Extrusion processing of protein rich food formulations

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ABSTRACT

Extrusion has been widely used as a high-temperature short-time process to produce commercially shelf stable extruded products. Many physical and chemical changes take place during the process, including the gelatinization of starch, denaturation of protein and even complete cooking. To fully understand changes during the process, evaluation of the effect of extrusion process variables on the extruded product is very important. There are many process and product-dependent variables associated with the extrusion process such as barrel temperature, screw speed, die diameter and raw material composition (moisture, starch, protein and fat contents). In general, commercial extruded products have mainly focused on starch-rich products which are generally are low in protein content. The overall objective of this research was to prepare high-value protein-rich products through the use of extrusion processing.

In order to evaluate the influence of added protein [soy protein isolate, (SPI)] to a corn-based system, a two step procedure was employed. Firstly, the effect of feed moisture, screw speed and barrel temperature on physical properties of extruded corn flour and SPI blends was evaluated to generate a basic understanding of the influence of operational parameters. This was expanded to include higher protein levels in the subsequent study. The physical properties of the extruded material considered were expansion ratio, bulk density, breaking strength, water solubility index, rehydration ratio and color. All these properties were significantly ($P \leq 0.05$) affected by the process variables. An optimization study was performed to determine optimum variable levels to achieve desirable properties of extruded product within selected constraints.

As residence time distribution (RTD) is an important aspect of the extrusion process. The RTD of SPI and corn flour mixtures was studied under different screw speeds (75, 100 and 125 rpm), raw material moisture (25, 30 and 35%) and die diameter (3 and 5 mm) configurations. Two conventional flow models served to represent the RTD patterns in the extruder: the frequency model (F distribution) and the cumulative RTD model (E-distribution). The parameters of these models – the half concentration internal age and

particle accumulation rate – were determined by a nonlinear regression. These models' parameters were found to be responsive to process variables, and both F and E distributions were well predicted.

As extruded products produced under the above conditions remained high in moisture content and water activity, in order to achieve shelf stability it was necessary to lower their moisture and water activity levels. The effect of extrusion process variables on the drying behavior of the product was studied next. Since there were many test samples, a simple drying set-up operating under moderate temperature (55 °C), humidity and airflow conditions was used. The extrusion process variables were found to significantly ($P \leq 0.05$) affect the drying behaviour of the product. Models were developed to predict drying times to reduce the product moisture to stable levels (water activity below 0.75).

Selected extrusion products with 50% protein content were subjected to frying at different temperatures (145°C, 165°C, 185°C) and for different durations (0 to 660 s). The resultant products' physical characteristics, including breaking strength, oil uptake, color and moisture content were evaluated, and a sensory test was performed to describe the acceptability of the products. Frying conditions which yielded products of acceptable quality were identified.

Overall, the research contributes to a better understanding of the extrusion process of high SPI content corn flour blends. Together with post extrusion treatments including drying and frying, the process can produce good quality protein-rich extrusion products for use in further preparations or as a fried snack.

RÉSUMÉ

Processus à haute température et de courte durée, l'extrusion permet la production de produits d'extrusion comestibles à longue vie commercial. Durant ce processus il survient des changements incluant la gélatinisation de l'amidon, la dénaturation des protéines, ainsi qu'une cuisson uniforme et complète. Pour bien maîtriser ces changements, une évaluation de l'effet des variables du processus d'extrusion sur l'extrudat est de rigueur. Plusieurs variables, soit la température du fourreau, la vitesse de la vis, le diamètre de la filière et la composition de la matière première (teneur en eau, en amidon, en protéines et en gras), sont liées au processus ainsi qu'au produit. Les produits commerciaux extrudés demeurent riche en amidon, mais pauvre en protéines. La présente recherche visa à préparer, par l'entremise d'une transformation par extrusion, des produits de haute valeur, riches en protéines.

Afin d'évaluer l'influence d'un ajout de protéine [isolats de protéine de soya, (IPS)] à un système à base de maïs, un processus à deux étapes fut étudiés. L'effet de la teneur en eau du matériel, de la vitesse de la vis et de la température du fourreau sur les propriétés physiques d'extrudats d'un mélange d'ISP et de farine de maïs furent évalués, pour évaluer l'influence des paramètres opérationnels. L'inclusion de teneurs en protéine plus élevés suivi. Les propriétés physiques de l'extrudat considérés furent le taux de foisonnement, la densité apparente, la résistance à la rupture, l'indice de solubilité dans l'eau, le taux de réhydratation, et la couleur. Toutes celles-ci furent influencées ($P \leq 0.05$) par les variables de transformation. Une optimisation des variables de transformation pour obtenir un extrudat aux propriétés voulues sous certaines contraintes d'opération suivit.

La distribution temps séjour (DTS) est un important aspect du processus d'extrusion. La DTS de mélanges d'IPS et de farine de maïs fut déterminée sous différentes vitesses de vis (75, 100 ou 125 rpm), teneurs en eau du matériel brut (25, 30 ou 35%) et diamètre de la filière (3 ou 5 mm). Deux modélisations conventionnelles du débit, l'une liée à la fréquence (distribution F) et l'autre cumulative (distribution E), servirent à représenter le

cours du DTS dans l'extrudeur. L'âge interne à mi-concentration et le taux d'accumulation de particules, déterminés par régression non-linéaire, répondirent bien aux variables de transformation, les distributions E et F étant prédites avec exactitude.

Comme ces extrudats maintinrent une teneur et une activité en eau élevée, il fut nécessaire, afin d'obtenir une bonne stabilité sur les tablettes, de diminuer ces derniers. Un étude sur l'effet des variable du processus d'extrusion sur le séchage subséquent de l'extrudat fit suite. Étant donné le grand nombre d'échantillons, un simple appareillage de séchage, fonctionnant à de températures, taux d'humidités et flux d'air moyens, fut utilisé. Les variables du processus d'extrusion influencèrent ($P \leq 0.05$) le séchage du produit. Des modélisations furent développées afin de prédire le temps nécessaire pour réduire la teneur en eau du produit à un niveau stable (activité de l'eau en deçà de 0.75).

Des extrudats d'une teneur en protéine de 50% furent frits à des températures de 145^{o}C, 165^{o}C, et 185^{o}C pour 0 à 660 s. La résistance à la rupture, l'absorption d'huile, la couleur et la teneur en eau des produits frits furent évalués. Un test organoleptique évalua l'acceptabilité des produits. Les conditions de friture donnant une qualité acceptable furent identifiées.

Ces études contribuèrent à une meilleure compréhension du processus d'extrusion de mélanges de farine de maïs à haute teneur en ISP. Apparié aux traitements post-extrusion de séchage ou de friture, le processus permet de produire des extrudats de qualité à haute teneur en protéines pouvant passer par une étape de préparation additionnelle ou être consommées directement comme croustille frite.

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CONTRIBUTIONS OF AUTHORS

Several presentations have been made based on the thesis research and some manuscripts have been prepared for publication. Different authors have been involved in some parts of the thesis work and they have been appropriately included in manuscripts either submitted or prepared for submission and their contributions to the work are as follows or in presentations:

Liang Yu is the PhD candidate who planned and conducted all the experiments, in consultation with his supervisor, gathered and analyzed the results, and prepared the first draft of all manuscripts for scientific publications.

Dr. Hosahalli S. Ramaswamy is the thesis supervisor, under whose guidance the research work was carried out, and who assisted the candidate in planning and conducting the research as well as in correcting, editing, reviewing and processing the manuscripts for publication.

Dr. Joyce Boye provided technical and operational guidance during the early part of the studies especially in the first part involved with evaluation of the extruder and residence time distribution studies.

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LIST OF PUBLICATIONS AND PRESENTATIONS

Part of this thesis has been prepared as manuscripts for publications:

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NOMENCLATURE

ANOVA	Analysis of variance
a_w	Water activity
B	Particle accumulation rate
BD	Bulk density
BS	Breaking stress
C	Particle concentration
CCRD	Central composite rotatable design
db	Dry base
D	Die diameter
EC	Emulsifying capacity
ECT	Total Extrudate Collection Time
ER	Expansion ratio
E(t)	Exit age distribution or RTD
E(θ)	Equivalent of E(t) in a normalized form
FPRT	Fastest particle residence time
F(t)	Cumulative RTD function
L	Lightness
Mc	Half-concentration internal age
MR	Moisture ratio
OAC	oil absorption capacity
QDA	Quantitative Descriptive Analysis
QRT	Quality Rating Test
R^2	Coefficient of determination
RR	Rehydration ratio
RSM	Response surface methodology
S	Speed
SPI	Soy protein isolate
t	Time
t_m	Mean residence time
t_v	Mean Variance

T	**Temperature**
M	**Moisture content**
WSI	**Water solubility index**
wb	**wet base**
WAI	**Water absorption index,**
WAC	**Water absorption capacity**
θ	**Normalized time** (t/t_m)
σ	**Variance**

CHAPTER 1 GENERAL INTRODUCTION

Introduction

Extrusion technology has been commercially prectised in the food industry for a long time (Harper, 1978; Rossen and Miller, 1973). Starch-based food materials, like corn, rice, and semolina, are ideal candidates for extrusion processing (Chinnaswamy and Hanna, 1988). As a high-temperature short-time process, extrusion involves simultaneous thermal and pressure treatment along with mechanical shearing. This can result in several changes in the extrudate, including the gelatinization of starch, denaturation of protein and even complete cooking, thereby resulting in a final product that may be ready to eat. Investigated in a number of studies (Gomez and Aguilera, 1983, 1984; Bhattacharya and Hanna, 1987), extrusion processing of corn and oat meal has been successfully commercialized, especially in the production of breakfast cereals. Rich in carbohydrates and fibre, extruded corn products are, however, relatively low in protein content. Consequently there is an increasing interest in enhancing the protein component of these products (Muhungu *et al.*, 1999; Konstance *et al.*, 1998).

Soybean is a low-cost, high quality protein source available worldwide. Besides protein fortification, soybean-supplemented foods also provide many other nutritional benefits. Soy-containing foods have been shown to reduce risks of breast and other cancers and to protect against cardiovascular disease and osteoporosis (Messina and Messina, 1991; Caragay, 1992; Potter, 1995). Soy protein is an edible high value nutritive component of the soybean. It is produced from raw whole soybeans by a multi-step process that removes the lipid and indigestible components to concentrate the protein and increase its availability. Depending on the particular steps used during processing, soy protein ingredients may take the form of isolated soy protein (ISP), soy protein concentrate, or soy flour. Each ingredient may be further processed into texturized soy protein or texturized vegetable protein (TVP) used in the manufacture of meat and poultry analogues by thermoplastic extrusion or steam texturization to impart structure and shape. In addition to protein, these soy protein ingredients contain other naturally occurring soy constituents, such as isoflavones, fiber, and saponins. The different processing steps used ultimately define the extent of retention of naturally occurring components in the final product. Soy protein is also consumed as a component of traditional fermented and non-fermented soy foods, such as tofu, tempeh, and miso, as well as whole soybeans, soynuts, soymilk, soy yogurt, and soy cheese. These products contain variable amounts of soy protein and other naturally occurring soy constituents depending on the specific technologies used in their production. Soy protein ingredients and soy protein–containing foods may partially replace or supplement animal or other vegetable protein sources in the human diet.

Some studies have investigated the extrusion process of corn-soy flour blends. Konstance *et al.* (2002) studied drying properties and storage stability of a high energy extruded food consisting of blends of full fat soy grits, corn meal, soy concentrate and soybean oil. Sun and Muthukumarappan (2002) studied the effects of soy flour content, feed moisture, screw speed and barrel temperature on physical properties of extruded products from a single-screw extruder. Park *et al.* (1993) studied the effects of feed moisture, corn starch levels and process temperature on selected physical properties of soy flour-corn starch-raw beef extruded products generated with a single-screw extruder.

With the rapid development of the soybean processing industry, soy protein isolates (SPI) have become widely available and are commonly used in the food industry. Compared with soy flour, SPI can provide highly concentrated protein, which is high in lysine, bears a bland flavor and is decreased in flatulence factors and sugars, thus improving overall product quality (Konstance *et al.*, 1998). Some studies have explored the use of soybean in this form. Camire and Clay (1991) incorporated soy fibre to SPI and corn flour blends to enhance the nutritional value of the extrudate. Ghorpade *et al.* (1997) studied the pore structure of the extrudate from corn starch and SPI blends. Konstance *et al.* (1998) developed a nutritious “instant” extruded product using corn meal, soy flakes, soy protein concentrate and soy oil. Faller *et al.* (1999) conducted consumer testing of extruded SPI and corn flour blends. Faller *et al.* (2000) added sugar to SPI and corn flour blends and studied its effects on the characteristic soy flavor and sensory characteristics of the extrudate. Seker (2005) evaluated the expansion ratio (ER) of extruded products bearing different SPI-corn starch blends. More recently, Brnčić *et al.* (2009) studied the influence of various whey protein concentrate additions and some extrusion process parameters on the physicochemical properties of directly-expanded corn-flour extruded products manufactured issued from a twin-screw co-rotating extruder. They found the lowest water solubility and highest water absorption index were associated with the greatest protein additions. During maize and wheat flours processing, Arhaliass *et al.* (2009) evaluated the expansion mechanism in the extrusion cooking process of a twin-screw extruder. They found that the maximum expansion was obtained with maize flour, and that the ratio of maize and wheat flours had an impact on the expansion mechanism. Since the extrusion process is an extremely complex process, and there are many variables that will affect the final product, studies with different variables, different processing conditions and extruder configuration adds value and knowledge for the efficient and optimized use of extrusion processing technology.

