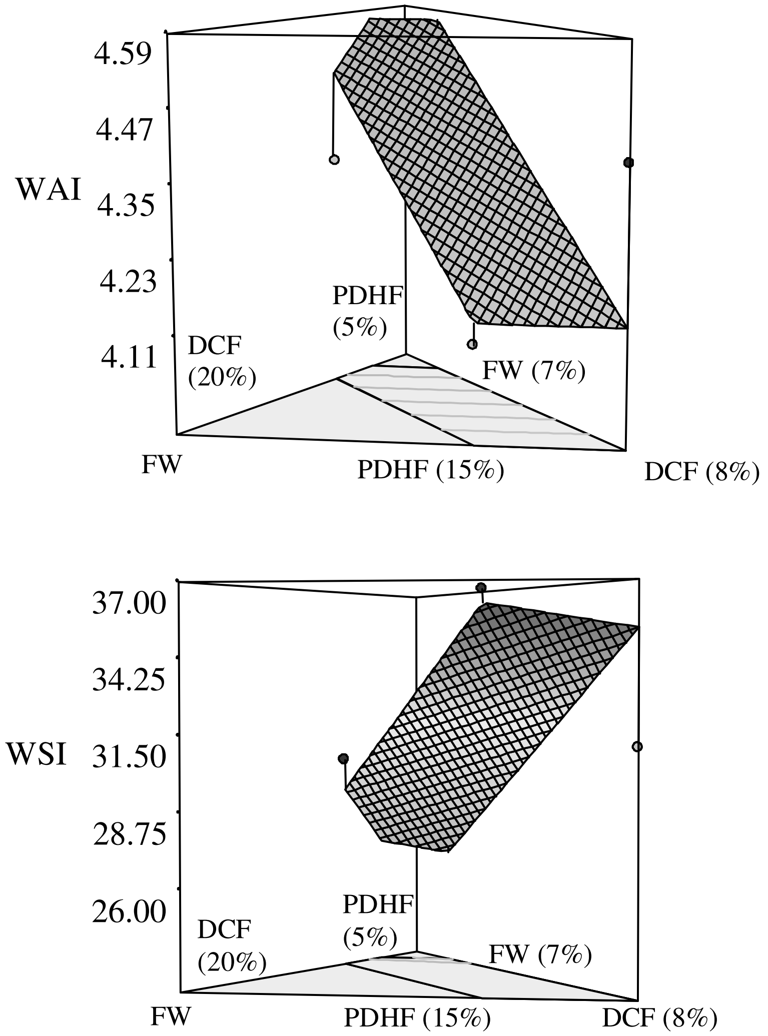


Figure 3.9 Response surface plots for water absorption and water solubility indices as a function of components at 12% moisture content, 200 rpm screw speed and 150°C barrel temperature (DCF, durum clear flour; PDHF, partially defatted hazelnut flour; FW, fruit waste)

WSI slightly decreased as the fruit waste percentage increased in the initial blend. In contrast to our study, Larrea et al. (2005a) reported the increase in WSI values of orange pulp with extrusion cooking process. They concluded that the extrusion process mainly allowed the solubilization of neutral pectic substance. In our study, fruit waste which mainly composed of orange pulp was blended into a cereal-based mixture. The differences observed here could be attributed to the nature of raw materials and the extrusion conditions which may affect the solubilization of pectic substance in orange pulp. It was observed that WSI was negatively correlated with porosity (r=-0.722, $p$<0.01) and WAI (r=-0.858, $p$<0.05) but positively correlated with bulk density (r=0.239, $p$<0.05) (Table A.3).

WAI is a gelatinization index and it is generally agreed that barrel temperature and feed moisture exert greatest effect on the extrudate by promoting gelatinization (Ding et al., 2005a). The feed moisture was found to have significant effect ($p$<0.01) on the WAI interaction with PDHF content (Table A.1). At higher PDHF content, there was no effect of moisture content on WAI. However, increasing moisture content from 12 to 18% increased the WAI of extrudates at lower PDHF content (Figure 3.10A). This result was consistent with the data obtained for radial expansion ratio. As the moisture content increased, both radial expansion ratio and water absorption index increased. At high moisture content, the viscosity of the starch would be low, allowing for extensive internal mixing and uniform heating which would account for enhanced starch gelatinization (Lawton and Handerson, 1972) which may lead to increased water absorption. Similar effects of increasing moisture content on WAI have been reported earlier for rice based extrudates (Ding et al., 2005a) and extrusion of rice with pea grit (Singh et al., 2007b).

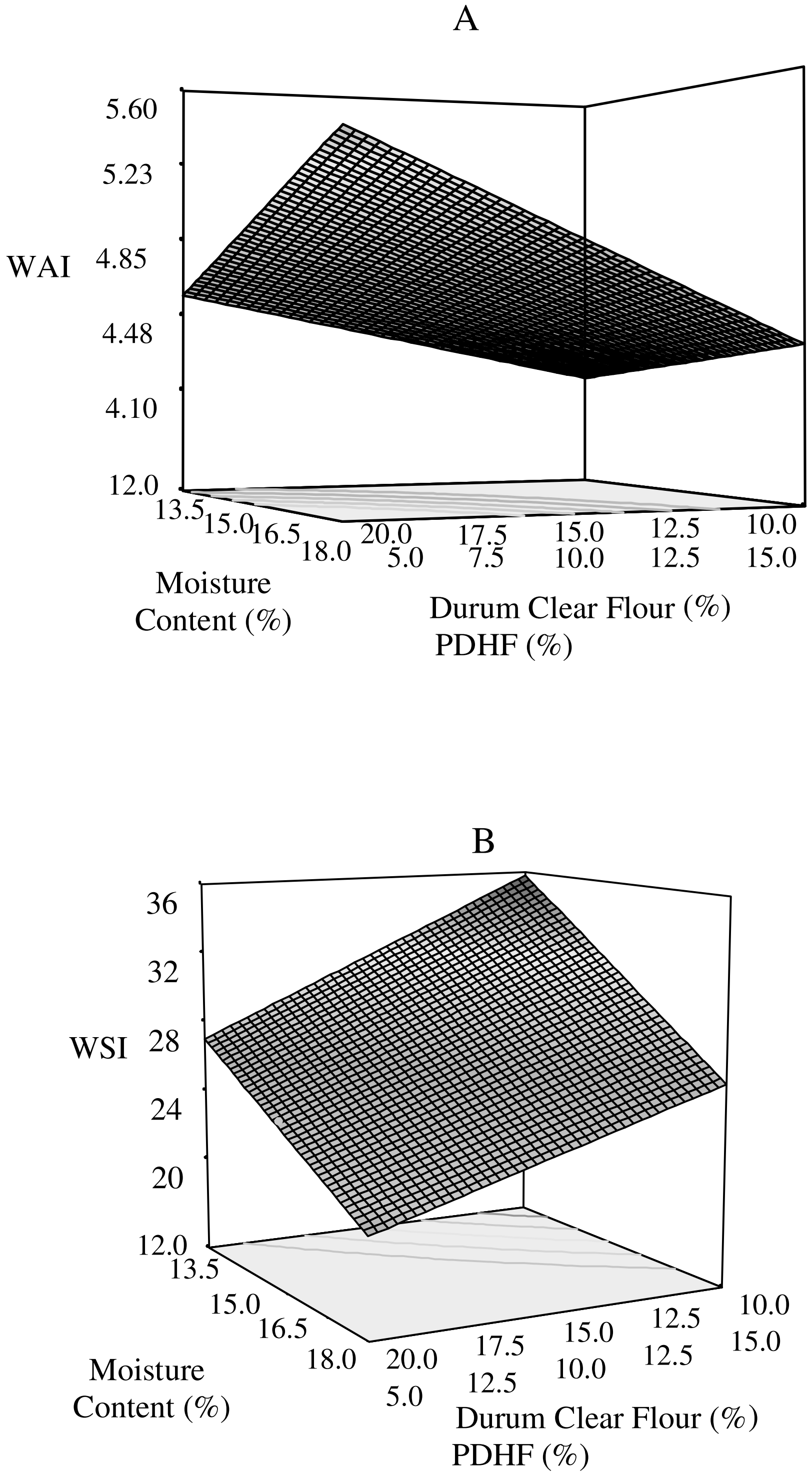
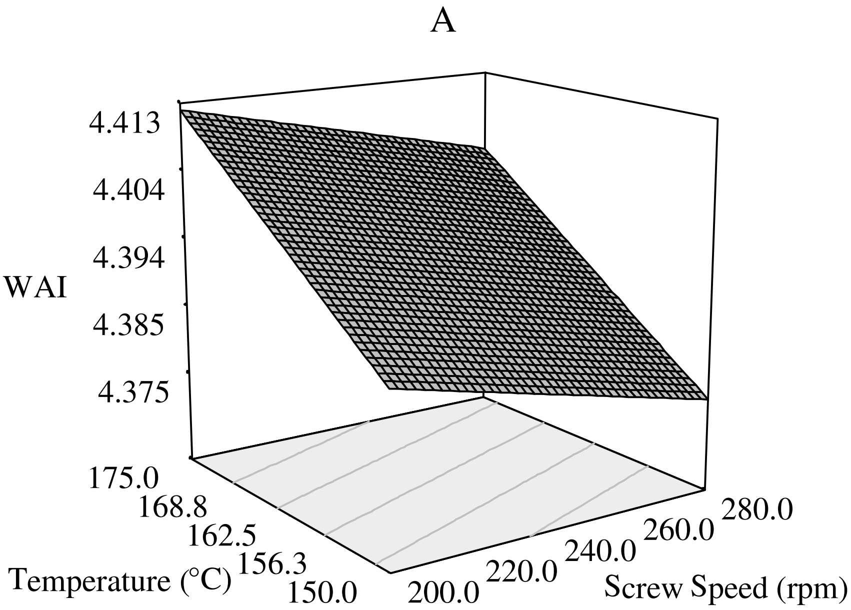


Figure 3.10 Response surface plots for water absorption and water solubility indices as a function of moisture content, durum clear flour and PDHF content at 5% fruit waste content, 200 rpm screw speed and 150°C barrel temperature (WAI, water absorption index; WSI, water solubility index; PDHF, partially defatted hazelnut flour)

The regression equation in Table A.1 showed that the effect of moisture content on WSI had significant ($p<0.01$) interaction with component variables used in this study. Increasing moisture content caused a decrease in WSI for all extrudate formulations (Figure 3.10B). Similar effects have been reported in literature for wheat and oat extrudates (Singh and Smith, 1997), rice-based extrudates (Ding et al., 2005a), cassava extrudates (Bedrie and Mellowes, 1991), extrusion of yam flour (Sebio and Chang, 2000) and maize-finger extrudates (Onyango et al., 2004). It was indicated that increasing WSI is caused by greater shear degradation of starch during extrusion at low moisture conditions.

WAI increased at the higher extrusion temperature, whereas it decreased with increasing screw speed (Figure 3.11A). WAI generally increases along with the increase in temperature, after which it decreases, probably due to increased dextrinization. It was found that WAI achieved a maximum value at extrusion temperatures of 180-200°C (Mercier and Feillet, 1975). The WAI depended on the degree of starch damage which was related to barrel temperature and maximal degradation occurred at high temperature (Anderson et al., 1969). Singh and Smith (1997) observed increase in WAI of wheat starch, whole wheat meal and oat extrudates with increase in both feed moisture content and temperature. WAI decreased as a function of screw speed (Figure 3.11A). High input of thermal energy due to high residence time (at low screw speeds) may lead to enhanced level of starch degradation and increased WAI. Temperature significantly affects the WSI in interaction with durum clear flour content ($p<0.1$). The effect of temperature and screw speed on WSI was shown in Figure 3.11B. WSI decreased at higher screw speed as in WAI. On the other hand, WSI slightly decreased with increasing temperature which had adverse effect on WAI.



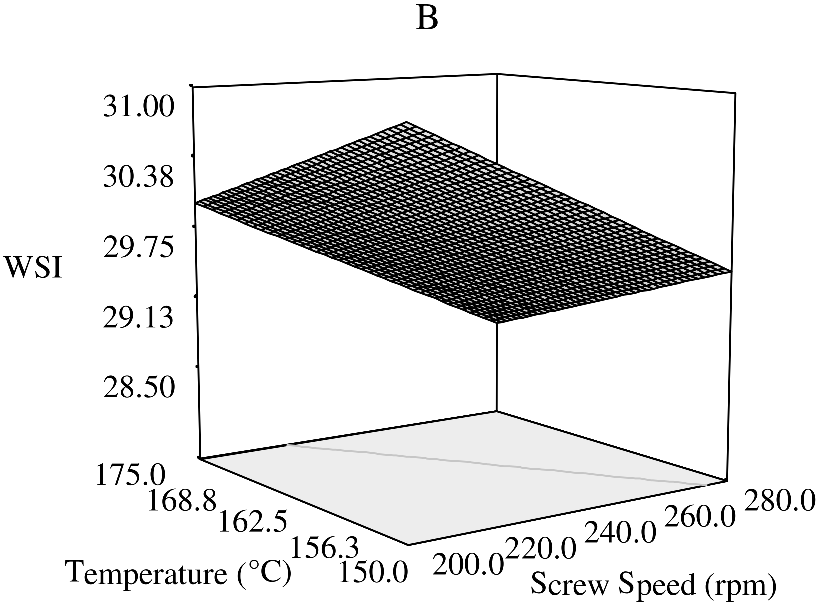


Figure 3.11 Response surface plots for water absorption and water solubility indices as a function of screw speed and temperature at 15.6% durum clear flour, 9.4% PDHF, 5% fruit waste content and 12% moisture content (WAI, water absorption index; WSI, water solubility index; PDHF, partially defatted hazelnut flour)

### 3.1.5 Color

The color of sample is denoted by the three color parameters the Hunter *L*, *a* and *b*. The *L* value gives a measure of the lightness of the product color. The redness and yellowness are denoted by the *a* and *b* values, respectively. The measured values of the color parameters of extruded blends varied in the range from 50.41±0.49 to 61.19±0.74 for lightness, 5.92±0.20 to 8.14±0.02 for redness and 14.25±0.72 to 18.06±0.05 for yellowness. The model coefficients accounting for the individual and combined effect of components and extrusion process parameters are reported in Table A.2. The analysis of variance test for *L*, *a* and *b* values of extruded samples was significantly ($p<0.01$) enhanced with the linear terms of durum clear, PDHF and fruit waste content. The color coefficients also had significant interactions among the components and process variables.

The color of the product tends to turn slightly darker when the PDHF content increased in the formulation (Figure 3.12). The increase in darkness can be attributed to the darkness of the PDHF compared to the nearly white rice and durum clear flour. Sacchetti et al. (2004) reported the darkening effect of chestnut flour addition to the rice. Lightness of extrudates increased up to 5% waste content, however further increase in waste content caused lightness to decrease. Ahmed (1999) reported the decrease in lightness of corn based extrudate with increasing flaxseed addition. He concluded that decrease in L value may have been due to more browning reaction because fiber addition increased the extrudate temperature or might have been caused by the pigments present in flax flour.

The response surface plot for the redness and yellowness (Figure 3.12) of the extrudates showed gradual increase in redness and yellowness with increasing additions of fruit waste. This trend can be attributed to the color of the fruit waste which contains orange peel, tomato pomace and grape seed. On the other hand, the addition of PDHF to the blend caused both redness and yellowness to decrease.

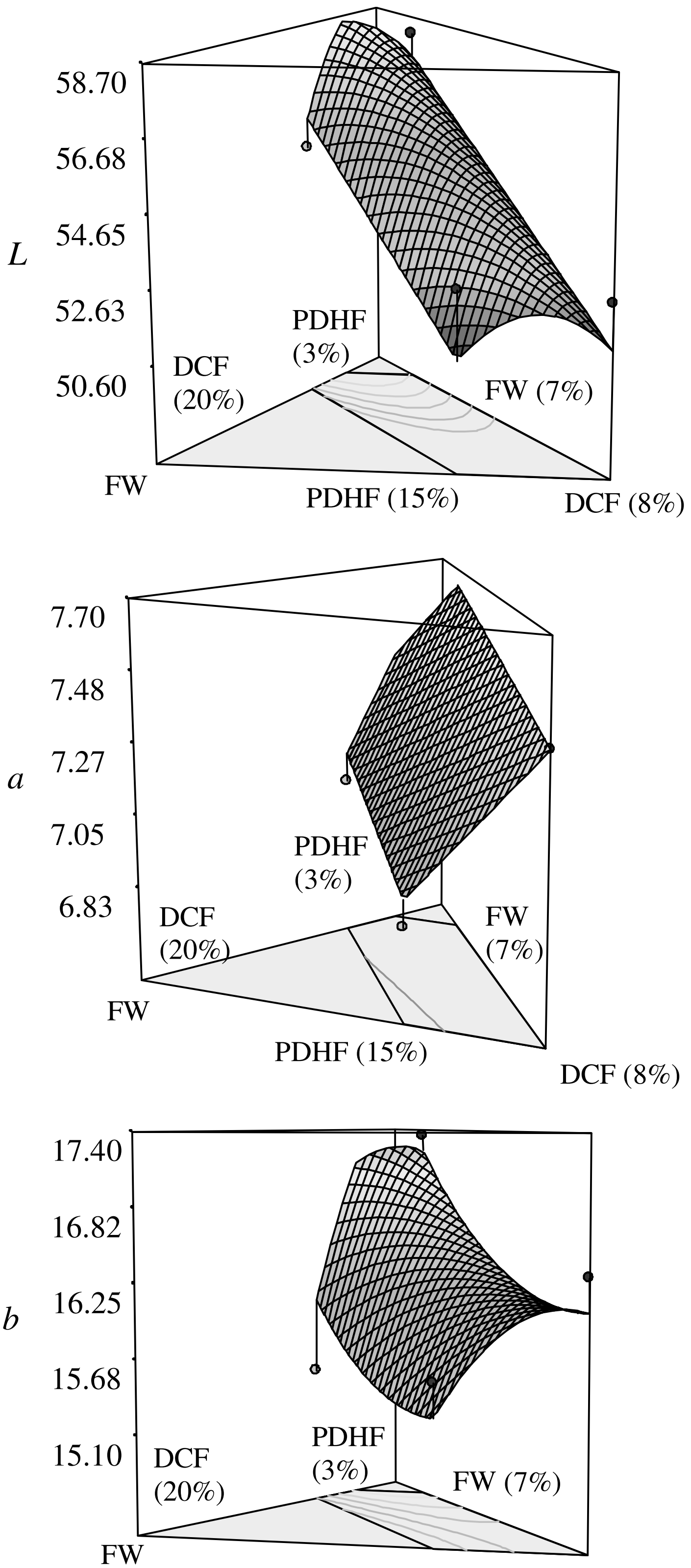


Figure 3.12 Response surface plot for color values (*L, a, b*) as a function of components at 12% moisture content, 200 rpm screw speed and 150°C barrel temperature (DCF, durum clear flour; PDHF, partially defatted hazelnut flour; FW, fruit waste)

Extrusion temperature was found to have no significant effect on extrudate lightness. On the other hand, lightness increased with increasing screw speed (Figure 3.13). Increasing screw speed would decrease the residence time in extrusion cooking, and thus reduces color change (Ilo et al., 1999). Increasing moisture content generally increased lightness of product. Similar trend was reported for the extruded maize grits (Ilo and Berghofer 1999).

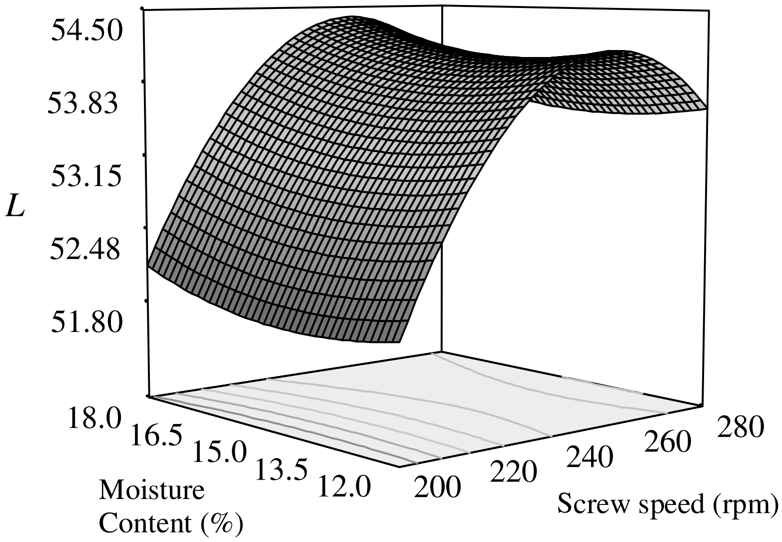


Figure 3.13 Response surface plot for *L* value as a function of moisture content and screw speed at 13.5% durum clear flour, 13.5% PDHF, 3% fruit waste content and barrel temperature of 150°C

The effect of process parameters on the redness was not significant in linear relation, but significant in interaction with component variables. The representation of *a* value of the extrudates as a function of barrel temperature and screw speed is given in Figure 3.14. The effect of temperature on redness was found to be dependent on blend formulations as indicated by linear interaction with PDHF and triple interaction with PDHF and moisture contents. The redness generally decreased with increasing barrel temperature at most blend formulations. Some of pigments naturally present in fruit waste may have been damaged by the thermal treatment and some browning may have made up color loss. The redness decreased with increasing screw speed. Increasing moisture content generally decreased redness of product at most composition of the blend. Ilo and Berghofer (1999) observed same trend for the extruded maize grits. The regression analysis of the measured data for the yellowness, as presented in Table A.2, showed that there was no significant effect of process variables on yellowness index.

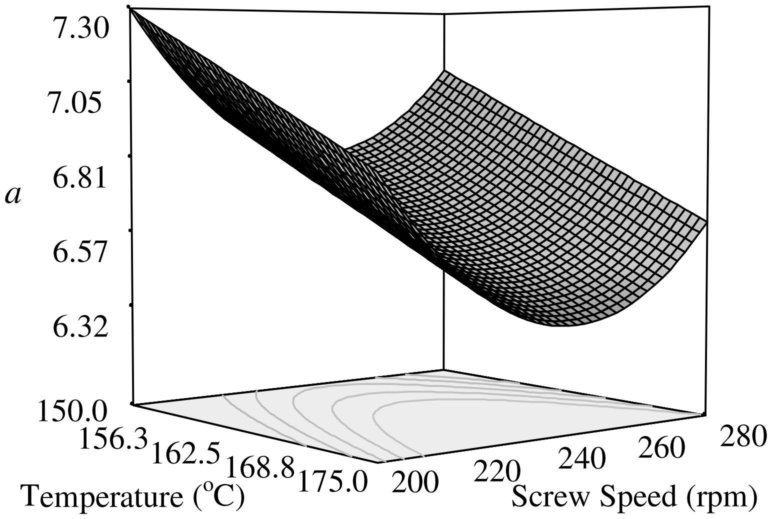


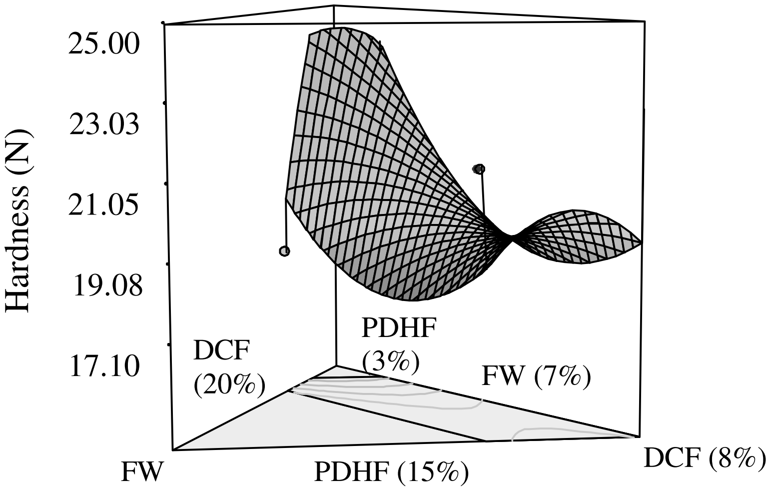
Figure 3.14 Response surface plot for *a* value as a function of temperature and screw speed at 18% durum clear flour, 7% PDHF, 5% fruit waste contents and 12% moisture content

### 3.1.6 Textural Properties

The textural properties of extruded products are generally described by the hardness and crispness. The hardness of expanded extrudate is a perception of human being and is associated with the expansion and cell structure of the product. The maximum peak force from texture analyzer represents the resistance of extrudate to initial penetration and is believed to be hardness of extrudate (Ding et al., 2005a; Ding et al., 2005b). Coefficient of regression models obtained for hardness (N) and total area (N×s) values of extruded samples following the response surface regression procedure are presented in Table A.2. Although the $r^2$ for these responses seems not very good, it was considered acceptable because these analyses generally show great variability with respect to process and component variables used in this study. Mendonça et al. (2000) observed similar results for the corn bran added snacks, $r^2$ for the hardness and fracturability ranged from 0.61 to 0.62. The entire main effects and interaction of effects were calculated for the significant models for hardness and total area. The composition of extruded samples was found to have linear effect ($p<0.01$) on the hardness and total area values. Not only single parameters showed significant influences on the hardness and total area values of the products, but also interactions among them and process variables showed significant effects in determining the textural properties of the extrudates (Table A.2).