High temperature short time, extrusion processing of SPI enriched in corn flour blends at high levels has not been well studied especially in terms of the relationships between process parameters and the physical properties of the extruded product and no major optimization considerations have been given. Considering the popularity of the application of corn and SPI in food extrusion, such a study is considered essential for product development and process control.

The objectives of the present work were therefore as follows:

1) Evaluate the influence of extrusion process variables on physical properties of extruded corn flour and SPI blends obtained using a twin-screw extrusion process, using RSM to understand the nature of the process and identify system variables.
2) Evaluate the residence time distribution (RTD) of corn flour and SPI blends in the twin-screw extruder.
3) Evaluation and functionality linked optimization of extrusion process for preparing protein rich extruded products from soy protein isolate-corn flour blends.
4) Study the drying behavior and moisture behavior of the corn flour and SPI blends extruded products.
5) Explore the preparation of other products based on extruded protein dense corn-SPI formulations.

CHAPTER 2 REVIEW OF LITERATURE

2.1 Food Extrusion

Extrusion is defined as "shaping by force through a specially designed opening often after previous heating of the material" (Harper, 1981). Extrusion is the continuous formation of semi-solid materials through a die. Several types of extruders are available in the market including ram or piston types and screw or worm types. Extrusion cooking combines the heating of food products with the act of extrusion to create a cooked and shaped food product. It is a process in which moistened, starchy, proteinaceous foods are cooked and worked into viscous, plastic-like dough. The results of cooking the food ingredients during extrusion are: gelatinization of starch, denaturation of protein, inactivation of raw food enzymes, destruction of naturally-occurring toxic substances, diminishing of microbial counts in the final product, etc. Upon discharge through the die, the hot, plastic extruded product expands rapidly with loss of moisture and heat because of the sudden decrease in pressure. After expansion cooling, and drying, the extruded product develops a rigid structure and maintains a porous texture. Advantages of food extrusion are versatility, high productivity, low cost, ability to shape the product, high product quality, energy efficiency, production of new foods, and no effluents or waste.

Extrusion cooking has been used in a large number of food applications as it has some unique positive features compared with other heat processes. Nowadays, the food extruder is considered as a high-temperature short-time bioreactor that transforms raw ingredients into a variety of modified intermediate and finished products. During the extrusion process, the material is treated not only by heating, but also by intense mechanical shearing, compression and torque, which are able to break the covalent bonds in biopolymers (Singh *et al.*, 2007). Thus, the functional properties of the food ingredients are rapidly modified due to the combined influence of temperature, pressure, shear and time (Carvalho and Mitchell, 2000). Food extrusion also permits the inactivate the undesirable enzymes that can affect the quality and eliminate several anti-nutritional factors, such as trypsin inhibitors, haemagglutinins, tannins and phytates (Bhandari *et al.*, 2001; Singh *et al.*, 2007).

Corn and wheat are widely consumed throughout the world as they are a good source of starch and several micronutrients, such as vitamin B_1, vitamin B_5, folate (vitamin B_9), dietary fiber, vitamin C, phosphorus and manganese. Widely used in making breakfast cereals and snacks through extrusion processes, corn provides products that present an attractive crunchy texture, golden color and desirable flavor, but are limited in protein content. Soybeans contain all three macro-nutrients required for a good diet (protein, carbohydrate and fat) and also contain high amounts of vitamins and minerals (National Soybean Research Library, 2007). Soy protein isolates (SPI), consisting of 90% soy protein, is widely used in the food industry as a protein source and is also available as a nutritional supplement in various forms. Incorporating SPI into corn flour significantly increases the nutritive value and quality characteristics of the extruded end product (Harper, 1989).

2.2 Introduction of Food Extruders

"A food extruder is a device that expedites the shaping and restructuring process of food ingredients" (Riaz, 2000). An extruder is a highly versatile machine that can be applied to different food processes. Nowadays, extrusion is widely used in cooking, forming, mixing, texturizing, and shaping food products under conditions that favor quality retention, high productivity, and low cost. In the past few years, the use of extruders have expanded rapidly in the world's food and feed industries.

Different types of extruders are available in the market: segmented screw/barrel single-screw 'wet' extruders, dry extruders, interrupted-flight screw extruders, single-screw extruders, and twin-screw extruders (Riaz, 2000; Guy, 2001).

2.2.1 Segmented screw/barrel single-screw 'wet' extruders

Segmented screw/barrel single-screw extruders are the most widely applied cooking extrusion design in the food-, pet food- and feed-processing industries (Guy, 2001). 'Wet' extruders are those equipped with barrels into which steam and water can be injected during processing. These machines are normally equipped with heating and cooling jackets. The process extrudate generally contains greater moisture than that obtained with other extruder types, resulting in products which range from fully cooked, light density corn snacks, to dense, partially cooked pastas (Rokey, 2000).

Making pasta using solid screws, the first major application of this low-shear, low-temperature forming process system, was developed in the 1920s and 1930s, and remains a standard process (Huber, 2000). Several new developments in the single-screw extruder have further increased its efficiency and versatility. A brief list of products made by single-screw extruders include: (i) direct expanded corn snacks, (ii) texturized vegetable protein, (iii) ready-to-eat breakfast cereal, (iv) production of full fat soy, (v) pet foods, (vi) floating and sinking aquatic feed, (vii) production of baby foods, (viii) rice bran stabilization, (ix) precooked or thermally modified starches, flours and grain, and (x) breading (Guy, 2001).

2.2.2 Dry extruders

The term 'dry' extrusion means that this type of extruder does not require an external source of heat or steam for injection or jacket heating, and all-product heating is accomplished by mechanical friction (Said, 2000). The dry extruder was initially developed for processing whole soybeans on the farm. Ingredients which present a wide moisture range, i.e. 10-40%, can be processed in this type of machine. Moisture loss in the form of steam occurs when the products exit the die. Some dry extruders have the option of water injection during extrusion. Usually starchy material requires some moisture in order to gelatinize. Dry extruders are single-screw extruders with screw segments and steam locks (choke plates) on the shaft for greater shear and heat creation. When material moves through the barrel, and comes up against these restrictions, it is unable to pass through, the pressure increases, and a back flow is created (Guy, 2001).

There is no basic difference between the 'wet' and 'dry' extruders, except that more shear occurs in dry extruders to create heat. In dry extrusion, pressure and temperature are at their maximum just before leaving the die. The die design and opening also play a very important role in pressure build-up. The cooking range in a dry extruder can be 82–160ºC (180–320ºF) with very high pressure. As soon as the material exits the extruder dies, the pressure is instantaneously released from products, causing their internal moisture to vaporize into steam, leading them to simultaneously expand and become sterilized.

A major use of the dry extruder is in preparing oilseeds for screw pressing of oil – primarily soybeans and cottonseed, although they have been applied to sunflower, peanut and canola seed processing. This process is used around the world for processing raw soybeans into full fat soybeans and partially defatted soybean meal. Dry extrusion can also precede the production of different cereal grain fractions or other starchy raw materials (Guy, 2001).

2.2.3 Interrupted-flight screw extruders

Interrupted-flight extruders (also called ‘expanders’) were developed and introduced by the Anderson International Company (Cleveland, Ohio) in the latter 1950s as the ‘Anderson Grain Expander,’ and serve in processing pet foods and other cereal products.

An expander is mechanically different from other extruders, because it is derived from a screw press. Screw presses and interrupted flight extruders are similar in that a revolving interrupted flight pushes the material through a cylindrical barrel and out through an opening at the barrel’s end.

Although extruders are often equipped with steam-heated/water-cooled jackets, commercial interrupted flight expanders are not usually jacketed and rely on direct steam injection for supplemental heat beyond that created by mechanical shear of the ingredients.

Expanders now play a major role in preparing oilseeds for solvent extraction, and special high shear heads have been developed for shearing oilseeds before screw pressing. Expanders remain in use for making pet foods and floating aquatic feeds.

2.2.4 Twin-screw extruders

Twin-screw extruders appeared in Europe over 30 years ago. Their wider adoption in North America began in the 1980s. Nowadays, twin-screw extruders have been designed to function in a wide range of applications. The advantages of twin- *vs.* single-screw extruders have led to them replacing single-screw extruder in many applications.

The term 'twin-screw' applies to extruders with two screws of equal length placed inside the same barrel (Guy, 2001). Twin-screw extruders are more complicated than single-screw extruders, but at the same time provide much more flexibility and better control. Twin-screw extruders are generally categorized according to the direction of screw rotation and to the degree to which the screws intermesh (Figure 2.1) (Vlachopoulos and Strutt, 2003)

1. Counter- rotating twin-screw
2. Co-rotating twin-screw
3. Non-intermeshing counter-rotating

The twin-screw extruder was originally developed for processing plastics. In the food industry, twin-screw extruders were widely used from the mid-1980s to the mid-1990s. Food companies began using twin-screw extruders for producing products like sticky caramels and candies that could not be made with single-screw machines. Very soon, twin-screw extruders became popular with the food manufacturers for specialized food items.

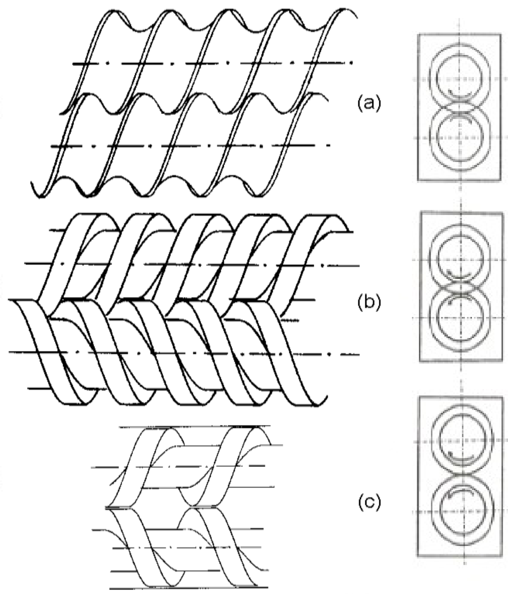


Figure 2.1 Three common types of twin-screw extruders (Vlachopoulos and Strutt, 2003)
(a) Intermeshing co-rotating;
(b) Intermeshing counter-rotating;
(c) Non-intermeshing counter-rotating.

2.2.5 Comparison of single-screw extrusion and twin-screw extrusion

Compared to single-screw extruders, twin-screw extruders are more flexible in controlling both product and process parameters. They have a flexible design permitting easy cleaning and rapid product changeover (Hauck, 1988).

Single-screw extruders are limited to a maximum fat level in the formula of 12–17%. Greater fat levels reduce friction because of their lubrication effects, thus not allowing the hardware to transform mechanical energy into heat for cooking purposes. Comparatively, fat levels in recipes for twin-screw extruders can be as high as 18–22% while still maintaining the required mechanical energy (Guy, 2001). Moisture is another key factor. A twin-screw extruder can process a much wider range of moisture content in the feed stock than a single-screw extruder. Typical process parameters for different extrusion processes (Table 2.1), and typical production capacities and costs of different types of extruders (Table 2.2) are compared below.

Table 2.1 Typical process parameters of different kinds of extruders (Riaz, 2000)

Process	Temp. (C)	Max. Pressure (bar)	Moisture (%)	Max. Fat (%)	Cook* (%)
Pellet press	60-100		12-18	12	15-30
Expander/ pellet press	90-130	35-40	12-18	12	20-55
Dry extrusion	110-140	40-65	12-18	12**	60-90
Wet extrusion					
Single-screw	80-140	15-30	15-35	22	80-100
Twin-screw	60-160	15-40	10-45	27	80-100

*% cook represents starch gelatinization as measured by enzyme susceptibility

** Dry extrusion successfully processes full fat soy (18-20% fat) and other ingredients where final product durability is not a concern.

Table 2.2 Typical production capacities and cost of different kinds of extruders (Riaz, 2000)

Process	Capacity range (Mg/h)	Average life major wear components (hours)	Average wear cost ($/Mg)	
			Full fat complete soy	Diet
Pellet press	2-60	2,300	N/A	1.23
Expander/ pellet press	2-40	700*	0.6**	1.14
Dry extrusion	0.5-2	1,000	1.0	1.78
Wet extrusion				
Single-screw	1-22	5,000	0.5	0.89
Twin-screw	1-14	5,000	1.85	2.01

* Reported 9,800 tons before replacement at rate of 14 tons/h

** Expander only, pellet press not used.

Processors should consider twin-screw extruders in the following situations (Riaz, 2000):

- Frequent product changeovers
- Products with high internal fat content (above 17%)
- Addition of a high level of fresh meat in the product (up to 35%)

• Uniform size and shapes
• Ultra-small product sizes (less than 1.5 mm)
• Products made with low density powder
• Special formulations

2.3 Extruder screw

As in any other extruder, the screw (Figure 2.2) has three sections: the feeding section conveys the material downstream; the compression section shears, heats and kneads the mass into a continuous dough; and the metering section shears and heats the dough further, as it delivers it to the die exit. Throughout the barrel there are grooves that prevent slippage at the walls. The screw has shallow flights of decreasing pitch as it advances forward, so the shear is gradually increased (Harper 1981).