For extruded foods it was desirable to have low values for hardness (Mendonça et al., 2000). Hardness measured for all extrudates varied between 14.93±1.41 and 33.38±1.78 N. The response surface for the hardness showed saddle shape as shown in Figure 3.15. Fruit waste and PDHF contents were the most important parameters affecting the hardness of extrudates in complex manner. Both high and low fruit waste contents in the blend resulted lowest values for hardness, whereas intermediate level of fruit waste resulted harder extrudates at increased PDHF content. However at low PDHF content, increasing fruit waste content in blend increased hardness of extrudates.

Increasing PDHF content up to ~12 % improved product hardness, however beyond this value, extrudate hardness increased. Decrease in hardness of extrudates may be related with the addition of oil in the PDHF to the blends which improved extrusion process and texture. Fat provides a powerful lubricant effect in extrusion cooking and it improves texture; however excess fat reduces product expansion (Cheftel, 1986). Suknark et al. (1997) reported when partially defatted peanut flour was substituted to starch at low levels (15-30%), the shear strength of the extrudates decreased. Similar findings were reported for the rice flour-amaranth blends. They reported that increasing amaranth content up to 21% decreased breaking strength of the extrudates, but further increase in amaranth content increased breaking strength of the extrudates (Ilo et al., 1999). Increasing hardness of extrudates with further PDHF addition may be due to increasing protein content of blend. Proteins absorb more water, lowering the extent of starch gelatinization and expansion (Suknark et al., 1997) and thus probably increasing the hardness of extrudates. Bahattacharya and Hanna (1987) reported that extrudates which have greater gelatinization will increase the expansion and extend the starch bonds resulting in weakened bonds, thus reduced hardness.



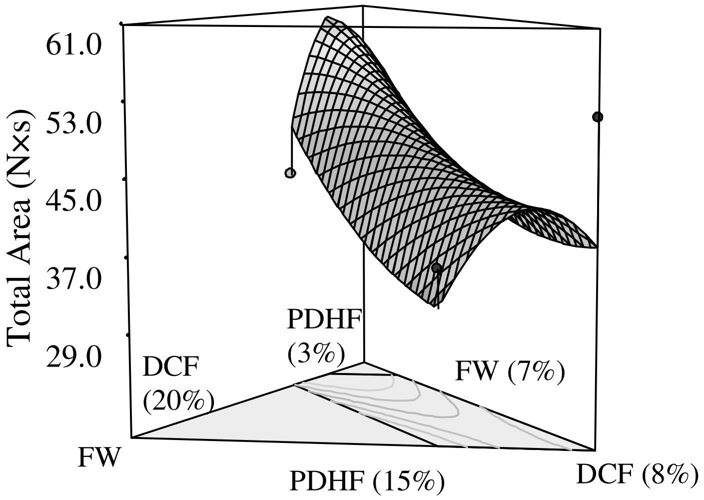


Figure 3.15 Response surface plot for textural parameters (hardness and total area) as a function of components at 12% moisture content, 200 rpm screw speed and 150°C barrel temperature (DCF, durum clear flour; PDHF, partially defatted hazelnut flour; FW, fruit waste)

No significant effect of moisture content and temperature on hardness was observed for the given data. Screw speed significantly affects the hardness in interaction with waste content ($p<0.01$), and also in triple interaction with durum clear, fruit waste and PDHF content (Table A.2). At low and high fruit waste content, increasing screw speed increased the hardness, whereas at intermediate waste content (5%), there was no effect of screw speed on hardness (Figure 3.16). A low screw speed is associated with high residence time; consequently the food material inside the extruder receives more input of thermal energy in a low shear environment. High input of thermal energy due to high residence time (at low screw speeds) leads to the creation of enhanced level of superheated steam, hence the product will have good expansion which creates flaky and porous structures due to formation of air cells, the stress during shear will also be low (Bahattacharya, 1997).

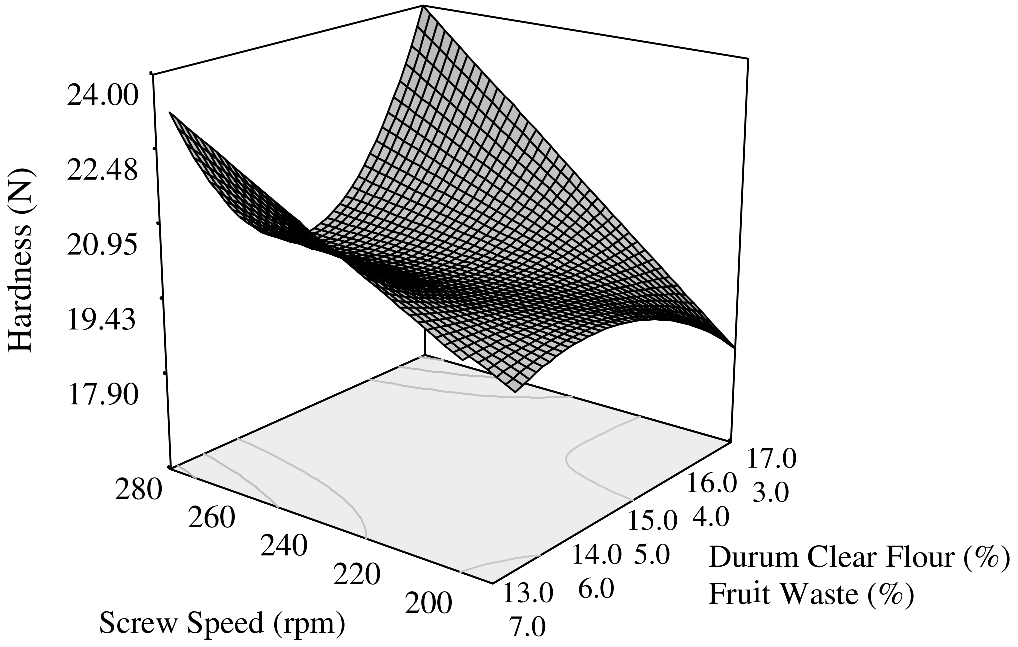


Figure 3.16 Response surface plot for hardness as a function of screw speed, durum clear flour and fruit waste content at 12% moisture content, 10% PDHF content and 150°C barrel temperature

Change in total area (N×s) with respect to composition showed similar trend with the hardness as shown in Figure 3.15. As in hardness, total area was maximum at intermediate fruit waste content, whereas it decreased with increasing PDHF content. This may be related to the reduced expansion with increasing PDHF content. The reduced starch conversion and compressed bubble growth would result in a dense product and reduced crispness (area under the force-deformation curve) (Ding et al., 2005a), as observed in this work.

## 3.2 Sensory Evaluation of Extruded Foods

The photographs of samples used for the sensory analysis are given in Figure 3.17. The statistical evaluation of sensory properties of the extruded products is given in Table 3.2. The results show that most products showed no distinctive orange and hazelnut flavor. The intensity ratings for the hazelnut flavor were greater with respect to the orange flavor. Bitterness varied significantly between samples ($p$<0.05), PDHF level apparently responsible for this effect. The samples with the higher PDHF levels (12.2 to 15%) also had higher bitterness scores (3.0 to 3.6, slightly bitter) than the low PDHF samples (5 to 7%) with bitterness scores of 1.9 to 2.6 (very slightly bitter). All extrudates showed no distinctive off-flavor. There was no significant differences in off-flavor score ($p$<0.05) among extrudates.

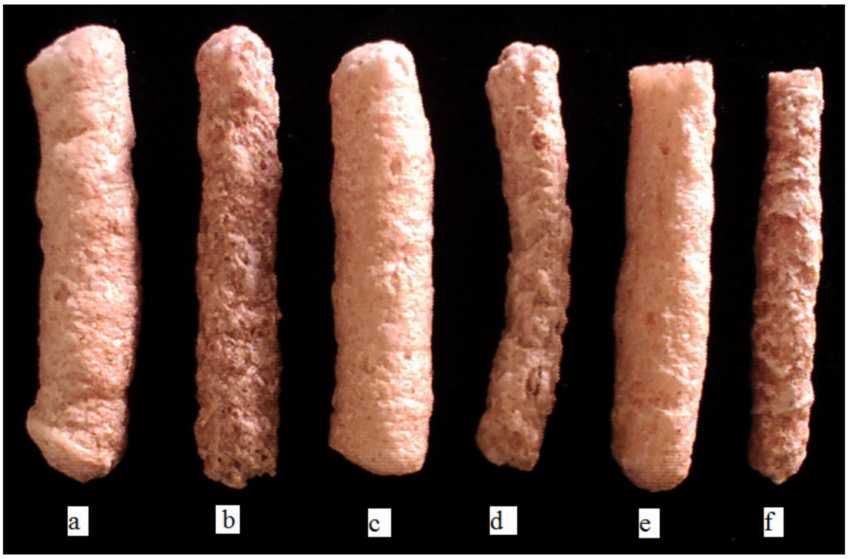


Figure 3.17 Photographs of extruded samples evaluated by the sensory panel (DCF, durum clear flour; PDHF, partially defatted hazelnut flour; FW, fruit waste)
(a) DCF = 20%, PDHF = 7%, FW = 3% (b) DCF = 12%, PDHF = 15%, FW = 3%
(c) DCF = 20%, PDHF = 5%, FW = 5% (d) DCF = 12.8%, PDHF = 12.2%, FW = 5%
(e) DCF = 18%, PDHF = 5%, FW = 7% (f) DCF = 8%, PDHF = 15%, FW = 7%

Hedonic scores for the sensory quality of extruded products having various formulation ranged from 2.9 (dislike) to 6.8 (like slightly) for all attributes. The samples with lower PDHF levels (5 to 7%) had higher color scores (5.6 to 6.4) than the high PDHF (12.2 to 15%) with color scores of 2.9 to 3.4. This was probably due to darker color of the PDHF compared to fruit waste and durum clear flour. This result was consistent with the data obtained by instrumental measurement such that, the color of the product became darker when the PDHF percentage increased in extrudate formulation. There was no significant effect (*p*<0.05) of fruit waste addition on sensory scores for the color. Air cell homogeneity of samples did not vary significantly (*p*<0.05), with the scores for this attribute falling in a narrow range from 5.2 to 6 (just alike good in homogeneity). Textural properties of all extrudates, especially crispness and breakability values generally took high scores from the panelists. Extrudate formulation did not significantly affect the sensory hardness. On the other hand, the extrudate having 5% fruit waste and 12.2% PDHF had lower sensory score for the crispness and breakability. Mostly, the extrudate having lower PDHF content were preferred with respect to crispness scores. The overall acceptability scores varied significantly (p<0.05) amongst samples, with scores ranging from 3.6 (dislike moderately) to 6.6 (like slightly). The extruded samples with low PDHF content had higher levels of overall acceptance than the ones with high PDHF content. Thus, it appears that a high percentage of PDHF led to the extrudate with negative overall acceptance. There was no significant effect (*p*<0.05) of fruit waste addition on sensory properties of produced extrudates. These results indicated that extrusion of PDHF, fruit waste and durum clear flour in combination with rice grit can produce acceptable extrudate. However, the PDHF content was the most important parameter affecting the sensory properties of extrudates.

Table 3.2 Mean scores for sensory properties of extrudates

| Extrudate Formulation (%) DCF/PDHF/FW | | Intensity Scores† | | | | Hedonic Scores* | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | Sample Name | Orange Flavor | Hazelnut Flavor | Bitterness | Off-Flavor | Color | Air Cell Homogeneity | Hardness | Crispness | Breakability | Overall Acceptability |
| 20/7/3 | a | 1.5a | 3.3ab | 2.6ab | 1.9a | 5.6b | 5.6a | 5.4a | 6.2b | 5.7ab | 6.1c |
| 12/15/3 | b | 1.9a | 3.1ab | 3.4b | 2.0a | 2.9a | 6.0a | 5.4a | 6.1b | 6.3ab | 4.6b |
| 20/5/5 | c | 1.6a | 3.4b | 1.9a | 1.9a | 5.7b | 5.2a | 5.2a | 6.0b | 5.9ab | 6.2c |
| 12.8/12.2/5 | d | 2.0a | 2.4a | 3.0b | 2.1a | 3.4a | 5.9a | 5.2a | 4.7a | 5.4a | 3.6a |
| 18/5/7 | e | 2.1a | 2.9ab | 2.6ab | 2.0a | 6.4b | 5.4a | 5.8a | 6.2b | 6.5b | 6.6c |
| 8/15/7 | f | 2.1a | 3.2ab | 3.6b | 2.0a | 3.4a | 5.6a | 6.8a | 5.7b | 6.0ab | 4.4b |

Values with the same letter are not significantly different at $p<0.05$ level. DCF, durum clear flour, PDHF, partially defatted hazelnut flour, FW, fruit waste

† On a nine point scale, 1-absent, 9-very high

* On a nine-point scale, 1-dislike extremely, 9-like extremely

### 3.3 Functional Properties of Extruded Foods

There is a growing interest in fortifying cereals with high phenolic and fiber containing functional additives from a health standpoint. The fruit wastes, defatted hazelnut flour and durum clear flour can be used in combination with cereal flours for production of nutritionally-balanced convenient extruded snack foods due to their valuable characteristics. Durum clear flour, partially defatted hazelnut flour (PDHF) and fruit wastes were chosen for their relatively high fiber (Villarroel et al., 1989; Braddock 1999; Esposito et al., 2005) and phenolic content (Bocco et al., 1998; Jayaprakasha et al., 2001; Schieber et al., 2001; Shahidi, et al., 2007; Liyana-Pathirana and Shahidi, 2007) to be included in the formulation of extruded food produced in this study.

Response surface methodology was used to model functional properties of extruded samples with respect to component and process variables. Coefficients of regression models obtained for functional properties of extruded samples following the response surface regression procedure are presented in Table A.4. The coefficient of determinations of regression equations changed from 0.719 to 0.988 with significant probability values ($p<0.0001$) and non-significant lack of fit values (Table A.4). These equations could be adequately used as predictor models with relatively high coefficient of determinations and non-significant lack of fit.

#### 3.3.1 Total Phenolic Content and Antioxidant Activity

The concentrations of total phenolic compounds and antioxidant activity of raw materials were found respectively for fruit waste blend as 21.46±0.11 mg ferulic acid/g sample (d.b.) and 75.0±0.7%; for PDHF as 7.60±0.04 mg ferulic acid/g sample (d.b.) and 38.0±0.4%; for durum clear flour as 2.68±0.01 mg ferulic acid/g sample (d.b.) and 16.1±0.8%; for rice grit as 0.70±0.01 mg ferulic acid/g sample (d.b.) and 3.7±0.6%. All raw materials had a marked phenolic content and antioxidant activity except rice grit. The values obtained for the raw materials were comparable to the reported values in the literature. Fruit waste blend showed highest antioxidant activity in comparison to other raw materials. Fruit waste blend was mainly composed of orange peel which has been shown to possess high antioxidant activity (Bocco et al., 1998; Schieber et al., 2001).

The blend also contained grape seed and tomato pomace. Grape seeds are rich sources of polyphenolics such as catechin and epicatechin which have been shown to act as strong antioxidants and exert health promoting effects (Jayaprakasha et al., 2001). Lycopene in tomato pomace exhibits the highest antioxidant activity and singlet oxygen quenching ability of all dietary carotenoids (Kaur et al., 2006). A high phenolic content and antioxidant activity were also observed for the PDHF. Shahidi et al. (2007) found that hazelnut skin and defatted hazelnut kernel had high levels of phenolic compounds (13.7-577.7 mg catechin/g extract), which were mainly gallic, caffeic, *p*-Coumaric, ferulic and sinapic acids. They suggested that hazelnut by-products could potentially be considered as an excellent and readily available source of natural antioxidants.The total phenolic content and antioxidant activity were also high for the durum clear flour. It was shown that the concentration of bioactive constituents was greater in the outer layers of the grain; thus the bran fraction demonstrates higher antioxidant activity than other milling fractions (Beta et al., 2005; Esposito et al., 2005; Liyana-Pathirana and Shahidi, 2007). As phenolic compounds are found to be concentrated in the outermost layers, the bran fractions obtained as milling by-products may be used as a natural source of antioxidants and as a value-added product in the preparation of functional food ingredients and/or for enrichment of certain products (Liyana-Pathirana and Shahidi, 2007).

Total phenolic content and antioxidant activity measured for all extruded samples ranged between 2.52±0.02-9.66±0.26 mg ferulic acid/g sample (d.b.) and 7.1±0.5-33.0±0.8% respectively. The coefficient of determinations of regression equations changed from 0.719 to 0.850 with significant probability values ($p<0.0001$) (Table A.4). Fruit waste and PDHF content had significant effect and positive correlation with both total phenolic content and antioxidant activity. However, it was negatively affected by the linear coefficient of durum clear flour content. The effect of PDHF content was more than that of the both durum clear flour and fruit waste contents. As well as, the linear effect of feed moisture content which had interaction with fruit waste, PDHF and durum clear flour content was found to significantly influence the total phenolic content and antioxidant activity. In extruded samples, antioxidant activity had positive correlation

($r=0.926$, $p<0.01$) to the content of phenolic compounds, suggesting that the antioxidant activity of produced extrudate is due to a greater extent to their phenolic content.

The effect of feed composition on the total phenolic content and antioxidant activity of extrudates is presented in Figure 3.18. Results indicated that increasing PDHF and fruit waste content caused increase in both total phenolic content and antioxidant activity of the extrudates. The increase in phenolic content with addition fruit waste is comparable to those reported for corn-based extrudate (Camire et al, 2007). They reported that phenolic and anthocyanin content of extrudates increased with addition of 1% of dehydrated fruit powders including blueberry, cranberry, Concord grape and raspberry. Fruit waste blend used in this study had high phenolic content and antioxidant activity. This might be due to the several types of flavonoids found in orange peel and grape seed (Bocco et al., 1998; Schieber et al., 2001; Jayaprakasha et al., 2001).

The regression equation in Table A.4 showed that the effect of moisture content on total phenolic content and antioxidant activity had highly significant interaction with component variables ($x_1$, $x_2$ and $x_3$) used in this study. Figure 3.19 shows the effects of moisture content and the temperature on both total phenolic content and antioxidant activity of extruded samples. Increasing moisture content and temperature caused a decrease in total phenolic content and antioxidant activity for all extrudate formulations. In food systems such as extruded products, phenolics are subjected to various degrees of heat-moisture treatments. During processing, phenolics other than those endogenous in cereal grains may be formed as by-products of enzymatic or thermal degradation or as products of polymerization of simple phenolics. Phenolics can become modified such that their solubility and functional group properties are altered (Beta, 2003). Dlamini et al. (2007) expressed that the higher moisture content probably promoted phenolic and tannin polymerization, which affected extractability of phenols and tannins, and reduced antioxidant activity. Increasing barrel temperature may have a destructive effect on phenolic antioxidants which would be easily damaged from high temperature environment (Kalt, 2005). It was stated that extrusion caused a reduction in total phenolics of extruded oat cereals (Viscidi et al., 2004), similar to our findings. Similar trend was also reported for total phenols and tannins in whole and decorticated tannin sorghums (Dlamini et al., 2007) and total anthocyanin content of extruded blueberry-corn cereals (Chaovanalikit et al., 2003).

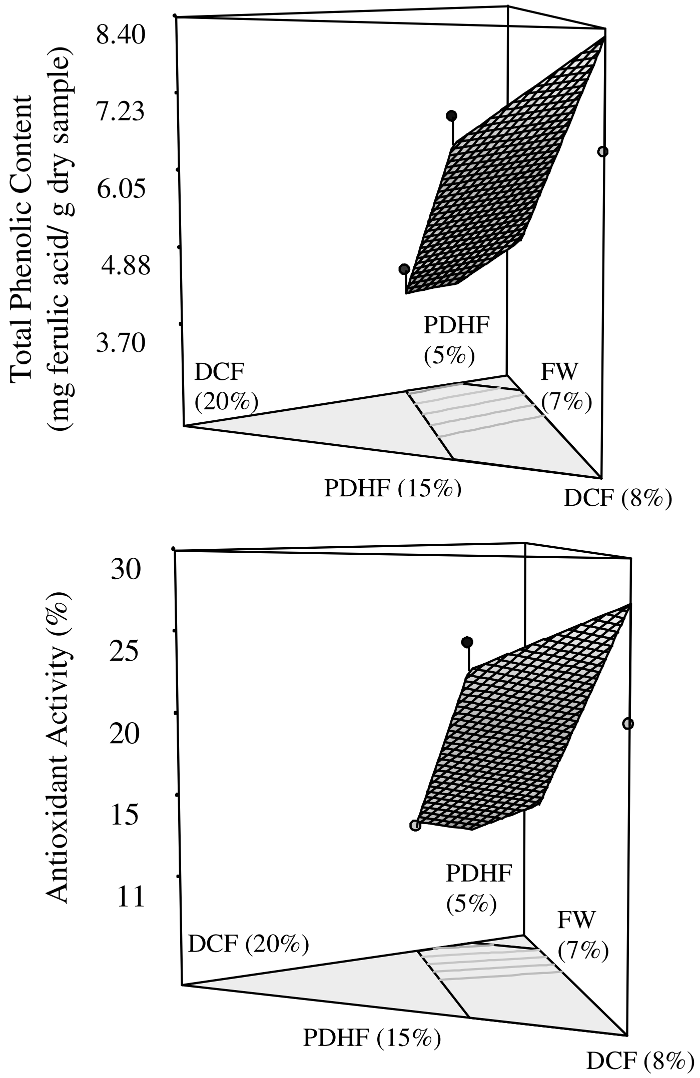


Figure 3.18 Response surface plots for total phenolic content and antioxidant activity as a function of feed composition at 12% feed moisture content, 200 rpm screw speed and 150°C barrel temperature (DCF, durum clear flour; PDHF, partially defatted hazelnut flour; FW, fruit waste)

Screw speed significantly affects the total phenolic content in interaction with PDHF content (*p*<0.05). Increasing screw speed caused a slight increase in total phenolic content for most compositions of extruded samples. Increased screw speed associates with decreased residence times (shorter reaction times), so destruction of phenolic compounds during extrusion process would be lower in case of increased screw speeds.