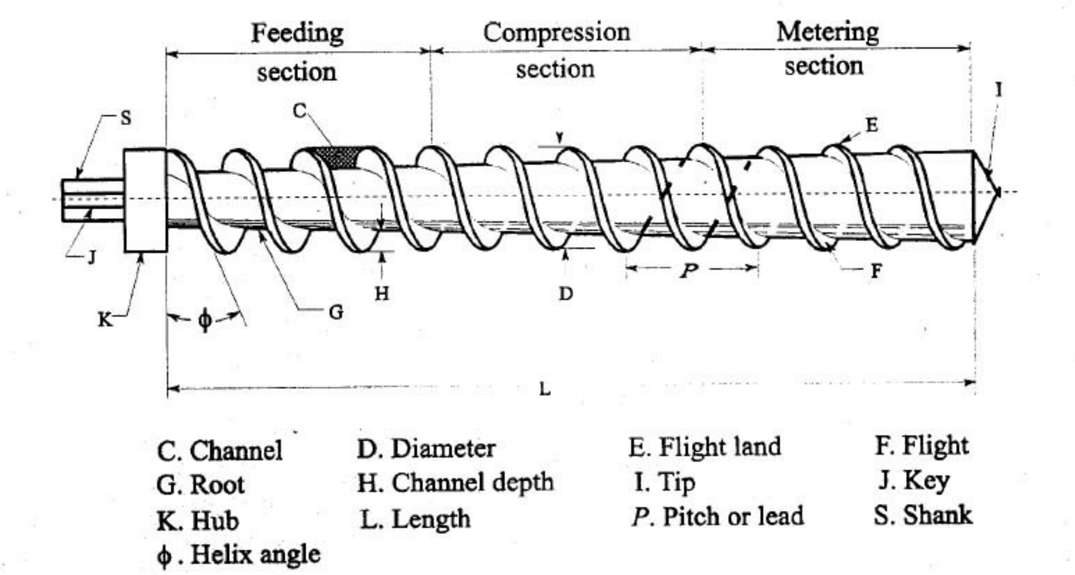


Figure 2.2 Geometric features of a single-screw (Vlachopoulos and Strutt, 2003)

2.4 Raw materials for extrusion cooking

Different kinds of materials are used in making extruded foods and feeds. While extruded foods and feeds do not differ fundamentally in their ingredients from foods and feeds made by other processes, they do differ in that they generate different product types. The extrusion process may provide the same color, flavors and the same or even improved nutritional qualities compared to other product types. The transformation of raw materials during processing is one of the most important factors that distinguish one food process and food type from another.

The selection of optimal ingredients for processing is important in extrusion cooking. Classifying different types of material helps to developing a good process. Ingredients are classified according to either their components or their functionality.

2.4.1 Classification of extrusion cooking ingredients by components

Wheat and corn flours are the most commonly used materials for food extrusion. Other materials like rice flour, soy, potato, rye, barley, oats, sorghum, cassava, tapioca, buckwheat, pea flour and other related materials can also be used. Corn meal is the most common ingredient of expanded snacks in the food market. Because of its composition, ratio of vitreous to floury endosperm, and particle size, under optimal extruding conditions corn meal makes for a light, highly expanded, crunchy and soft product. The basic recipe may vary widely; however, the main ingredient components which affect the characteristics of the final product are starch, water, protein, fiber, oil, additives and particle size.

2.4.1.1 Starch

Starch is the main component of the final product; it provides the underlying structure (Guy, 2001). Starch is contained in a large variety of plant crops, such as cereals (50-80% starch), legumes (25-50% starch) and tubers (60-90% starch) (Harper, 1989).

Starch granules are gelatinized and dispersed during extrusion, resulting in the formation of a continuous phase of the melt inside the extruder. Average molecular weight is decreased, which allows for optimum formation and stability of air cells at the die exit. Both amylose and amylopectin are needed to give the best expansion characteristics (Huber, 2001).

2.4.1.2 Water

Water is an important medium in extrusion. It is needed for starch gelatinization and ingredient dispersion. In the formation of a viscous fluid, it is conveyed and cooked. Air cell creation and expansion by evaporation at the die exit also depends on the optimum moisture content of raw materials. Janssen (1978) in summarizing the principal differences between twin- and single-screw extruders concluded that the former may be more suitable for handling wet material. Moisture is always listed as a separated variable in addition to feed ingredients because it is often controlled separately in the extruder. Moisture can be added directly to the feed, injected into the barrel, or added in the form of steam to the pre-conditioner or barrel; it will also affect the temperature of the feed material (Harper, 1981).

2.4.1.3 Protein

Single- and twin-screw extrusion of protein has been the subject of several studies. In particular, textured plant protein (TPP) was once a hot topic. Textured products manufactured by this process are increasingly being sold as meat analogues in Japan (Harper, 1989) and China. Given the nutritional factor content of plant proteins, a range of different products high in plant protein are preferred by consumers. Some detail information about soy protein is listed in later part of this passage.

2.4.1.4 Fiber

Fiber has the nutritive value in food products and it has been connected with a healthy modern diet. Fibrous materials such as bran can be part of the dispersed phase of extruded products, included in the starchy continuous phase (Guy, 2001). Fiber is chemically unchanged by the extrusion process, and influences the expansion of the product (Huber, 2001). Fibrous fragments disrupt the starchy film of air cell walls, reducing their formation and swelling, and altering air cell size.

2.4.1.5 Lipid

Lipids have two functions in extrusion process; they can influence the quality of the product and act as a lubricant during the process. Most of the lipids will melt at 40ºC, and the shear process will break them down into 10 μm parcels and then disperse them throughout the system. When the moisture of the material is lower than 25%, the addition of 0.5-1% lipids will greatly reduce the energy input needed for extrusion. When oil content rises up to 2-3%, it can have undesirable effects, such as reducing the extrudate expansion (Zhang, 1998).

2.4.1.6 Additives

Additives are also important in the makeup of the final product, as their reactions during the extrusion process can greatly affect the flavor or color of the product. Flavoring or coloring agents are mixed into the product and confer a different appearance to the product during the process. Additives used for increasing expansion and cell wall formation/swelling are termed nucleating agents and include sodium bicarbonate and calcium carbonate. Monoglycerides are commonly used in commercial operations (Guy, 2001) as surfactants (lubricants). Compared to regular corn puffs, the emulsifiers from rice bran have been shown to produce corn meal extruded products of lower bulk density, lighter structure, softer texture and more evenly distributed air cells (Barron, 2002).

2.4.2 Classification of ingredients by functions

The ingredients in a recipe can serve different functions during processing, making the extrusion processing quite complex and often leading to confusion amongst extrusion cooking technologists and machine operators. In order to understand the extrusion cooking process better, the Campden and Chorleywood Food Research Association (CCFRA) classified the ingredients systematically. Guy (1994) published a seven-group classification based on the grouping of ingredients according to their functional role using a physicochemical approach: structure-forming materials, dispersed-phase filling materials, ingredients that act as plasticizers and lubricants, soluble solids, nucleating substances, coloring substances, and flavoring substances. Examples of such groups in recipes for extruded products such as snack foods and breakfast cereals are given in Tables 2.3 (Guy, 2001).

2.4.2.1 Structure-forming materials

An extruded product's structure is created by forming a melt fluid from biopolymers and blowing bubbles of water vapor into the fluid to form foam (Guy, 1994). Structure forming polymers must have a minimum molecular weight sufficient to give enough fluid viscosity to prevent or control the shrinkage of an extrudate after it has reached its maximum expansion and rupture of the gas cells. If the extrudate is too viscous at this point, there will be rapid shrinkage and loss of apparent expansion in extruded products.

Proteins may be used to form structures in extruded products at high concentrations. For example soy proteins may be used to produce an expanded structure in textured vegetable protein (TVP), if their concentration in the recipe exceeds 40% w/w, at moisture levels of 30–40% w/w. These are globular proteins of 80–100D, making them significantly smaller than starch polymers in the melt fluid. However, they link together to form larger structures as they flow through a die channel. They aggregate and form higher viscosity complexes, which serve to form crude films and retain some of the expanding water vapor. Their viscosity on cooling is sufficient to prevent shrinkage and allows an alveolar structure to be formed. Other proteins, which are found in legumes and in the endosperm of wheat flour, have similar transitions. Wheat gluten is a hydrophobic protein, which can form polymers of greater molecular weight higher than the native form (Guy, 2001).

Table 2.3 Examples of recipes for extruded snack foods by weight (Guy, 2001)

Group	Common name	Corn curl	Maize/potato	Wheat
1	Maize grits	80.7	50	-
	Potato granules	-	20	-
	Potato starch	-	5	-
	Wheat flour	-	-	70
2	Wheat gluten	-	2	-
	Soy flour	-	-	5
	Wheat bran	-	-	10
3	Vegetable oil	0.5	1.5	1
	Water	16	18	16
	Monoglyceride	0.3	0.3	0.3
4	Salt	1	1	1.5
	Maltodextrins	-	5	-
5	Calcium carbonate	-	1.5	-
6	Milk powder	1	2	2.5
7	Glucose/peptides	0.15	0.15	0.1

2.4.2.2 Dispersed-phase filling materials

When extruded products like snacks or pet foods are made from starch-rich recipes, a continuous phase of starch polymers is found when microscopic sections of the product are observed. Several dispersed phases exist in the continuous starch structure; protein and fibrous materials such as cellulose or bran form the most significant structures. In all cases, the presence of the dispersed-phase materials affects the nature of the extrusion process in two ways: (i) their physical presence in the cell walls will reduce the potential for expansion of the starch film by disrupting cell walls when their structures penetrate the walls of the film; this effect is easily observed with wheat bran, and (ii) the presence of dispersed filler affects the elastic recoil or die swell effect of the fluid as it leaves the die exit. Pure starch fluids are very elastic and when they are deformed as they enter the die, they store elastic energy in their molecular structures. This energy is released as the fluid leaves the die and causes a swelling effect normal to the direction of flow in the die. Plastics research has shown that the presence of inert fillers, such as carbon black, reduces die swelling in plastics extrusions, until die swell disappears entirely at concentrations of 30–40% added filler (Guy, 2001). A similar effect was also detected in recipes containing added proteins or bran in wheat starch extrusions (Guy, 2001).

2.4.2.3 Ingredients that act as plasticizers and lubricants

Water, oils and fats are listed in this group. Physical interactions in low moisture dough cause frictional and mechanical energy dissipation. This energy source serves to heat the dough mass. The heating rate is very high in low moisture systems, so that for recipes up to 25% moisture no external heating is required to reach an operating temperature of 150ºC. The addition of ingredients such as water serves to reduce interactions by plasticizing the dry polymer forms, transforming them from solids to deformable plastic fluids. The addition of increasing amounts of water reduces the dissipation of mechanical energy and reduces the heat input as the moisture level is increased (Guy, 2001).

Oil and fats can reduce the shear level during the extrusion process when starch, fiber and proteins exist in the system. These materials serve to lubricate both the interacting particles in the dough mass and the particles that are rubbing against the metal surfaces of screws and barrel. The effect of lubricants is more powerful than that of plasticizers in terms of their active concentrations. Oils and fats produce large effects on the processing of starch at levels of 1–2%; however, higher levels may reduce the breakdown of starch polymers to such an extent that no expansion is obtained from a recipe. In certain recipes the effect of high levels of oils and fats is reduced by the addition of materials, such as bone meal, that can absorb the lipids in hollow rigid structures (Guy, 2001).

2.4.2.4 Soluble solids

Low molecular weight materials like salts and sugars are added in the process for flavoring or humectants properties. During the initial mixing stage of processing, the soluble materials will dissolve in free dough water. Different soluble solids' concentrations and their chemical interaction with starch and protein polymers will affect the extrusion process greatly. The dilution of other ingredients can occur when small molecules are added into the recipe. If they replace starch, the viscous effect of the large polymers will be reduced and the hot melt fluid will become less viscous unless the water levels are reduced. Only strong acids have been shown to have a significant effect on the degradation of starch in direct action on the polymers (Guy, 2001).

2.4.2.5 Nucleating substances

Two materials have been demonstrated to increase the numbers of bubbles present in an expanding extrudate, powdered calcium carbonate 'creta preparata' and talc (magnesium silicate). These bubble nucleation substances have been found to increase the numbers of bubbles appearing in the hot melt fluid of an extruder. Addition of a finely powdered material that remains insoluble in the dough provides surfaces to reduce the energy required for the formation of individual bubbles and can increase their numbers from a few hundred to over 70×10^3 per ml (Guy, 2001).

2.4.2.6 Coloring substances

Common in food industry, coloring materials, included in the recipe to generate colored extruded products, include heat stable colors and the color precursors which undergo thermal reactions to generate the desired color. Both natural and artificial colors are used in the extrusion process (Guy, 2001).

2.4.2.7 Flavoring substances

Flavoring substances act in a similar manner as coloring substances, and can be added either during the extrusion process or during subsequent treatment. There are some other flavors that may be added or exist in the material and form flavors during the extrusion process by the thermal reactions (Guy, 2001).