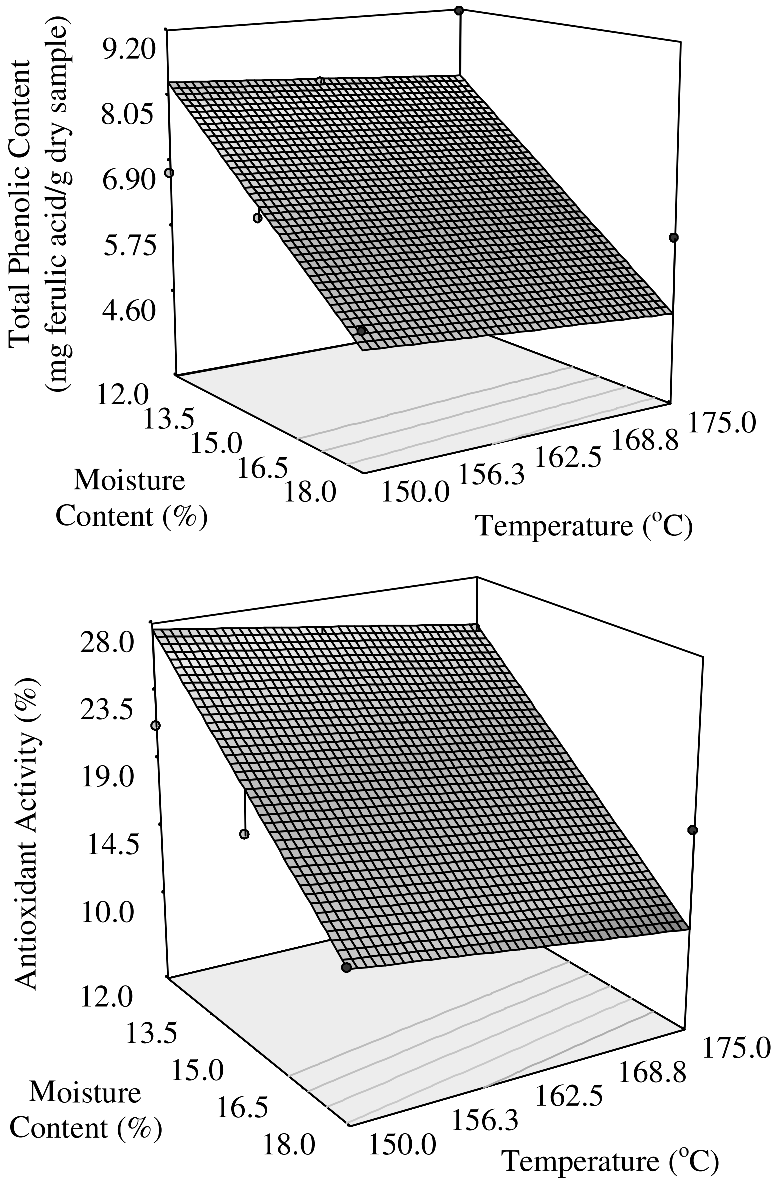


Figure 3.19 Response surface plots for total phenolic content and antioxidant activity as a function of moisture content and temperature (The extrusion variables studied at 8% durum clear flour, 15% PDHF and 7% fruit waste contents and 200 rpm screw speed)

### 3.3.2 Measurement of Gelatinization by DSC and Polarized Light Microscopy Techniques

In this study, DSC was used to analyse the gelatinization of starch in raw materials and selected unextruded blends and extrudates. In the case of starch gelatinization, the DSC thermograms give the possibility of transition enthalpies occurred during melting of the crystallites in the starch as well as precise measurement of the transition temperatures (Xie et al., 2006). Gelatinization endotherms of raw materials, unextruded blend and extrudate with excess water at temperature range between 10 and 120°C is provided in Figure 3.19. Among the raw materials, durum clear flour and rice grit produced endothermic peak while there was no endothermic peak in the temperature range investigated for the PDHF. It was observed that there is a large gelatinization endotherms appearing at about 70°C for rice grit and 65°C for durum clear flour similar to previous report of Sagol et al. (2006).

Unextruded blend also showed endothermic peak around 70°C which is the indication of starch gelatinization at this temperature range (Figure 3.20). However, no peak was observed in the gelatinization range for the extruded sample. This may indicate that extrusion processes gelatinized the most of starch granules. Although, extruded samples partially included raw starch granules, they showed even DSC curve because the amount of raw starch left in extruded samples may be minimal and undetectable by the DSC. Gomez and Aguilera (1984) postulated that the amount of native starch left in extruded corn starch was minimal and undetectable by DSC procedure as in similar to our study. Bhatnagar and Hanna (1996) used the DSC for measuring the degree of starch gelatinization in single and twin screw extruded corn starch samples which were taken from different locations along the extruder barrel. They reported that the gelatinization measured by DSC indicated 90-95% starch gelatinization in the samples which were taken from die exit in both single screw and twin screw extruders.

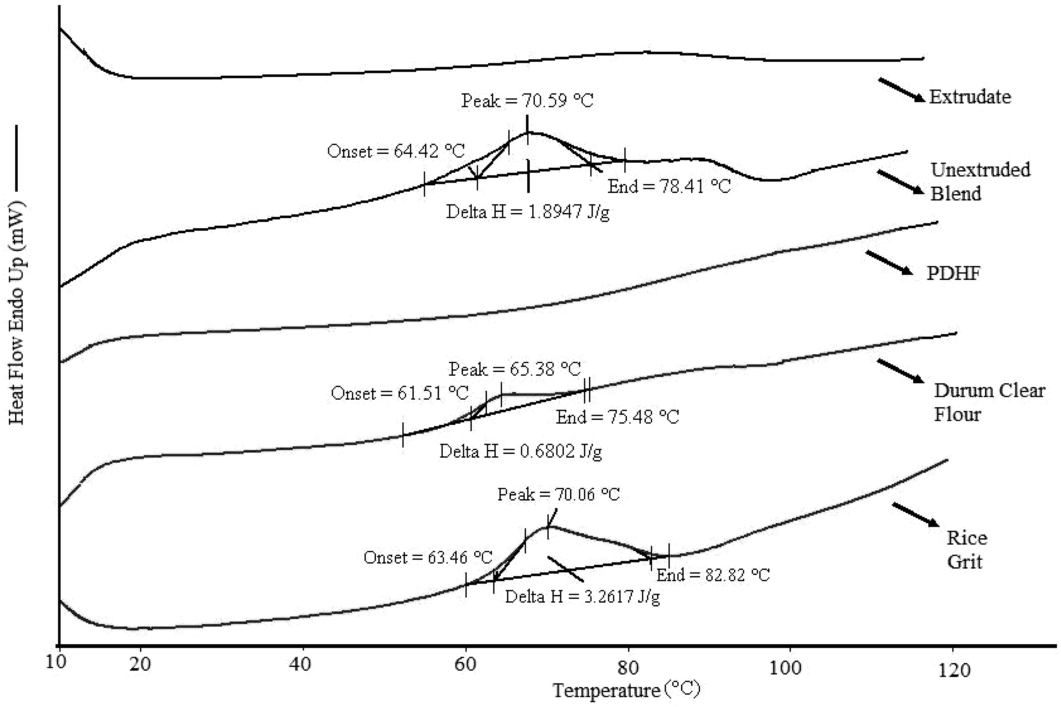


Figure 3.20 DSC curves of raw materials, unextruded blend and extruded sample

Polarized light microscopy is typically used to investigate the physical state of starch granules, which in the crystalline state are characterized by the presence of a Maltese cross pattern. When starch granules are viewed under the microscope using polarized light, they exhibit a phenomenon known as birefringence. The refraction of polarized light by the intact crystalline regions in starch gives characteristic "Maltese cross" patterns on each granule. The disappearance of these crosses (loss of birefringence) is an indication of the irreversible swelling of starch granules that occurs above the gelatinization temperature (Zimeri and Kokini, 2003).

Figures 3.21 and 3.22 show starch granules of the ground rice grit (a and b) and durum clear flour (a and b) samples displaying a clear ‘‘Maltese cross’’ under polarized light, respectively. The minority of the starch granules in extruded sample (Figure 3.23 a and b) retained their birefringence. Bright-field and polarized-light microscopy studies showed that partially birefringent granules in extruded sample had irregular shapes and retained birefringence in a portion of each granule. Although high extrusion temperature used in the present study exceeded the gelatinization temperature of starch, birefringence was still observed in some of the starch granules in extruded sample. This may indicate incomplete gelatinization of starch in extruded samples. Gelatinization takes place typically with low amount of water and at high pressure conditions under extrusion cooking environment. The loss of crystallinity during extrusion is no longer caused by penetration of water but by mechanical disruption of the molecular bonds by the intense shear fields within extruder (Wen et al., 1990). Therefore, under extrusion at low moisture content, a mixture of small amounts of gelatinized and melted states of starch as well as fragmentation exist simultaneously (Lai and Kokini, 1991). Gomez and Aguilera (1984) stated that dextrinization appeared to become the predominant mechanism of starch fragmentation during low moisture and high-shear extrusion. Amount of water during extrusion process was limited, so there may be insufficient water amount for complete swelling and gelatinization of starch granules in extruded sample which exhibited partial ''Maltese cross'' under polarized light.

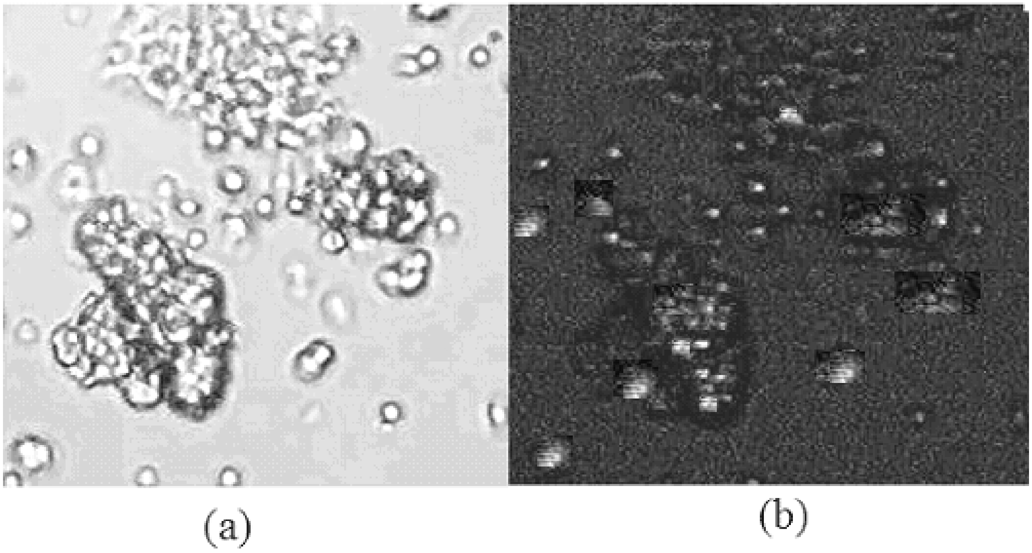

(a) (b)