2.5 Soy bean and soy protein isolate

2.5.1 Soy production in the world

United States, Brazil, Argentina, China, India, Paraguay and Canada are the world's major soy producers (Table 2.4) supplying soy as food, vegetable oil, and animal feed. Golbitz and Jordan (2006) predicted that Brazil and Argentina will become the leading soy producing country in the world as they have large amounts of arable land, ample water resources, and low cost labor. They further predict that Brazil will surpass the US in soy exports.

2.5.2 Soy utilization

Asian nations are estimated to consume 95% of soybean production in the form of as a direct as human foodstuff. Asia has a long tradition of soy food production and consumption, whereas in Western nations, soybeans have only been consumed directly in recent years, though their acceptance has increased significantly.

Forms of soy food consumption vary widely around the world: in Asia, soy is mainly used in traditional foods such as tofu, soymilk and fermented products, whereas in Western nations, soybeans are consumed more commonly in the form of refined soy protein ingredients (used in food processing) than in as tofu or soymilk. There are many soy foods currently available in the market including tofu, tempeh, miso, soy sauce, okara, natto, soynuts as well as the cheese and cheese alternatives. Among these soymilk will be described as an example.

Table 2.4 World soybean production by major producers (Riaz, 2006*， World production statistics**) production (Million metric tons)

	00/01 *	01/02 *	02/03 *	03/04 *	04/05 *	05/06 **	06/07 **	07/08 **	08/09 **	09/10 **
United States	75.1	78.7	75.0	66.8	85.5	83.5	87.0	72.9	80.7	88.5
Brazil	39.5	43.5	52.0	52.6	63.0	57.0	59.0	61.0	57.0	62.0
Argentina	27.8	30.0	35.5	34.0	39.0	40.5	48.8	46.2	32.0	52.5
China	15.4	15.4	16.5	15.4	18.0	16.4	16.0	14.0	15.5	14.5
India	5.3	5.4	4.0	6.8	6.5	7.0	7.7	9.5	9.1	9.0
Paraguay	3.5	3.6	4.5	4.0	5.0	3.6	5.9	6.9	3.9	6.7
Canada	---	---	---	---	---	3.2	3.5	2.7	3.3	3.5
All others	9.4	8.6	9.6	10.2	11.6	9.5	9.3	8.0	9.1	9.4
Total	175.9	185.1	197.1	189.8	228.6	220.7	237.1	221.1	210.6	246.1

Traditionally, soymilk is the liquid extract of the soybean, which can be used in the preparation of tofu or as a nutritious beverage. However, the "bean taste" of traditionally-produced soymilk is not accepted by most Westerners. Nowadays a number of modern food processing techniques are performed to improve the quality of soymilk in terms of producing a bland product with greater appeal to Western tastes. Isoflavones retained in soymilk, are considered the most active phytochemicals in soybeans. Soy protein isolate (SPI) is used as a base to produce soymilk, to which other nutritional factors like vitamins A, D and calcium are also added to make it resemble cow's milk. It can also be put in cereal or made into yogurt, pudding, or ice cream.

2.5.3 Soy foods

Traditional soy foods include soymilk, tofu, miso, natto and tempeh, the last three of which are fermented foods, generally unattractive to Western tastes. Traditional soymilks made from whole beans have a strong "bean taste", which is a positive attribute in some Asian countries, but generally negative in the West.

Reduction of bean taste by using refined ingredients such as isolated soy protein, or by using proprietary taste masking or neutralizing agents (Pszczola, 2000) has made soymilks much more acceptable to Western people. New manufacturing processes and new soy ingredients have been developed to enable the manufacture of a broader range of products acceptable in the West. These include soy cheeses, soy yoghurts (Pszczola, 2000) and soy breads in Australia (Jorgensen *et al.*, 1999). New processes have also allowed the production of a larger range of meat alternatives more acceptable to the consumer. New soybean varieties with specific nutritional characteristics (*e.g.*, high oleic acid, low saturated fat, low stachyose, and low lipoxygenase) and new applications have been or are being developed (American Soybean Association and United Soybean Board, 2001). Soy breakfast cereal products have been made by some companies, but the product range is still limited and details on high soy protein content breakfast cereals are scanty.

2.5.4 Nutrition and health benefits of soy

Soybeans contain all three macro-nutrients required for good nutrition: complete protein, carbohydrate and fat, as well as vitamins and minerals, including calcium, folic acid and iron (National Soybean Research Library, 2007). Soybeans are the only plant food that contains complete protein. Soybean protein provides all the essential amino acids in the amounts needed for human health. The amino acid profile of soy protein is nearly equivalent in quality to meat, milk and egg protein.

The possible health benefits associated with eating soy foods is concentrated in three main areas - cardiovascular disease (Dotzel, 1999), cancer (Wiseman, 1997) and postmenopausal symptoms (Eden, 2001) which includes hot flashes, osteoporosis, estrogen replacement therapy, and cardiovascular function. Other linked areas are also been reported as cognitive function and neurologic diseases (White *et al.*, 1996), and bone development (Cooper and Melton, 1992). These benefits are associated with soy isoflavones.

2.6 Operation parameters and effects on the physical properties of the extruded products

Extrusion cooking technology serves the food industry as a method of high temperature-short duration processing. This method allows the development of new products such as cereal-based snacks including dietary fiber, baby foods, breakfast cereals and modified starch from cereals (Sebio and Chang, 2000). The place of extrusion foods in the snack food market has grown rapidly in the past 30 years mainly because it can economically produce a variety of products with an attractive texture, size and shape (Frame, 1994).

Corn meal is a major ingredient for extruded foods, such as ready-to-eat breakfast cereals and snacks. Numerous studies have investigated corn meal and the extrusion process. In recent years, products combining corn and other legume grains are getting more attention in the food industry. Molina *et al.* (1977) mentioned that of the approximately 13000 known species of legume grains, only a small number are used directly as a food item. The increased interest vegetable proteins for health reasons, may lead to a greater consumption of legumes, particularly in fortified foods and traditional dishes produced through extrusion technology.

Soy protein isolates (SPI), containing 90% soy protein, is widely used in the food industry as a protein source and is also available as a nutritional supplement in various forms. Incorporating SPI into corn flour significantly increases the nutritive value and quality characteristics of the extruded end product (Harper, 1989).

The many independent and response variables involved in the extrusion process make it a complex process to investigate. The independent variables including feed rate, raw material combination, raw material moisture content, barrel temperature, screw speed, screw profile, die shape, die size, barrel length and barrel diameter. The response variables including the motor torque, barrel fill, product temperature, specific mechanical energy (SME), die pressure and the residence time distribution (RTD). Of these variables, the independent variables can be controlled before the process; in other words, they are either a feature of the machine or something the machine allows it to control. The response variables will be affected by the independent variables and directly influence the raw material as it is processed into the final product (Emine and Faller, 2002). All these variables are important to the process and need to be examined.

Physical properties are important factors in extruded products and will directly affect the customer's acceptability of the final products. In order to develop more information regarding the relationship between processing variables and the physical properties of the extruded products, numerous studies have been done on the different raw materials and extrusion variables. Pansawat *et al.* (2008) studied the secondary extrusion variables and physical properties of fish- and rice-based snacks. Ding *et al.* (2006) studied the effect of extrusion conditions on the functional and physical properties of wheat-based expanded snacks. Cha *et al.* (2001) studied the physical properties of starch-based foams as affected by extrusion temperature and moisture content. Onwulata *et al.* (2011) studied the effect of dairy proteins in extrusion processing and other applications.

Several physical parameters have been selected to describe the properties of extruded products, and the relationship between these physical parameters and certain important extrusion process variables has been studied. The physical parameters including expansion ratio, bulk density, breaking stress, water solubility index, rehydration ratio and color. A response surface methodology is generally used to study the relations between extrusion variables and these physical properties.

As high protein content extrusion products are the target products in the present study, it will focus on understanding the important relationship between the extrusion machine mechanical parameters (variables) and the extruded products' physical properties.

2.6.1 Expansion ratio (ER)

In order to describe the expansion of the extruded products, Expansion ratio (ER) is used to evaluate the products. ER is defined as the ratio of the diameter of the extrudate to the diameter of the die, it will directly affect the product's density and volume. ER was used in many extrusion studies, Altan et al. (2008b) used ER to describe the barley flour–grape pomace blends extrusion process. Suknark et al. (1997) studied different starch and partially defatted peanut flour extrusion process, ER was one of the tested physical properties.

2.6.2 Bulk density (BD)

Bulk density is an important factor to many products; it will greatly affect the products' packaging volume and acceptability. BD is defined as the mass of a particulate material divided by the total volume they occupy. In the case of extruded products, the total volume includes particle volume, inter-particle void volume and internal pore volume. BD has been used in many different studies as one of the physical properties. Seker (2005), Altan et al. (2008), Park *et al.* (1993) all used BD as one of their products' physical properties.

2.6.3 Breaking stress (BS)

Breaking stress (BS) is the maximum stress that can be applied to a material, and describes the hardness of the sample. The common practice is to use a three point breaking test (Zasypkin and Lee, 1998) to measure the maximum force required to break the extrudate sample. Many studies used BS to describe the hardness of the products, like Bhattacharya (1997) used BS as one of the parameters in a rice and green gram (*Vigna radiata* L.) blend extrusion study, Sun and Muthukumarappan (2002) studied the effect of different variables to the extruded products' BS.

2.6.4 Water solubility index (WSI)

The most common forms of soluble solids used in extrusion are small carbohydrates and salt. They dissolve in the aqueous phase and form a more viscous plasticizing fluid but have little effect on most of the structure-forming biopolymers at low levels (<5%). However, they can reduce the level of starch in a recipe by their very presence in the melt fluid. For those products which will serve together with liquids, this factor could be very important. Anderson *et al.* (1969) described a method for testing the WSI and many of other studies followed his method. The effect of the extrusion processing parameters to WSI in different type of extrusion products were studied, Hagenimana *et al.* (2006) and Sekar (2005) tested the effect of extrusion parameter to the products' WSI in their studies.

2.6.5 Rehydration ratio (RR)

The rehydration ratio or index is the ratio of extrudate sample after rehydration to that prior to rehydration, expressed as a percentage (Lewicki, 1998). Many dehydrated products are used after being rehydrated, so the rehydration ration is an important parameter of dried products. Breakfast cereal products are frequently rehydrated prior to consumption, so the rehydration ratio is an important parameter as it will determine how much liquid the product can be absorbed.

2.6.6 Color

Color is important for almost any product, and the same is true of extrusion products. In order to describe a color, the suitable color systems are need to be selected, like the CMYK color system is the color system used for printing, the RGB colors are light primaries and the colors are created with light which can be found in electrical products like computer screens. In the present lab experiments, Hunter color system was used. The Hunter L, a, b color scale evolved during the 1950s and 1960s and it soon replaced the former XYZ system. In Hunter color system, L represents lightness ranging from 0 (black) to 100 (white); a represents green (negative a value) to red (positive a value); and b represents blue (negative b value) to yellow (positive b value). Many studies have used the Hunter system to describe the color of the products, including Sun and Muthukumarappan (2002), Park *et al.* (1993), Singh *et al.* (2007), etc.

2.6.7 Response surface methodology (RSM)

Response surface methodology (RSM) is a statistical method used to describe the relationship between process variables and product quality characteristics (Giovanni, 1983). In statistics, the response surface methodology (RSM) explores the relationships between multiple explanatory variables and one or more response variables. The method was introduced by G. E. P. Box and K. B. Wilson in 1951. The main idea of RSM is to use a sequence of designed experiments to obtain an optimal response. RSM model gives an approximation result, but it is widely used because such a model is easy to estimate and apply, even when little is known about the process.

RSM is commonly used in food extrusion studies. Nwabueze (2007) used RSM to investigate the effects of process variables on trypsin inhibitor activity of extruded African breadfruit, corn flour and soy flour blends. Park *et al.* (1993) studied physical properties of extruded corn flour, soy flour and raw beef blends using RSM. Chen *et al.* (1991) used RSM to investigate the effects of extrusion parameters on sensory properties of the extrudate. Altan *et al.* (2009) studied physical properties of Barley flour and barley flour-pomace (tomato, grape) blends extruded in a co-rotating twin-screw extruder using RSM. Altan *et al.* (2008a) studied the nutrition profile in a new extruded-expanded snack food by the RSM method. In the report by Pansawat *et al.* (2008), RSM was used to study the effects of extrusion conditions on secondary extrusion variables. Sibel and Fahrettin (2008) studied functional characteristics of the extruded snack food based on rice grit in combination with fruit waste, durum clear flour and partially defatted hazelnut flour by RSM method. Altan *et al.* (2008b) used RSM to study three independent variables of barley flour and tomato pomace blends processed in a co-rotating twin-screw extruder.

In the present study, the software Design-expert version 6.0 has been used to treat the data and do the RSM analysis.

2.7 Residence time distribution (RTD)

Amongst the previously listed response variables (section 2.6), Residence Time Distribution (RTD) is one affected by several other factors; however, the importance of RTD is such that it is include in almost all extrusion process studies. The concept of resident time distribution was first proposed by MacMullin and Weber (1935), while Danckwerts (1953) developed the theory and authored many important studies on RTD, later in the 1950s. The residence time distribution (RTD) can be used to characterize the mixing and flow within reactors and to compare the behavior of real reactors to their ideal models. RTD is a useful tool in estimating the yield of a given reaction and designing future reactors.