Figure 3.21 Bright-field (a) and polarized light (b) micrographs of rice sample

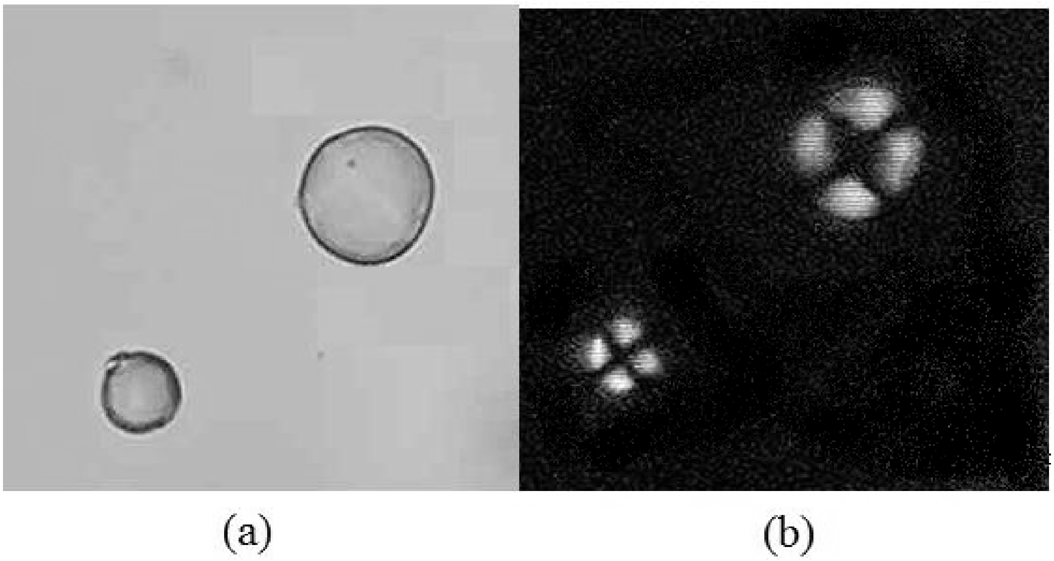

(a) (b)

Figure 3.22 Bright-field (a) and polarized light (b) micrographs of durum clear flour sample

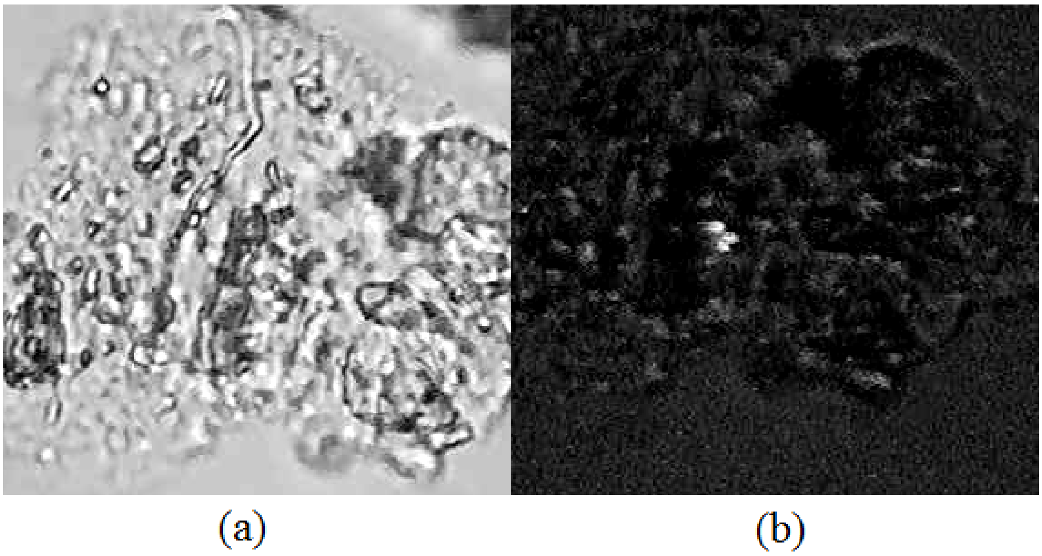

(a) (b)

Figure 3.23 Bright-field (a) and polarized light (b) micrographs of extruded sample

### 3.3.3 Percent Starch Gelatinization and Starch Digestibility

Extrusion has been shown to thoroughly gelatinize starch even at very low food moisture levels, thus also enhancing starch digestibility (Gomez and Aguilera, 1983). In this study, it was observed that there was still ungelatinized portion of starch in extruded samples as indicated by information from polarized light microscopy. Percent starch gelatinization and in *vitro* starch digestibility measured for all extruded samples ranged between 81±0.4-97±0.9% and 275.0±4-393.4±2 mg maltose/g sample (d.b.), respectively. In extruded samples, percent starch gelatinization were found to be positively correlated ($p<0.01$, r=0.585) with the starch digestibility values. Regression models fitted to experimental results showed good correlation coefficients for extrudate properties (Table A.4). Regression analyses showed that percent starch gelatinization and starch digestibility of samples were significantly affected ($p<0.01$) by linear terms of component variables ($x_1$, $x_2$, $x_3$), and their interaction terms ($x_1x_2$, $x_1x_3$, $x_2x_3$). The negative linear coefficient of PDHF and fruit waste content contributed the decrease of both percent starch gelatinization and starch digestibility, whereas the positive linear coefficient of durum clear flour had reverse trend on both parameters. The effects of process variables on measured parameters showed significant interaction ($p<0.1$ and $p<0.05$) with component variables used in this study. This interaction terms contribute that the effects of process variables on percent starch gelatinization and starch digestibility was dependent on the durum clear flour, PDHF and fruit waste content.

The starch digestibility of raw materials was measured as 0.5, 5.0, 28.5 and 69.2 mg maltose/g sample (d.b.) for fruit waste blend, PDHF, durum clear flour and rice grit, respectively. Extrusion cooking significantly ($p<0.01$) increased starch digestibility of extrudates when compared to the unextruded raw materials. These results are in agreement with other studies where it has been reported that extrusion improves digestibility of cereals (Dahlin and Lorenz, 1993; Onyango et al., 2004; Hagenimana, et al., 2006). Extrusion processing usually results in a more complete gelatinization and disintegration of starch granules (Svihus et al., 2005). The rupture of starch granules during extrusion, making the substrate more accessible, facilitated the amylolytic degradation due to hydration, loss of structural integrity and partial solubilization of starch molecules (Onyango et al., 2004; Hagenimana et al., 2006).

Figure 3.24 shows the change in percent starch gelatinization and starch digestibility of extruded samples with feed composition. Results indicated that increasing PDHF content caused decrease in both percent starch gelatinization and starch digestibility of the extruded samples. The increasing PDHF content may affect the extent of starch gelatinization in the extruder due to its relatively high protein content compared to rice grit and fruit waste. The addition of proteins to starches increases sites for cross-linking. Proteins can crosslink in extruder through stronger covalent and ionic bonds (Harper, 1986), thus changing the water distribution in the extruder barrel (Madeka and Kokini, 1992). This may cause reduced starch conversion during extrusion of the samples with increasing PDHF content and thus, reduced starch digestibility values. It was reported that the interactions between the protein matrix and starch may affect starch digestibility and protein digestion usually precedes starch digestion (Svihus et al., 2005).

The fruit waste content had quadratic effect on starch digestibility. Starch digestibility of samples decreased with increasing fruit waste content. On the other hand, there was no significant change in percent starch gelatinization of extruded samples with increasing fruit waste content (Figure 3.24). 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 (Svihus et al., 2005). Fruit waste was mainly composed of orange peel in which pectin is predominantly found (Larrea et al., 2005b). Non- starch polysaccharides may impair digestion both directly by reducing contact between digestive enzymes and starch, and indirectly through a reduced swelling of the starch granule and through interactions with gelatinization properties during processing of feeds (Svihus et al., 2005). Extrusion cooking is responsible from changing the extent of molecular associations between components, e.g. the amylose-lipid complex. Formation of amylose-lipid complex is reported to retard the rate of carbohydrate utilization in gut (Hagenimana et al., 2006).

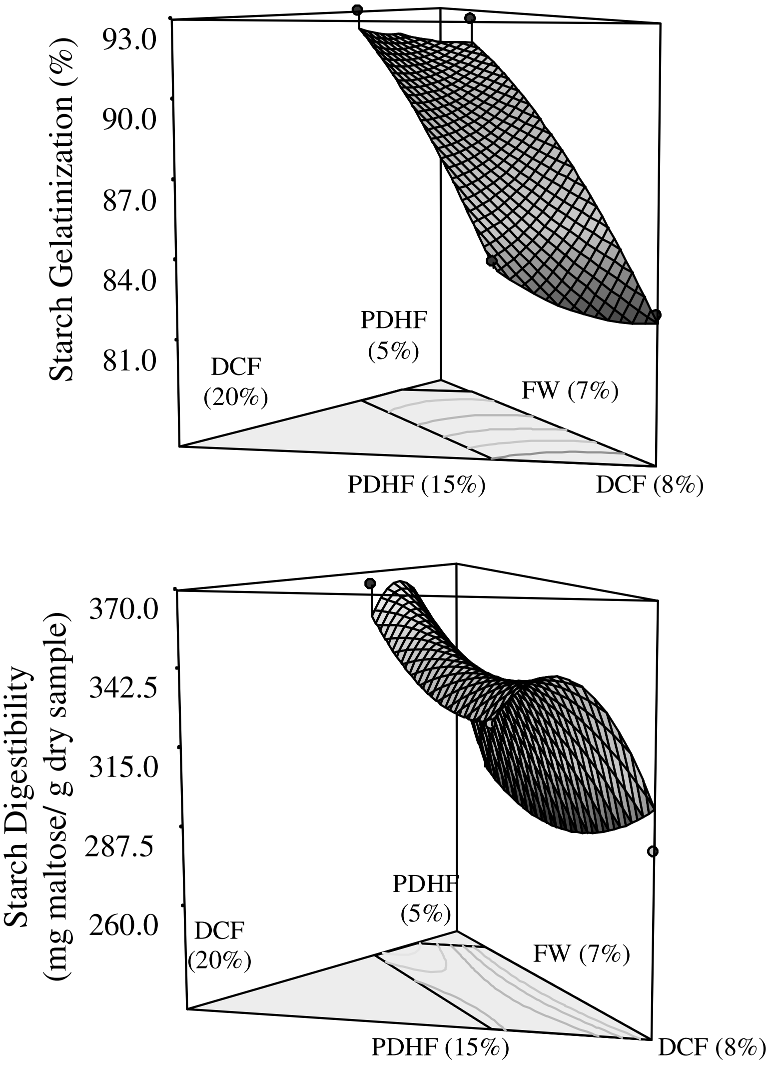
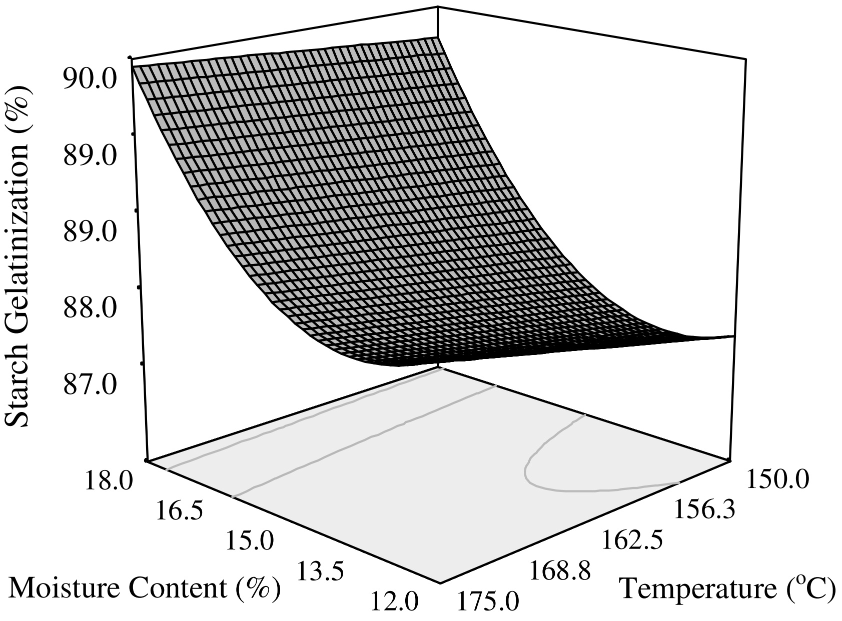


Figure 3.24 Response surface plots for percent starch gelatinization and starch digestibility as a function of feed composition at 12% feed moisture content, 200 rpm screw speed and 150°C barrel temperature (DCF, durum clear flour; PDHF, partially defatted hazelnut flour; FW, fruit waste)

During extrusion, shear force physically tears apart starch granules and allows faster transfer of water into the interior starch molecule. Moisture content in combination with temperature has a significant effect on starch conversion (Lai and Kokini, 1991). In this study, feed moisture content significantly affects the percent starch gelatinization in interaction with durum clear flour and PDHF contents ($p$<0.05) and also triple interaction with component and process variables ($p<0.01$). The quadratic term of feed moisture content in the surface model of percent starch gelatinization was also significant ($p<0.01$, Table A.4). Moisture content affected starch digestibility with significant interaction ($p<0.05$) with PDHF. The influence of barrel temperature on percent starch gelatinization was significantly dependent ($p<0.05$) on the PDHF and fruit waste contents and triple interactions of PDHF, fruit waste and moisture contents ($p<0.01$, Table A.4).