Residence time is a broadly useful concept that expresses how fast something moves through a system in equilibrium. It is the average time a substance spends within a specified region of space. In the extrusion process, RTD is used to describe the time that material spends in the extruder. The chemical and physical changes during the extrusion process are closely related to the RTD of the material. Thus, RTD studies provide information about the degree of mixing, severity and uniformity of thermo-chemical and rheological reactions that the material undergo during its passage through the extruder. The RTD is influenced many factors including barrel temperature, screw speed, screw configuration, feed composition, etc., thus an understanding of the RTD of an extrusion process can help to understand the overall process.

Residence time distributions are measured by introducing a non-reactive tracer into the system at the inlet. As the tracer is transferred into the chamber, its concentration is changed according to a known function and the response is found by measuring the concentration of the tracer at the outlet. The selected tracer should not modify the physical characteristics of the fluid (equal density, equal viscosity) and the introduction of the tracer should not modify the hydrodynamic conditions. In most of RTD studies, the change in tracer concentration will either be a pulse or a step. Bounie (1988) compared two heat-stable tracers - zinc oxide and erythrosin in the GAMMA model. Both Davidson *et al*. (1983) and Fichtali *et al*. (1995) used manganese dioxide as the tracer for the evaluation of the RTD. Other materials have also been used in the study of RTD, such as wheat flour (Yeh *et al*., 1992) and rice flour (Peng *et al*., 1994).

The RTD of SPI and corn flour mixture extrusions has received little attention, however, its evaluation is an important aspect of the current study. A second aspect of the present study is to evaluate the effect of the three independent variables (screw speed, feed moisture, and die diameter) on the RTD of SPI and corn flour mixtures processed through in a twin-screw extruder. Quantification of the effects of variables on the mean RTD and its variability can help to control and fine tune strategies to optimize product quality and safety.

2.7.1 Theory of RTD

Residence time is the amount of time that a particle spends in a particular system. This measurement varies directly with the amount of substance that is present in the system. The residence time distribution describes how materials are transported within a system.

Several studies have been focused on describing the RTD theory using E and F functions (Levenspiel,1972; Yeh *et al*., 1992; Abdelrahim *et al*., 1993a,b; Fichtali *et al*.,1995; Ramaswamy *et al*., 1995a,b; Iwe and Ngoddy, 2001). Both E and F curve can be obtained through the introduction of tracer particles at the entrance of the system and then collecting them at the exit. The particle can be introduced can be introduced in pulse or step mode. More detailed information about the theory of RTD is given in section 4.2

2.8 High soy protein content extrusion products

Since the consumer frequently demands healthy and nutritious foods, finding nutritional products is a food producer's constant target. Under most traditional cooking methods, degradations of the nutritional quality of finished food products that arise from high processing temperatures is a constant challenge. Extrusion cooking is a high temperature short time (HTST) cooking technique which provides thermal and shear energy to a food material undergoing significant physical and chemical changes. The HTST heating process can minimize heat degradation of food nutrients, while improving digestibility by gelatinizing starch and denaturing protein (Harper, 1981). This cooking technique is preferable to other food-processing techniques in terms of a continuous process with high productivity and significant nutrient retention (Guy, 2001; Singh, Gamlath and Wakeling, 2007). While the functional properties of the food ingredients are modified during the harsh extrusion process (Asp and Bjorck, 1989)， anti-nutritional or toxic compounds are also destroyed or inactivated: *i.e.*, trypsin inhibitors, hemagglutinins, and gossypol, undesirable enzymes such as lipoxigenases, peroxidases, lipoxidases and lipases, microorganisms and other food-borne pests (Harper, 1981).

Extrusion cooking offers continuous processing while maintaining significant nutrient levels (Guy, 2001). Extrusion of corn flour products has been extensively studied by Chinnaswamy and Hanna (1998) and Gomez and Aguilera (1984); however, when such products are made exclusively with this ingredient, they lack nutritional value. This has generated interest in increasing the nutritional value of extruded foods by adding other nutritional ingredients to the mix during food extrusion processing (Guy, 2001; Konstance *et al.,* 1998). Incorporating soy protein isolates (SPI) into corn flour can significantly increase the nutritive value and quality characteristics of the extruded product (Harper, 1989). With the development of the soybean processing industry, this crop has proven to be a low cost, and widely available source of superior quality protein. The use of SPI in extrusion products can provide a highly concentrated protein source, high in lysine, with a bland flavor, while reducing the flatulence factors and reducing sugars associated with whole soy flour. Thus SPI, widely used as a functional ingredient in the food industry, is considered to contribute to the overall improvement in extrusion product quality (Konstance *et al.,* 1998).

Showing a high potential to improve the nutritional profile of starch-based extruded food products (Sun and Muthukumarappan, 2002), SPI also possesses several potential health benefits. People with high (*vs.* low) soybean intakes have lower rates of coronary heart disease, breast cancer and osteoporosis (Liu, 2004; Sun and Muthukumarappan, 2002). Indeed, a statement that "including 25 g of soy protein per day in a diet low in saturated fat and cholesterol may reduce the risk of heart disease by lowering blood cholesterol levels" was approved by the US Food and Drug Administration (FDA, 1999). To uphold this soy protein health claim a single serving of the food must contain a minimum of 6.25 g of soy protein (FDA, 1999). Meeting the FDA soy protein threshold necessary to make such health claims can be achieved by producing SPI-enriched food products (i.e. extruded soy-corn blend food products) for all three meals and for snacks.

A number of studies have attempted to meet these goals. Konstance *et al.* (1998) produced extruded products combining corn meal with soy flakes, soy protein concentrate and soy oil. Faller *et al.* (1999) developed acceptable extruded snack products containing soy protein, and evaluated the influence of soy protein type, soy content, and moisture content. Muhungu *et al.* (1999) extruded corn flour and soy protein to investigate the influence of barrel temperature, moisture content, and relative residence time on extruder response and isoflavone profile. Sun and Muthukumarappan (2002) examined the effects of defatted soy flour content, feed moisture, screw speed and temperature on the functionality of soy-based extruded products. Seker (2005) evaluated the expansion ratio, bulk density and water solubility index of extruded products of SPI-modified corn starch mixtures. The effects of moisture content, screw speed and soybean content on the textural qualities of soybean-corn starch extruded products were studied by Li *et al.* (2005). Notwithstanding their potential importance, processing conditions (*e.g.*, extrusion temperature, feeding moisture and SPI content) which optimize end-product physical quality parameters (e.g., expansion ratio, bulk density, breaking stress, water solubility index, rehydration ratio and color) during the extrusion of high protein content (32.2-66.6%) corn starch blends have yet to be studied.

2.9 Drying process

Extrusion cooking is widely used in the food industry, as it offers continuous processing while maintaining significant nutrient levels (Guy, 2001). However, in order to maintain storage stability, moisture content is one important factor one cannot ignore. In our previous study (Yu e*t al.* 2009), physical characters of extruded corn flour, SPI blends (with a constant ratio of 20% SPI to 80% corn flour) were studied. In the present follow-up study, high protein content (33.2%—66.7%) products are studied. For all these studies, the water activity of the extrudes have always exceeded 0.9, so, in order to get a stable product, a subsequent drying process is necessary.

The dehydration (or drying) process is probably the oldest and most frequently used method of food preservation. It is currently a widespread technique in the food industry as well as a subject of continuous interest in food research. Dehydration refers to the removal of moisture from a material with the primary objective of reducing microbial activity and product deterioration (Ratti, 2001). Drying has been used as a method to preserve foods for centuries. Sun drying of fruits and smoking of fish and meat are both well-known processes that originated in antiquity. In fact, Persian and Chinese people dried fruits and vegetables in the sun as long as 5000 years ago (Ratti, 2009).

Sun-dried dates, figs, raisins, apricots and some other products were supposedly developed by the aboriginal habitants of the Mediterranean Basin and Near East (Salunkhe *et al.,* 1991; Van Arsdel *et al.,* 1963). Based on the use of an artificial heat source, various dehydration technologies were developed at the beginning of the 20th century. The name dehydration was given to drying methods undertaken under controlled conditions. After the first record of artificial drying of foods appears in the 18th century, vegetables treated in hot water subsequently placed in a hot stove for drying (Van Arsdel *et al*., 1963), drying technology saw accelerated development and many methods appeared for drying food, including hot air drying, vacuum drying, drum drying, spray drying, freeze-drying, and so forth.

2.9.1 Drying (dehydration) principles

Drying is a process whereby one removes water from a wet material and obtains a relatively dry product. A wet material placed in a medium having lower water partial pressure (at the same temperature) will dehydrate until equilibrium is reached. The medium is usually air at pressures ranging from a high vacuum to atmospheric (or higher), although superheated steam, hot oil, solvents, and solutions may also be used for this purpose (Karel and Lund, 2003).

The food dehydration process is a balance between the water contained in a foodstuff and the water present in the drying medium, which can be air or liquid. During the drying process, water evaporates or is removed from the food surface to the surrounded medium. In an air-based medium drying process, water is evaporated from the surface of the food into the surrounding air, and the water inside the food will transfer to the surface, and the process will continue until equilibrium conditions are achieved. These equilibrium conditions represent a balance between moisture vapor pressure at the surface of food and that of the surrounded air (Ramaswamy and Marcotte, 2006). During the evolution of the drying process, the moisture is removed continuously. However, the rate is not necessarily constant throughout. Especially toward the final stages of drying, there is very little moisture removed.

2.9.2 Drying curve

A drying curve is normally obtained by plotting some form of moisture change with respect to time: *e.g.*, moisture content *vs.* time, moisture loss *vs.* time, rate of moisture loss *vs.* time, rate of moisture loss *vs.* moisture content, and residual moisture *vs.* time (Ramaswamy and Marcotte, 2006). A drying curve will give information on the time necessary for a product to be dried under certain specific conditions. Furthermore, it will help to design or to calculate the size of the dryer necessary. In order to obtain a drying curve, a test sample of known mass is placed in a dryer under appropriate test conditions (constant temperature, humidity, and air velocity), and the moisture loss from the product is monitored over time. Figure 2.3 shows some typical setups used for drying experiments. (Ramaswamy and Marcotte, 2006)

Moisture content is the percentage of water in the product. In order to calculate this, sample weight can be recorded either directly on a scale within the drying apparatus, or by taking out the sample periodically and recording its weight. It is possible to estimate the sample moisture content periodically using the sample weight and the original sample weight and the original sample moisture content. The recorded data can be used to estimate the proper drying time to reach a desired value of moisture content. Two ways, wet basis (% wb) and dry basis (% db), are used in describing the moisture content of food. Figure 2.4 shows three types of drying curves that are widely used.

Figure 2.4 represents typical drying curves using moisture content and drying rates and shows typical temperature variations during test runs. Six different points (ABCDEF) are identified. The period from A-C characterized the free movement of moisture from the food to the air. This is a constant rate drying period, the drying rate depends primarily on the air conditions and not on the product conditions of a relatively high-moisture material. This condition is also been called saturated surface drying due to the air leaving the dryer being mostly saturated, The potential for moisture migration from the interior to the surface is greater than the evaporation rate at the surface.

The following period C-D is the first falling rate period, in this period, the moisture migration from the interior to the surface is less than the evaporation rate at the surface. . This period is also called unsaturated surface drying, since the air leaving the dryer remains quite moist, but it is not saturated, and the air temperature begins to rise above the wet bulb level starting and continues to do so until the drying is finished. This stage proceeds until the surface film of the liquid is entirely evaporated.

The second falling rate period is D–E. Compared to the first falling rate drying period C-D, the rate of the second falling rate drying period drops even more rapidly. The drying rate is mainly limited by the diffusion of moisture from within the product to the surface. In the period of E–F, no significant drying occurs as equilibrium conditions are reached. The moisture content within the food is in equilibrium with that of the air, and the air fails to pick up any moisture from the product.

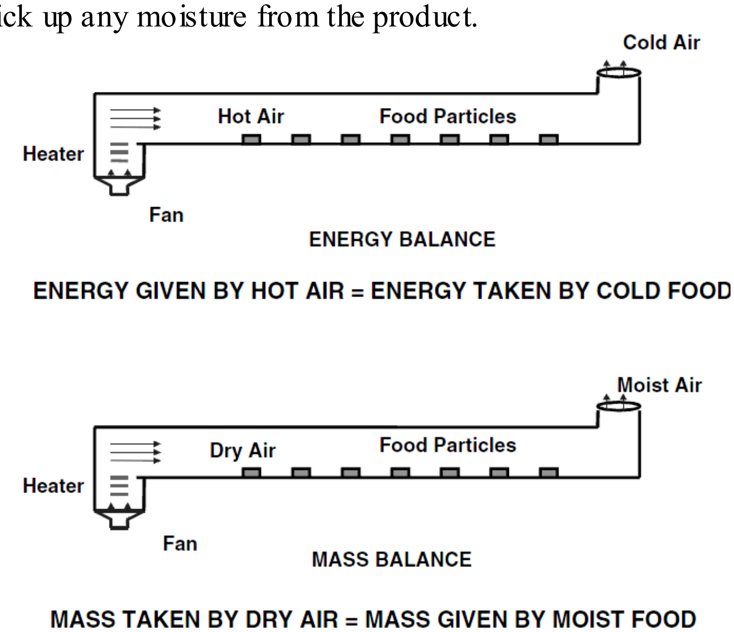


Figure 2.3 Typical air-drying processes. (Ramaswamy and Marcotte, 2006)

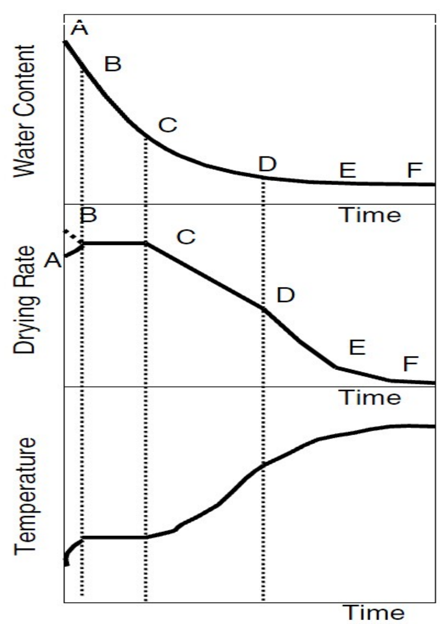


Figure 2.4 Drying curves using moisture content, drying rate, and temperature. (Ramaswamy and Marcotte, 2006)

2.9.3 Water activity (a_w)

Water activity is a dimensionless quantity used to represent the energy status of the water in a system. It is defined as the vapor pressure of water above a sample divided by that of pure water at the same temperature; therefore, pure distilled water has a water activity of exactly one. It is widely used in food science as a simple, straightforward measure of the dryness of food; foods typically have an optimum water activity at which they have the longest shelf life. Water activity is an important factor which will affect the storage properties of food, as it is the factor most strongly linked to microbial growth. Figure 2.5 shows the relationship between water activity and the growth of microorganisms.

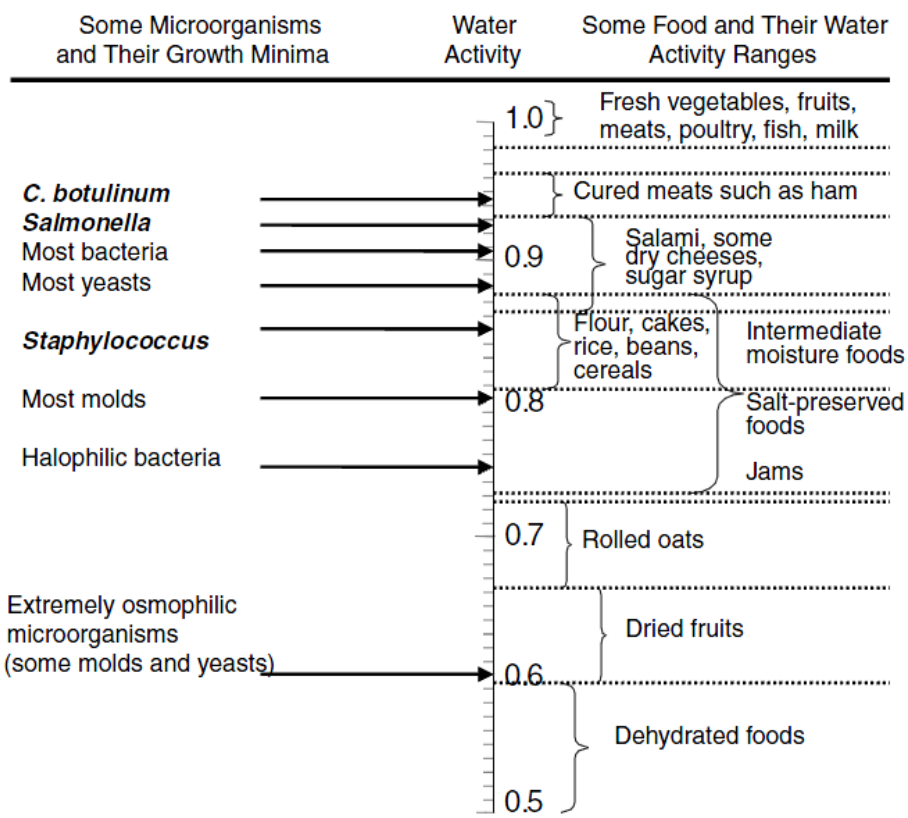
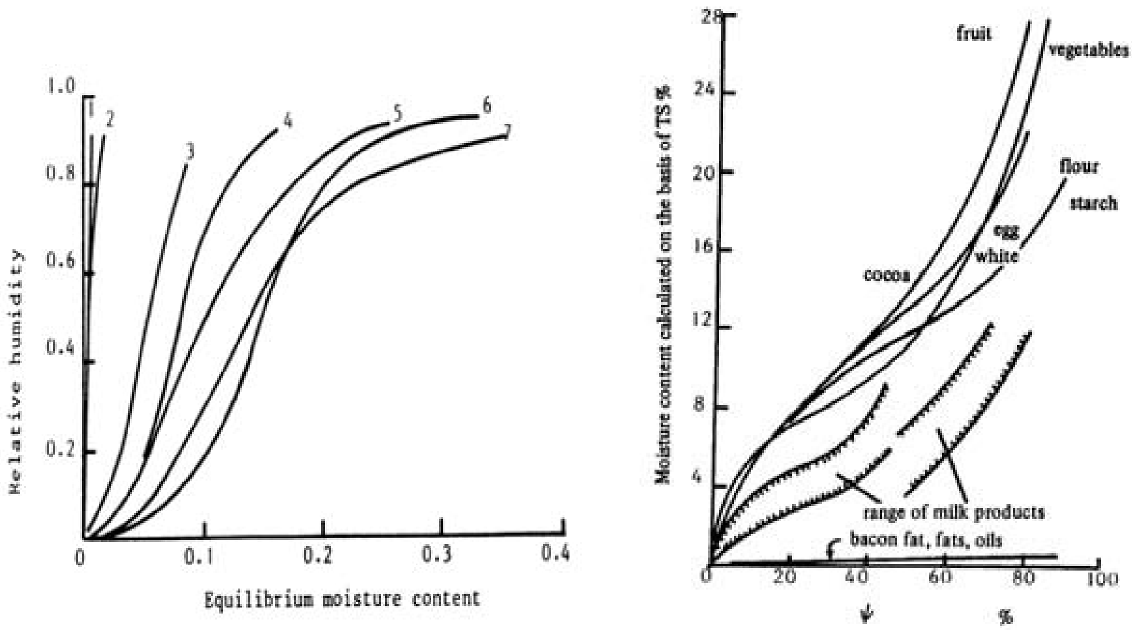


Figure 2.5 Relationship between Water activity and the growth of microorganisms. (Ramaswamy and Marcotte, 2006)

2.9.4 Sorption isotherms

The equilibrium relative humidity of the air (ERH) and the equilibrium moisture content within solid foods (Me) are parameters important to an understanding of the dehydration process. This equilibrium condition is used to determine the end point of drying and for the evaluation of product stability during storage. This relationship is highly temperature dependent and needs to be determined at a constant temperature. Multiple ERH-Me relationships developed at various temperatures can be plotted in the form of graphs called *moisture sorption isotherms* (Ramaswamy and Marcotte, 2006). Figure 2.6 is a typical figure of sorption isotherms.



a_w *vs.* Moisture content (db) Moisture content (db) *vs.* a_w

Figure 2.6 Two representations of sorption isotherms (Mujumdar and Menon, 1995)

The state of water within the food will greatly influence the drying process and product stability. The water in a material can be roughly separated into unbound water (free water) and bound water. Bound water is further divided into two types: water bound in multiple layers (it is less strongly bound and it is free water), the other type is bound through an adsorption mechanism, this moisture is referred to as *monolayer moisture* and is strongly bound by adsorption forces. Figure 2.7 is listed the relationships between sorption isotherms and the water condition inside the food are illustrated in Figure 2.7.

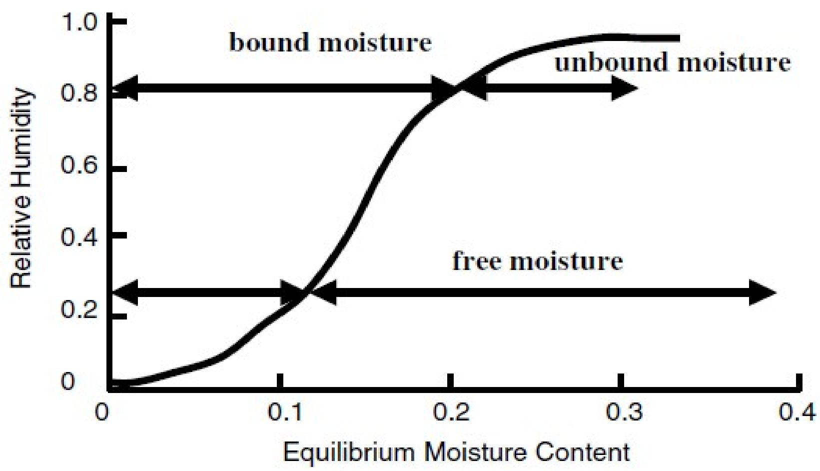


Figure 2.7 Me–ERH relationship or moisture sorption isotherm (Ramaswamy and Marcotte, 2006)

Food quality and safety are amongst the main goals of food science and technology. Measuring water activity provides information on the stability of food, its availability for the growth of microorganisms (spoilers and pathogens) and degradation processes (enzymatic and non-enzymatic deteriorative reactions). Water activity is therefore the best parameter for the prediction and the assurance of food quality and safety. Indeed, spoilage and pathogenic bacteria do not generally grow at a water activity value that is less than 0.90; mould growth ceases at water activity values smaller than 0.80 with some few exceptions such as *Zygosaccharomyces rouxii* that is capable of growing at the lower water activity values of 0.61-0.65. With the exception of fat oxidation, the kinetics of deteriorative reactions is low at low values of water activity. In fact, when water activity is low, oxygen easily gets into contact with the fat and it oxidizes the unsaturated fatty acids.

The sorption isotherm is important as it tells whether water will migrate to the environment from the food or the latter will absorb water from its surrounding atmosphere. It is crucial to know the sorption properties of food as they indicate appropriate storage conditions, avoiding changes in water activity due to either loss or gain of water. Therefore besides other factors like pH, chemical composition among others, it is required to be knowledgeable about water activity, how it is influenced by moisture content in order to control the quality and assure the safety of food.

2.10 Frying process

Deep-fat frying is one of the oldest processes of food preparation and consists basically in the immersion of food pieces in hot oil. It is a widely used technique in food industry. The high temperature frying process causes the transfer of the oil to the food, and concurrent water evaporation. Frying is often used as a method for creating unique flavors and textures in processed foods to improve their overall palatability. The high temperature of the frying fat, typically leads to the appreciated textural characteristics of fried foods, and also causes a complex group of chemical reactions, like Maillard reaction and caramelization, leading to the development of browning (BeMiller and Whistler, 1996). Factors that affect heat and mass transfer are the thermal and physicochemical properties of the food and the oil, the geometry of the food and the temperature of the oil. Deep fat frying is the simultaneous cooking and drying process through contact with hot oil and it involves simultaneous heat and mass transfer. In these processes oil plays a dual role in terms of serving as a heat transfer medium and in improving the texture and flavor of the product.

Most frying processes occur at an oil temperature between 160 and 180°C and are characterized by high drying rates (Baumann and Escher, 1995). This fast drying is critical to the improvement of the mechanical and structural properties of the final product. These conditions lead to high heat transfer rates, rapid cooking, browning, texture and flavor development (Farkas *et al.,* 1996). During deep frying of products the latent heat of vaporization from the product is the major means of heat transfer and this process of energy removal maintains the temperature of food-oil interface at around 100^{o}C, thus preventing burning of the product (Blumenthal, 1991). When the moisture content decreases the amount of heat carried off by the latent heat of evaporation decreases and burning can occur, leading to poor product quality. For finished fried products the final moisture content of the product is often related to its expected tenderness. Oil added during the frying process is one important constituent to improve textural quality of fried starch based products apart from their nutritional value. Generally in protein-starch enriched extrudate frying has wide range of influence on product quality and their complex interaction in determining the texture, color and oil uptake of fried products. However the effect of post extrusion frying process of soy protein isolate incorporated in corn flour blends has not been studied well on typically fried extruded products.

2.10.1 Maillard reaction

The Maillard reaction occurs between a reducing sugar and an amino group and initiates a sequence of consecutive and parallel reactions. The French scientist L.C. Maillard was the first to observe and described the production of darkly-colored compounds when solutions of glucose and lysine were heated. In some later studies, different sugars and amino acids were found to cause similar reactions and that the reducing group (aldehyde or ketone) on the sugar was important for the reaction. Proteins were then shown to undergo similar reactions, and later people discovered that the proteins' free amino groups were the main reactive groups.

During the frying process, the reducing sugars in corn flour and the protein from soy protein will allow Maillard reactions to occur, which can confer new characteristics on the final products. Consequently certain sensory test can be important.

2.10.2 Sensory evaluation

Sensory tests are widely used in the food industry. Using sensory information as a part of a marketing decision has been given unprecedented attention. Being able to identify and quantitatively model the key drivers for a product's acceptance is now generally recognized as a core resource for any sensory program. Thus, combining sensory tests with new-product innovation can provide information to research and development people so they can develop more acceptable products. The importance of understanding consumer likes, dislikes and preferences are clearly known among sensory practitioners. In the field of sensory science there exists an array of techniques, or methods, from which the sensory practitioner may choose in order to gauge consumer like or dislike of a product (Stone and Sidel, 2004).