Increasing moisture content caused an increase in percent starch gelatinization and starch digestibility for most formulations of the extrudates (Figure 3.25). At high moisture content, the viscosity of the starch would be low, allowing for extensive internal mixing and uniform heating which would account for enhanced starch gelatinization (Lawton and Handerson, 1972). As seen in Figure 3.25, increasing temperature caused slight increase in the percent starch gelatinization of extruded samples. However, there was no significant effect of barrel temperature on starch digestibility of extruded samples. High temperature and high shear rate cause disruption of starch granule structure which leads to mechanical damage of starch molecules and/or gelatinization in the starch fractions (Asp and Björck, 1989). An increased percent starch gelatinization with increasing barrel temperature agrees with the results of Bahattacharya and Hanna (1987). In their study, they found that as the extruder barrel temperature increased from 155 to 164°C, the percent gelatinization increased from 73.6 to 98.4 in the waxy corn extrudates; while it increased from 40 to 55.2 in the ordinary corn extrudates. Chiang and Johnson (1977) observed that the interaction between temperature and moisture content was most important factor affecting starch conversion of wheat flour. In their research, starch gelatinization decreased slightly with increasing moisture contents (18-27%) at low temperature (65-80$^{o}$C), whereas at higher temperature (95-110$^{o}$C) it increased with increasing moisture content which was consistent with the results of this study.



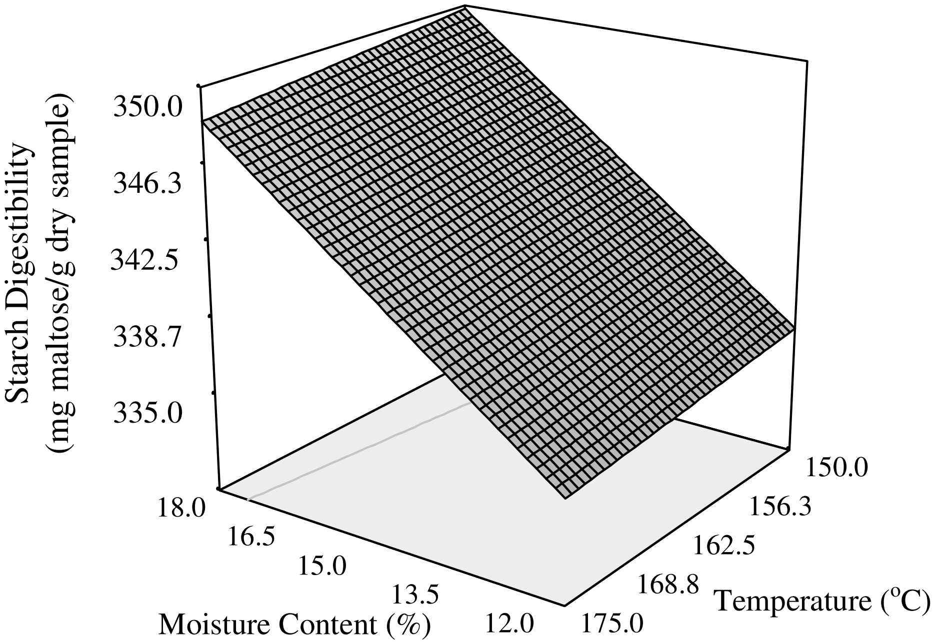


Figure 3.25 Response surface plots for percent starch gelatinization and starch digestibility as a function of moisture content and temperature (The extrusion variables studied at 15% durum clear flour, 10% PDHF and 5% fruit waste contents and 200 rpm screw speed)

The increase in percent gelatinization of starch at increased moisture content would probably results an increase in susceptibility of starch to enzyme action. Gomez and Aguilera (1983) have demonstrated increased enzyme susceptibility of whole ground corn after low moisture extrusion, presumably because of gelatinization and dextrinization or fragmentation of the polysaccharide chain. The availability of the extruded starch to digestive enzymes is reported to be related to the severity of the extrusion cooking (Hagenimana et al., 2006). Higher feed moisture content, extrusion temperature and lower screw speeds appeared to be overall recommendations for increasing carbohydrate digestibility (Dahlin and Lorenz, 1993).

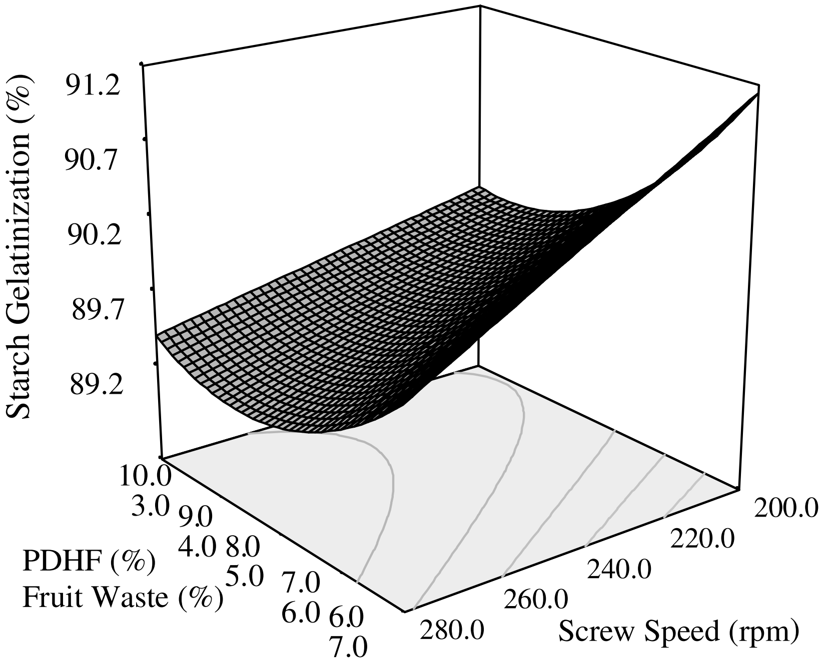


Figure 3.26 Response surface plots for percent starch gelatinization as a function of PDHF & fruit waste content and screw speed (The extrusion variables studied at 17% durum clear flour and 12% moisture contents and 150°C barrel temperature)

The influence of screw speed on the percent starch gelatinization was significantly dependent (*p*<0.01) on the PDHF and fruit waste contents and also triple interactions of durum clear flour, PDHF, fruit waste and moisture contents (*p*<0.01) as shown in Table 4. Increasing screw speed from 200 to 280 rpm caused a decrease in percent starch gelatinization values for most compositions (Figure 3.26). This decrease may be explained on the basis of low residence time in the extruder at high screw speed. A lower screw speed allows enough residence time for the gelatinization and solubilization of starch to occur (Dahlin and Lorenz, 1993). Similarly, Chiang and Johnson (1977) explained that increasing screw speed, decreased starch gelatinization due to a decreasing retention time in the extruder.

# CHAPTER 4

# CONCLUSIONS

The present study investigates production of a ready-to-eat, puffed and value-added extruded food by using the side products of various food industries. Durum clear flour of macaroni production, partially defatted hazelnut flour of hazelnut oil production and fruit wastes of various fruit juice production were evaluated as the components of the extruded food product. Response surface methodology was successful for modeling of various response variables with respect to process variables (screw speed, moisture content, and barrel temperature) and mixture components (durum clear flour, partially defatted hazelnut flour and fruit waste blend) used in this study. Proposed models approximate the response surfaces and can be used suitably for prediction at any values of the parameters within experimental range.

Extrusion of most combinations of durum clear flour, PDHF and fruit waste produced expanded extrudates at most extrusion conditions; however blends having 15% PDHF usually failed to extrudate into products with consistent shapes and degrees of expansion. The product responses were mostly dependent on PDHF content rather than fruit waste content. Increasing PDHF content caused increase in bulk density and water solubility index, but decrease in radial expansion index, porosity and water absorption index of the extruded products. Increasing PDHF content up to ~12 % improved product hardness, however beyond this value, extrudate hardness increased. The color of the product tends to turn slightly darker when the PDHF percentage increased in the formulation. The gradual increase in redness and yellowness was observed in extrudates with additions of fruit waste.

Changing process conditions affected the physical properties of extrudates. Increasing moisture content and decreasing temperature caused an increase in expansion ratio and porosity for most compositions. The influence of feed moisture content and temperature on the bulk density was significantly dependent ($p<0.05$) on the composition of the extrudates. Increasing moisture content caused a decrease in WSI and increase in WAI for many extrudate formulations. WSI and WAI decreased at higher screw speed. WSI slightly decreased with increasing temperature which had adverse effect on WAI. No significant effect of moisture content and temperature on hardness was observed for the given data. Among the experimental conditions used in the present study, well expanded extrudates with acceptable sensory attributes were obtained at low PDHF content.

The fruit wastes, defatted hazelnut flour and durum clear flour can be used in combination with rice grit for production of value-added convenient extruded snack foods due to their valuable characteristics. In extruded samples, antioxidant activity had positive correlation to the content of phenolic compounds, suggesting that the antioxidant activity of produced extrudate is due to a greater extent to their phenolic content. Percent starch gelatinization were also found to be positively correlated with the starch digestibility values.

Extrusion cooking process significantly increased starch digestibility of extrudates when compared to the unextruded raw materials. Increasing PDHF and fruit waste content caused increase in total phenolic content and antioxidant activity of the extruded samples, whereas percent starch gelatinization and starch digestibility values decreased with added PDHF. Starch digestibility of samples decreased with increasing fruit waste content. Changing process conditions affected the total phenolic content, antioxidant activity, percent starch gelatinization and starch digestibility values of produced extrudates.

The contribution of the present work is to propose an alternative technology for the utilization of food-processing wastes for the development of a useful, value-added extruded food product. The possibility to use these wastes in food formulations can represent an exciting opportunity to obtain new functional ingredients to use in an enormous variety of food preparations. More research does need to be done for further enhancing the nutritional profile and physical characteristics by modifications to the original formulation.

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# APPENDICES

Table A.1 Coefficient of variables in the predictive model for radial expansion ratio, bulk density, porosity, WAI and WSI

| Variables | Radial Expansion Ratio | Bulk Density (g/cm$^3$) | Porosity | WAI (g/g dry sample) | WSI (g/g dry sample) |
|---|---|---|---|---|---|
| $x_1$ | $1.371^{***}$ | $0.130^{***}$ | $-1.035^{***}$ | $0.133^{***}$ | $0.995^{***}$ |
| $x_2$ | $-1.919^{***}$ | $0.203^{***}$ | $0.132^{***}$ | $0.059^{***}$ | $4.311^{***}$ |
| $x_3$ | $0.834^{***}$ | $-0.668^{***}$ | $1.410^{***}$ | $0.136^{***}$ | $0.103^{***}$ |
| $x_1x_4$ | $-6.906\ 10^{-3**}$ | ns | $0.036^{***}$ | ns | $-0.044^{***}$ |
| $x_1x_5$ | ns | $-2.522\ 10^{-4**}$ | ns | ns | ns |
| $x_1x_6$ | ns | $-5.117\ 10^{-4**}$ | ns | ns | $3.083\ 10^{-3*}$ |
| $x_2x_4$ | $0.052^{**}$ | $-0.014^{***}$ | ns | $-2.727\ 10^{-3***}$ | $-0.092^{***}$ |
| $x_3x_4$ | $-0.047^{**}$ | ns | ns | ns | $0.036^{***}$ |
| $x_2x_5$ | $1.04110^{-3*}$ | $-9.044\ 10\text{-}5^{**}$ | $5.617\ 10^{-4*}$ | ns | ns |
| $x_2x_6$ | $6.10110^{-3***}$ | $-7.183\ 10^{-4***}$ | $-3.986\ 10^{-3***}$ | ns | ns |
| $x_3x_4$ | ns | ns | $-0.135^{*}$ | ns | ns |
| $x_1x_4x_5$ | ns | $4.850\ 10^{-6***}$ | ns | ns | ns |
| $x_1x_5x_6$ | ns | $1.208\ 10^{-6**}$ | ns | ns | ns |
| $x_2x_5x_6$ | ns | $1.204\ 10^{-6**}$ | ns | ns | ns |
| $x_3x_4x_6$ | ns | $-6.034\ 10^{-5**}$ | $3.436\ 10^{-4***}$ | ns | ns |
| $x_3x_5x_6$ | ns | $-8.118\ 10^{-6**}$ | $1.363\ 10^{-5*}$ | ns | ns |
| $x_1x_4^2$ | ns | $4.311\ 10^{-4**}$ | ns | ns | ns |
| $x_2x_6^2$ | ns | ns | $2.264\ 10^{-5*}$ | ns | ns |
| $x_3x_4^2$ | ns | $-6.585\ 10^{-4***}$ | $2.71310^{-3***}$ | ns | ns |
| Model (*F*-valu*e*) | $8.58^{\S}$ | $10.16^{\S}$ | $12.38^{\S}$ | $12.90^{\S}$ | $16.54^{\S}$ |
| $r^2$ | 0.686 | 0.802 | 0.789 | 0.710 | 0.758 |
| Adjusted $r^2$ | 0.606 | 0.715 | 0.725 | 0.655 | 0.712 |
| Predicted $r^2$ | 0.502 | 0.632 | 0.680 | 0.584 | 0.651 |
| *Lack of Fit* (*p*-value)$^{\S\S}$ | 0.7820 | 0.6120 | 0.0676 | 0.5903 | 0.9607 |

$x_1$, Durum Clear Flour (%); $x_2$, PDHF (%); $x_3$, Fruit Waste (%); $x_4$, Moisture Content (%); $x_5$, Screw Speed (rpm); $x_6$, Temperature (°C)
*significant at $p<0.1$; **significant at $p<0.05$; ***significant at $p<0.01$; ns, nonsignificant; § significant at a $p<0.0001$ level
§§ Want the selected model to have non-significant lack of fit ($p>0.05$)

Table A.2 Coefficient of variables in the predictive model for hardness, total area and color

| Variables | L | a | b | Hardness (N) | Total Area (Ns) |
|---|---|---|---|---|---|
| $x_1$ | 8.745$^{***}$ | 0.419$^{***}$ | 0.529$^{***}$ | -1.687$^{***}$ | -3.540$^{***}$ |
| $x_2$ | -8.691$^{***}$ | -1.808$^{***}$ | 0.547$^{***}$ | 2.650$^{***}$ | -4.425$^{***}$ |
| $x_3$ | -3.052$^{***}$ | 7.130$^{***}$ | -0.721$^{***}$ | -71.846$^{***}$ | -287.74$^{***}$ |
| $x_1x_2$ | ns | ns | -0.0132 | -0.343$^{***}$ | ns |
| $x_1x_3$ | 0.278$^{***}$ | ns | 0.0724 | 3.602$^{***}$ | 13.515$^{*}$ |
| $x_1x_6$ | -0.0217$^{*}$ | ns | ns | ns | ns |
| $x_2x_3$ | 0.279$^{***}$ | ns | 0.0593 | 3.175$^{**}$ | 14.355$^{**}$ |
| $x_2x_4$ | 0.222$^{***}$ | 0.122$^{**}$ | ns | ns | ns |
| $x_3x_4$ | ns | ns | ns | ns | 6.813$^{**}$ |
| $x_2x_5$ | ns | 5.725 $10^{-3*}$ | ns | ns | ns |
| $x_2x_6$ | 0.0231$^{**}$ | 4.274 $10^{-3***}$ | ns | ns | ns |
| $x_3x_5$ | 0.0195$^{**}$ | ns | ns | 0.338$^{***}$ | 0.859$^{***}$ |
| $x_2x_4x_6$ | ns | -2.137 $10^{-4***}$ | ns | ns | ns |
| $x_2x_5x_6$ | -9.500 $10^{-5***}$ | ns | ns | ns | ns |
| $x_1x_3x_5$ | ns | ns | ns | -0.0166$^{***}$ | -0.041$^{***}$ |
| $x_2x_3x_5$ | ns | ns | ns | -0.0151$^{**}$ | -0.040$^{***}$ |
| $x_1x_4^2$ | ns | 1.368 $10^{-3**}$ | ns | ns | ns |
| $x_1x_5^2$ | -7.963 $10^{-5***}$ | -1.559 $10^{-5***}$ | ns | ns | ns |
| $x_2x_4^2$ | 7.855 $10^{-3**}$ | ns | ns | ns | ns |
| $x_2x_5^2$ | ns | -1.167 $10^{-5***}$ | ns | ns | ns |
| $x_3x_5^2$ | ns | 1.186 $10^{-4**}$ | ns | ns | ns |
| Model (*F*-value) | 27.11$^{§}$ | 8.18$^{§}$ | 35.09$^{§}$ | 7.38$^{§}$ | 10.72$^{§}$ |
| $r^2$ | 0.862 | 0.655 | 0.732 | 0.583 | 0.764 |
| Adjusted $r^2$ | 0.831 | 0.574 | 0.711 | 0.504 | 0.692 |
| Predicted $r^2$ | 0.783 | 0.523 | 0.680 | 0.456 | 0.595 |
| *Lack of Fit (p*-value*)*$^{§§}$ | 0.1650 | 0.7622 | 0.1124 | 0.1195 | 0.2796 |

$x_1$, Durum Clear Flour (%); $x_2$, PDHF (%); $x_3$, Fruit Waste (%); $x_{4,}$ Moisture Content (%); $x_5$, Screw Speed (rpm); $x_6$, Temperature (°C)
*significant at $p<0.1$; **significant at $p<0.05$; ***significant at $p<0.01$; ns, nonsignificant; § significant at a $p<0.0001$ level
§§ Want the selected model to have non-significant lack of fit ($p>0.05$)

Table A.3 Correlation coefficients between physical properties of extrudates

| | Radial Expansion Ratio | Bulk Density | Porosity | WAI | WSI |
|---|---|---|---|---|---|
| Radial Expansion Ratio | 1 | 0.018 | -0.051 | -0.058 | 0.189 |
| Bulk Density | | 1 | -0.692** | -0.252* | 0.239* |
| Porosity | | | 1 | 0.677** | -0.722** |
| WAI | | | | 1 | -0.858* |
| WSI | | | | | 1 |

*significant at *p*<0.05, **significant at *p*<0.01

Table A.4 Coefficient of variables in the predictive model for functional properties

| Variables | Total Phenolic Content (mg ferulic acid/g dry sample) | Antioxidant Activity (%) | Starch Gelatinization (%) | Starch Digestibility (mg maltose/ g dry sample) |
|---|---|---|---|---|
| $x_1$ | -0.247$^{***}$ | -1.404$^{***}$ | 9.850$^{***}$ | 10.555$^{***}$ |
| $x_2$ | 1.242$^{***}$ | 4.889$^{***}$ | -11.612$^{***}$ | -37.878$^{***}$ |
| $x_3$ | 0.171$^{***}$ | 1.497$^{***}$ | -10.636$^{***}$ | -187.120$^{***}$ |
| $x_1x_2$ | ns | ns | 0.062$^{***}$ | 3.241$^{***}$ |
| $x_1x_3$ | ns | ns | -0.254$^{**}$ | 5.492$^{***}$ |
| $x_1x_4$ | 3.010 $10^{-3***}$ | 8.093 $10^{-3\ ***}$ | -0.587$^{**}$ | ns |
| $x_1x_6$ | ns | ns | ns | -0.013$^{*}$ |
| $x_2x_3$ | ns | ns | 2.455$^{*}$ | 12.260$^{***}$ |
| $x_2x_4$ | -0.028$^{**}$ | ns | 1.143$^{***}$ | -0.049$^{**}$ |
| $x_2x_5$ | -9.337 $10^{-4**}$ | ns | 0.013$^{***}$ | ns |
| $x_2x_6$ | ns | ns | 0.061$^{**}$ | 0.319$^{*}$ |
| $x_3x_4$ | ns | -0.025$^{*}$ | ns | ns |
| $x_3x_5$ | ns | ns | 0.023$^{***}$ | ns |
| $x_3x_6$ | ns | ns | 0.049$^{***}$ | ns |
| $x_1x_2x_4$ | ns | ns | -0.015$^{***}$ | ns |
| $x_1x_2x_5$ | ns | ns | 8.134 $10^{-4***}$ | ns |
| $x_1x_2x_6$ | ns | ns | ns | -0.029$^{**}$ |
| $x_2x_3x_5$ | ns | ns | -3.486 $10^{-3***}$ | ns |
| $x_2x_3x_6$ | ns | ns | -0.010$^{***}$ | ns |
| $x_3x_4x_5$ | ns | ns | -1.595 $10^{-3***}$ | ns |
| $x_3x_4x_6$ | ns | ns | -4.591 $10^{-3***}$ | ns |
| $x_3x_4^2$ | ns | ns | 8.367 $10^{-3***}$ | ns |
| Model (*F*-valu*e*) | 30.09$^{§}$ | 13.498$^{§}$ | 36.67$^{§}$ | 9.88$^{§}$ |
| $r^2$ | 0.850 | 0.719 | 0.988 | 0.831 |
| Adjusted $r^2$ | 0.826 | 0.665 | 0.980 | 0.747 |
| Predicted $r^2$ | 0.781 | 0.614 | 0.953 | 0.602 |
| *Lack of Fit (p*-value*)*$^{§§}$ | 0.0551 | 0.1192 | 0.0653 | 0.4426 |

$x_1$, Durum Clear Flour (%); $x_2$, PDHF (%); $x_3$, Fruit Waste (%); $x_4$, Moisture Content (%); $x_5$, Screw Speed (rpm); $x_6$, Temperature (°C)
*significant at $p<0.1$; **significant at $p<0.05$; ***significant at $p<0.01$; ns, nonsignificant; § significant at a $p<0.0001$ level
§§ Want the selected model to have non-significant lack of fit ($p>0.05$)

Appendix 1.

## Sensory Evaluation Forms

## (A)

Dear Panelists,

You will receive samples in random order. Please rank the samples for the following sensory properties in the order from low to high sample number. Please rinse your mouth after tasting each sample.

**Taste**

Orange flavor

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |

Hazelnut Flavor

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |

Bitterness

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |

Off-Flavor

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |

Appendix 1.continued

(B)

**Appearance**

Color

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | | | | | | | |
| 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |

Air Cell Homogeneity

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | | | | | | | |
| 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |

**Texture**

Hardness

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | | | | | | | |
| 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |

Crispness

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | | | | | | | |
| 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |

Breakability

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | | | | | | | |
| 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |

**Overall Acceptability**

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | | | | | | | |
| 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |

Appendix 1.continued

## Definition of some terms used in the sensory evaluation

**Air Cell Homogeneity**: Homogeneity of air cell size in the sample.

**Hardness:** Force necessary for breakdown of food between molar teeth

**Crispness:** Level of noise during biting

**Breakability:** Force necessary for crumbling and breaking into smaller size

**Evaluation for (A)**
1-Absent
5- Moderate
9-Very high

**Evaluation for (B)**
1- Dislike extremely
2- Dislike
3- Dislike moderately
4- Dislike slightly
5- Neither like nor Dislike
6- Like slightly
7- Like moderately
8- Like
9- Like extremely

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## PUBLICATIONS

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