Three common methods employed are the paired-preference, ranking, and rating-scale techniques. The rating-scale technique is quite popular and has the advantage of simplicity in handling situations where a large number of foods must be judged (Schutz, 2006). Within the rating-scale technique three types of successive-category scales can be distinguished, they are: quality judgment; like-dislike scale, commonly known as the hedonic scale (Peryam and Girardot, 1952); and action scale (Schutz, 2006). Table 2.5 illustrated these three types of scale methods.

Table 2.5 Three types of successive category-rating scales used for measurement of food attitudes

Quality judgment	Like-dislike affect	Action
		I would like to eat this food
Extremely poor	Dislike extremely	Rarely or never
Very poor	Dislike very much	Once a month
Poor	Dislike moderately	Once every two weeks
Below fair- above poor	Dislike slightly	Once a week
Fair	Neither like nor dislike	Twice a week
Below good- above fair	Like slightly	Several times a week
Good	Like moderately	Once a day
Very good	Like very much	Twice a day
Excellent	Like extremely	

Quantitative descriptive analysis (**QDA**) has gained acceptance for sensory evaluation of various foods (Stone and Sidel, 1998). QDA can be conducted as a kind of quality judgment (Table 2.5). QDA is based on the ability to train panelists to measure specific attributes of a product in a reproducible manner, so as to yield a comprehensive quantitative product description amenable to statistical analyses (Chapman *et al.,* 2001). In a QDA approach, panelists recruited from the general public work together in a focus group to identify key product attributes and appropriate intensity scales specific to a product. This group of panelists is then trained to reliably identify and score product attributes. Ten to twelve individual per panel is recommended in QDA methodology (Stone and Sidel, 2004). The panel leader works as a communication facilitator without involvement and interference with panel discussions. References can be used for generating sensory terminologies, especially when panelists are confused and disagree with each other on some sensory attributes during training sessions (Stone and Sidel, 2004).

Line scales are used for panel training and data collection in QDA tests. This line scale is associated with a sensory intensity word. The scale direction goes from left to right with increasing intensities, e.g., weak to strong. During data collection, panelists measure sensory intensities independently at an individual booth without reference to intensities standards. Panelists are allowed to use different parts of the scale to determine the sensory intensities by themselves. As a result, the difference among products produced by QDA will be a relative measurement; the importance of an absolute scale value has been neglected. Subjects' reliability is evaluated by their repeated measurements on product attributes (Stone and Sidel, 2004).

Quality rating test can be included in rating methods. Within the three normal types of rating system listed in Table 2.5, quality judgment has been used primarily in the laboratory with small panels whose members usually have some specific experience or knowledge about the products under investigation, but this is not always easy to translate into the attitudes or behavior of consumers. When inexperienced judges are used with such a scale, evidence exists from studies at the Quartermaster Food Ss Container Institute (Schutz, 2006) that they rate according to what they like rather than for inherent quality differences. Here, the quality rating test gives the judges the freedom to give a mark about their own preference about the products to show the feeling of how much the customer like the products.

2.11 Organization of this thesis

The thesis is organized into eight chapters: first chapter of general introduction; second for literature review, followed by five chapters (third to seventh) for describing the thesis research, followed by the eighth for general conclusions, contribution to knowledge and future studies. The final chapter is the references. More specifically,

Chapter 1 provides a brief overview of the problem and lists the overall project objectives.

Chapter 2 reviews information on food extrusion, food extruder, materials used in food extrusion, and some concepts used during in the present study.

Chapter 3 presented the first experiment in the present study, a basic study of the extrusion work. It provides useful information about our extruder, the corn flour, and SPI blend extrusion products.

Chapter 4 describes the study of the resident time distribution, and provides further information about how the corn flour- SPI blend extrusion products run through the extruder.

Chapter 5 presents a very important part of the present study: formulations of protein rich products (total protein content 32.2-66.6%) were studied and optimization conditions were identified based on product specific constraints.

Chapter 6 describes the investigation of extruded products drying behavior, in order to get a stable product. Drying is always an important step for extrusion products. In this the product's moisture content, water activity, moisture ratio and the effect of the protein content, moisture content and processing temperature to the drying process are investigated.

Chapter 7 describes an attempt to produce a possible industrial product, combined the extrusion product with a frying process, which can bring wonderful new characters to the extrusion products.

Chapter 8 is the final chapter and presents general conclusions drawn from the present study.

The final section is References where all the cited references are detailed in an alphabetical order.

CHAPTER 3 TWIN SCREW EXTRUSION OF CORN FLOUR AND SOY PROTEIN ISOLATE (SPI) BLENDS: A RESPONSE SURFACE ANALYSIS

Abstract

The effects of feed moisture, screw speed and barrel temperature on physical properties of extruded corn flour and soy protein isolate (SPI) blends were investigated in a co-rotating twin screw extruder using a response surface methodology. Corn flour and SPI were mixed with ratio of 4:1. The screw speed was set at five levels between 60 and 140 rpm, barrel temperature between 140 and 180°C, and feed moisture between 18% and 38%. All physical properties of the extruded material evaluated - included expansion ratio, bulk density, breaking strength, water solubility index, rehydration ratio and color – were significantly ($p<0.05$) affected by the three process variables. Feed moisture was the most significant variable with quadratic effects on most of the physical properties. Response surface regression models were established to correlate the physical properties of the extruded product to the process variables. Understanding the effect of these variables on the product physical properties was deemed useful for the development of protein rich extruded products.

3.1 Introduction

Extrusion cooking has been used in a large number of food applications as it has some unique positive features compared with other heat processes. Nowadays, food extruder is considered as a high-temperature short-time bioreactor that transforms raw ingredients into a variety of modified intermediate and finished products. During the extrusion process, the material is treated not only by heating, but also by intense mechanical shearing, compression and torque, which are able to break the covalent bonds in biopolymers (Singh *et al.,* 2007). Thus, the functional properties of the food ingredients are rapidly modified due to the combined influence of temperature, pressure, shear and time (Carvalho and Mitchell, 2000). Food extruder also permits to inactivate the undesirable enzymes that may affect the quality and eliminate several anti-nutritional factors, such as trypsin inhibitors, haemagglutinins, tannins and phytates (Bhandari *et al.,* 2001; Singh *et al.,* 2007).

Corn and wheat are widely consumed throughout the world as they are a good source of starch and several micronutrients, such as vitamin B_1, vitamin B_5, folate, dietary fiber, vitamin C, phosphorus and manganese. In extrusion process, corn is widely used in making breakfast cereals and snacks and it offers attractive crunchy texture, golden color and desirable flavor; however, the protein content in corn products is somewhat low. Soybeans contain all three macro-nutrients required for a good diet (protein, carbohydrate and fat) and also contain high amounts of vitamins and minerals (National Soybean Research Library, 2007). Soy protein isolates (SPI) which contain 90% (w/w) protein is widely used in food industry as a protein source and is also available as a nutritional supplement in various forms. Incorporating SPI to corn flour will significantly increase the nutritive value and quality characteristics of the extruded end product (Harper, 1989).

The relationship between process parameters and physical properties of the extrudate which is incorporated of corn flour and SPI blends has not been studied in detail. This kind of study is the essential of product development and process control considering the popularity of the application of corn and SPI in food extrusion.

The objectives of the present work were therefore to investigate: **(a)** the influence of extrusion process variables on physical properties of extruded corn flour and SPI blends using a twin screw extrusion process by using RSM, and **(b)** establish regression models to predict the physical properties of the extruded material as a function of the process variables. This study forms the basis of our extrusion research program on soy protein enriched extruded products.

3.2 Materials and methods

3.2.1 Experiment materials

Corn flour from Brar Natural Flour Mills (Winnipeg, MB) was purchased locally (composition: 1.7% of lipids, 76.7% of starch and 10% of protein). Soy protein isolate (SPI) was received from American Health and Nutrition (Ann Arbor, MI), which contained 90% protein. The moisture contents of the flours were measured before mixing. The flours were mixed using a Hobart mixer (Hobart Food Equipment Group Canada, North York, Ontario, Canada) to give corn flour: SPI ratio of 4:1 (wet basis). Appropriate amount of water was added to adjust the mixture to the required moisture content as per the experimental design. The blends were sealed in polyethylene bags and equilibrated for 24 h at $4^{o}C$ before extrusion.

3.2.2 Extrusion process

Extrusion was performed in a co-rotating twin screw extruder (DS32-II, Jinan Saixin Food Machinery, Shandong, P. R. China), consisting of three independent zones of controlled temperature in the barrel. The diameter of the screw was 30 mm. The length to diameter ratio of the extruder barrel was 20:1. The diameter of the hole in the die was 5 mm with a die length of 27 mm. The screw speed and the temperature of the third barrel section (metering section) were adjusted to the required levels. The extruder was fed manually through a conical hopper, keeping the flights of the screw fully filled and avoiding accumulation of the material in the hopper.

After reaching stable conditions, extruded products were collected and cool dried under mild air flow conditions at room temperature overnight and then finish dried to moisture content of 9-10% (wet basis) by an air convection oven at 45°C and at an air flow of 0.1m/s. Dried samples were stored in air tight plastic containers at room temperature and used for analysis.

3.2.3 Experimental design

Extrusion is a complex process involving many variables. Among them, barrel temperature, screw speed and feed moisture are the most important factors. A central composite rotatable design (CCRD) (Draper, 1982) was used to incorporate these three independent variables. This design required 20 experimental runs with 8 (2^3) factorial points (three level for each variables), six star corner points (two for each variable) and 6 center points. Overall this yields 5 levels for each variable and permits a better assessment of their quadratic effects. The CCRD coded levels and experiment ranges of the 3 independent variables are shown in Table 3.1, these were selected based on preliminary tests.

In order to develop the RSM models, coded values are used which are obtained from the real values of independent variables screw speed (S) (rpm), barrel temperature (T) (^{o}C) and feed moisture (M) (%) as shown below:

$$S = \frac{(Screwspeed - 100) * 1.682}{40} \tag{3.1}$$

$$T = \frac{(Barreltemperature - 160) * 1.682}{20} \tag{3.2}$$

$$M = \frac{(Feedmoisture - 28) * 1.682}{10} \tag{3.3}$$

3.2.4 Physical properties

Several physical parameters were selected from those that have been used to describe the properties of the extruded products including: Expansion ratio (ER), Bulk density (BD), Breaking stress (BS), Water solubility index (WSI), Rehydration ratio (RR), Color.

Expansion ratio (ER)

The definition of ER was described in Chapter 2, it is the ratio of the diameter of the extrudate to the diameter of the die (Jyothi *et al.,* 2009). 20 randomly selected segments of each sample were measured using calliper and the average value was used to determine the ER in the experiment.

Table 3.1 CCRD experiment design: coded values and actual values

Run	Screw speed (rpm)	Barrel temperature (°C)	Feed moisture (%)
1	1 (127)	1 (172)	1 (35)
2	1 (127)	1 (172)	-1 (21)
3	1 (127)	-1 (148)	1 (35)
4	1 (127)	-1 (148)	-1 (21)
5	-1 (73)	1 (172)	1 (35)
6	-1 (73)	1 (172)	-1 (21)
7	-1 (73)	-1 (148)	1 (35)
8	-1 (73)	-1 (148)	-1 (21)
9	1.68 (140)	0 (160)	0 (28)
10	-1.68 (60)	0 (160)	0 (28)
11	0 (100)	1.68 (180)	0 (28)
12	0 (100)	-1.68 (140)	0 (28)
13	0 (100)	0 (160)	1.68 (38)
14	0 (100)	0 (160)	-1.68 (18)
15	0 (100)	0 (160)	0 (28)
16	0 (100)	0 (160)	0 (28)
17	0 (100)	0 (160)	0 (28)
18	0 (100)	0 (160)	0 (28)
19	0 (100)	0 (160)	0 (28)
20	0 (100)	0 (160)	0 (28)

Bulk density (BD)

Displacement method was used to measure the bulk density (Seker, 2005) in this study. Extruded products were cut into 25 mm long strands and about 15 g strands were weighed (M_{ext}, g). A 100 ml cylinder was used to measure the volume of the sample, samples were first put into the cylinder and yellow millet particles were added to fill up the cylinder. The extruded products were taken out and the volume of the yellow millet particles was measured (V_{ym}, ml). BD was calculated as below:

$$BD = \frac{M_{ext}}{100 - V_{ym}} \text{(g/ml)} \quad (3.4)$$

Breaking stress (BS)

Breaking stress was measured using the Lloyd texture machine with a 500 N load cell (Lloyd model LRX, Lloyd Instruments Ltd., Fareham, Han, UK). Three point breaking test (Zasypkin and Lee, 1998) was used to measure the maximum force required to break the extrudate samples. The extruded product was cut to obtain 35 mm-long strands, which were placed at right angle on two rounded stands (bridge) 30 mm apart. The rounded crosshead exerting force in the middle of the bridge was moving down at 5mm/min until breaking. BS (N/mm^2) was determined as the breaking force per unit cross section area. 8 measurements were made on each product, and the average value was used.

Water solubility index (WSI)

Water solubility index was determined using the method of Anderson, Conway, Pfeife and Griffin (1969). 2g of sample were ground and sieved through a standard sieve (#50), the sample was placed into a centrifuge tube and then soaked with 25ml distilled water at 30^{o}C for 10 minutes with intermittent shaking every 2 min. The subsequently sample was centrifuged at 5000 rpm for 15 min (Sorvall GLC-2B General Laboratory Centrifuge, Du Point Instrument). The supernatant was decanted into a Petri dish and dried at 105°C overnight. The weight of the dry solid (W_{ds}) was determined and the WSI (%) calculated

Rehydration ratio (RR)

Rehydration ratio was measured at 30°C. The extrudate was cut to obtain 35 mm-long strands and around 20g strands were weighed (M_1) and placed in 500 ml of water at 30°C for 15 min. The water was drained and the rehydrated samples were weighed (M_2). RR was defined as below:

$$RR = \frac{M_2 - M_1}{M_1} \times 100(\%) \tag{3.5}$$

Color

A Minolta colorimeter with an aperture of 1.2 cm diameter was used to test the color of the sample (Minolta Colorimeter Model CM-500d). The exposed area was sufficiently great relative to the illuminated area to avoid any light-trapping effect. The instrument was calibrated with a standard white tile (L = 77.58, a = -0.27, b = -26.63). The color was reported in terms of L, a and b values. Eight measurements on each sample were taken and the average value was used.

3.2.5 Data Analysis:

Results were analyzed using Design-Expert 6.0 (Stat-Ease Inc.) and the second-order polynomial model was established to predict the dependent variable (Y):

$$Y = c_0 \sum_{i=1}^{3} c_i X_i + \sum_{i=1}^{3} c_{ii} X_i^2 + \sum \sum_{i,j=1}^{3} c_{ij} X_i X_j \tag{3.6}$$

Where c_0, c_i, c_{ii}, c_{ij} are coefficients of intercept, linear, quadratic and interactive effects, respectively, and X_i, X_j are independent variables. The mapping of the fitted response was generated using Design-Expert 6.0 (Stat-Ease Inc.). The response surface was plotted as a function of two variables while keeping the remaining variable at the center point value.

3.3 Results and discussion

The data on mean values of all physical properties of extruded products along with their standard deviations are summarized in Table 3.2. Analyses of variance are summarized in Table 3.3. Models for all parameters were significant and all parameters were significantly ($p < 0.05$) affected by screw speed, barrel temperature and feed moisture, and their quadratic effects.

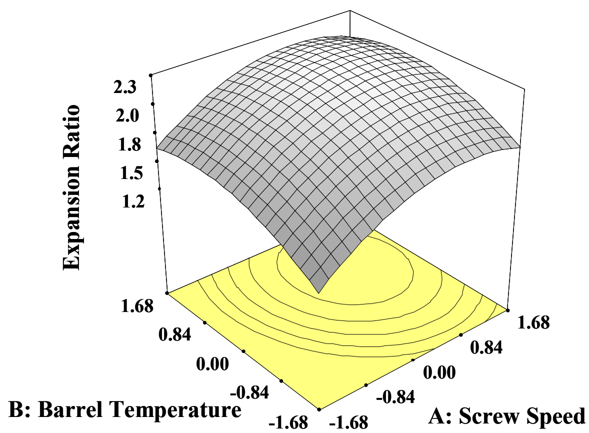
Various physical properties have been studied in different extruded products. Jyothi *et al.* (2009) studied the physical properties including bulk density, true density, porosity, and expansion ratio; water absorption index, water solubility index, oil absorption index, in the single extruder to process tuber starch. Rocha-Guzman *et al.* (2008) studied water absorption index (WAI), water absorption capacity (WAC), oil, absorption capacity (OAC), and emulsifying capacity (EC) in the extrusion process of normal bean cultivars flour. Őzer *et al.* (2004) studied the physical properties (bulk density, expansion, and porosity) of a nutritionally balanced extruded snack food by the RSM method. The influence of process variables on physical properties have been shown to be generally significant in all these studies.

3.3.1 Expansion ratio (ER)

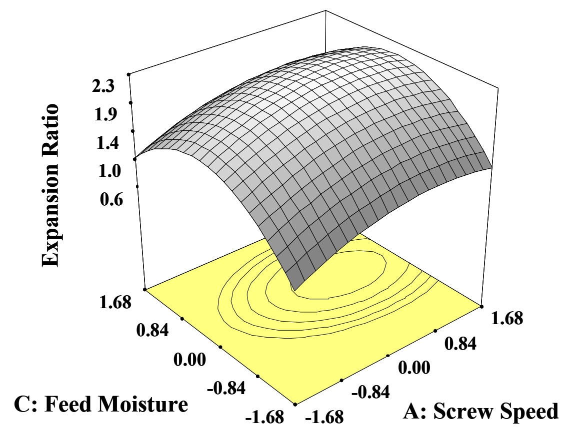
The fitted model for ER is shown in Eq. 3.7 (all independent variables in coded values) indicating quadratic effects with all three variables.

$$\begin{aligned} ER &= 2.164 + 0.142S + 0.097T + 0.12M \\ &- 0.103S^2 - 0.076T^2 - 0.279M^2 \end{aligned} \qquad (3.7)$$

Figure 3.1 shows the response surface plot of ER vs two independent variables with the third taken at the midpoint (coded 0) level. Figure 3.1-A is ER vs temperature and screw speed in which the feed moisture was controlled at 28% (coded value 0); Figure 3.1-B is ER vs feed moisture and screw speed with the barrel temperature set to 160ºC and Figure 3.1-C is ER vs feed moisture and temperature with the screw speed set at 100rpm.



A



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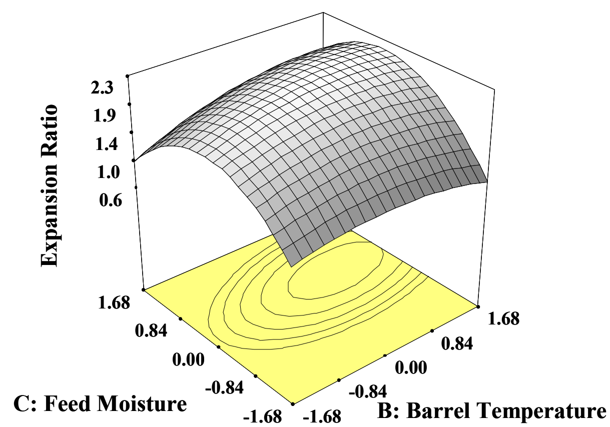


C

Figure 3.1 Effects of extrusion variables on ER

Table 3.2 Physical properties of the extruded product (mean values plus standard deviation in parenthesis)

Run	ER	BD (g/ml)	BS (N/m^2)	WSI (%)	RR (%)	L	a	b
1	2.02(0.04)	0.37(0.02)	0.75(0.12)	16.67(1.23)	135.8(8.10)	41.64(3.66)	4.08(0.50)	28.43(1.87)
2	1.59(0.03)	0.80(0.03)	0.58(0.08)	7.90(0.35)	46.66(2.78)	45.42(7.98)	3.30(0.48)	24.34(2.75)
3	1.76(0.07)	0.38(0.01)	1.09(0.07)	9.32(0.87)	76.66(4.82)	42.81(5.64)	3.57(0.32)	27.29(2.64)
4	1.55(0.04)	0.90(0.02)	0.63(0.07)	7.08(1.01)	40.60(2.39)	45.19(4.33)	2.86(0.63)	25.17(3.22)
5	1.90(0.06)	0.49(0.01)	0.94(0.14)	11.04(1.21)	99.56(6.72)	41.64(3.65)	4.15(0.22)	27.71(4.54)
6	1.41(0.02)	0.88(0.03)	0.87(0.09)	7.74(0.97)	40.30(1.87)	45.06(1.87)	3.28(0.43)	26.20(3.65)
7	1.56(0.02)	0.59(0.02)	1.19(0.21)	6.53(0.22)	68.66(1.95)	43.67(4.76)	4.42(0.51)	29.21(1.22)
8	1.39(0.10)	1.05(0.03)	0.83(0.11)	5.06(0.64)	35.73(2.63)	46.88(2.34)	2.75(0.17)	24.46(2.38)
9	2.34(0.04)	0.36(0.01)	0.82(0.08)	8.50(0.32)	82.31(9.77)	41.95(2.39)	4.04(0.32)	31.57(1.72)
10	1.58(0.02)	1.04(0.02)	1.54(0.09)	5.46(0.34)	58.50(8.38)	42.31(4.74)	4.38(0.39)	27.39(2.42)
11	2.23(0.05)	0.57(0.02)	0.86(0.12)	9.56(0.54)	74.02(5.24)	38.55(1.93)	7.58(0.62)	28.21(1.67)
12	1.84(0.11)	1.04(0.03)	1.75(0.14)	0.78(0.12)	55.44(5.73)	44.94(2.12)	3.54(0.17)	29.32(2.80)
13	1.56(0.07)	0.91(0.03)	0.85(0.16)	21.84(1.36)	142.8(9.21)	42.24(2.35)	3.66(0.26)	29.04(1.97)
14	1.36(0.08)	1.13(0.02)	0.57(0.07)	11.16(1.12)	8.25(0.96)	46.85(1.86)	3.34(0.42)	24.56(3.21)
15	2.19(0.05)	0.65(0.01)	1.08(0.12)	8.21(1.07)	71.40(6.44)	44.00(2.87)	3.75(0.25)	28.66(1.05)
16	2.16(0.05)	0.64(0.01)	0.95(0.14)	10.51(1.45)	70.39(5.24)	43.77(3.87)	3.56(0.36)	27.77(1.36)
17	2.13(0.04)	0.62(0.02)	1.04(0.10)	8.55(1.22)	72.01(7.39)	43.44(2.64)	3.48(0.22)	28.25(1.72)
18	2.11(0.02)	0.64(0.01)	1.13(0.09)	8.88(0.54)	69.82(6.21)	41.74(2.95)	4.48(0.18)	29.48(2.10)
19	2.20(0.04)	0.61(0.01)	1.07(0.11)	9.36(0.78)	71.01(6.35)	42.31(5.62)	4.45(0.15)	29.14(2.54)
20	2.16(0.05)	0.62(0.02)	1.07(0.14)	9.24(0.83)	70.65(5.78)	41.80(3.87)	4.52(0.32)	29.72(1.73)

Table 3.3 Analysis of variance (ANOVA) for the fit of experiment data to response surface model

Source	ER		BD		BS		WSI		RR		L	
	df	SOS*	df	SOS*	df	SOS*	df	SOS*	df	SOS*	df	SOS*
Model	6	1.821	4	0.805	4	1.207	7	324.795	4	17135.223	3	59.746
Residual	13	0.224	15	0.286	15	0.405	12	19.335	15	1199.373	16	18.295
R^2	0.89**		0.74**		0.75**		0.94**		0.94**		0.77**	

*SOS: Sum of squares;** P < 0.001.

Analysis of variance (ANOVA) for the p value of experiment data to response surface model

	ER	BD	BS	WSI	RR	L
Model	0.0004	0.0336	0.0150	< 0.0001	< 0.0001	0.0164
A	0.0033	0.0180	0.0136	0.0052	0.0170	0.5638
B	0.0261	0.0809	0.0104	< 0.0001	0.0027	0.0078
C	0.0088	0.0049	0.0444	< 0.0001	< 0.0001	0.0014
A2	0.0178	0.7936	0.9961	0.0174	0.7976	0.9807
B2	0.0626	0.5650	0.3715	0.0006	0.2945	0.7097
C2	< 0.0001	0.0397	0.0058	< 0.0001	0.6325	0.0273
AB	0.8796	0.7360	0.7314	0.7781	0.2720	0.4358
AC	0.9597	0.8328	0.7031	0.0950	0.2258	0.8983
BC	0.1921	0.7360	0.2819	0.0331	0.0111	0.6629

A: Screw speed, B: Barrel temperature, C: Moisture content

Feed moisture was the most significant factor affecting the ER. The effect of feed moisture on extrusion process has been observed to be complex. In this study, a quadratic effect of feed moisture on ER was found and the maximum ER was obtained when feed moisture was around 28%. Park *et al.* (1993) studied the extrusion of soy flour - corn starch - raw beef blends in a single screw extruder and reported similar results with 23% feed moisture predicted for having optimum ER. Sun and Muthukumarappan (2002) extruded corn flour and soy flour blends in a single screw extruder and also reported that ER increased with increasing feed moisture from 15% to 18% and then decreased with further increasing to 21%. With blends of full fat soy grits, corn meal, soy concentrate and soybean oil extruded in a twin screw extruder, Konstance *et al.* (2002) did not find significant change of ER when feed moisture was changed from 11% to 13% and then to 19%. Hagenimana *et al.* (2006) found that ER decreased with increasing feed moisture from 16% to 19% and 22% when rice flour was extruded in a twin screw extruder. Zasypkin and Lee (1998) reported that ER of extruded wheat flour or soy flour alone increased with lowering of the feed moisture from 18% to 17% and 16%, while for extruded blends, ER remained the same or was lower. It has been observed that suitable feed moisture depends on not only the extrusion condition, such as barrel temperature and screw speed, but also the feed composition, which can affect the water binding capacity (Park *et al.*, 1993). SPI has a high water holding capacity, which appeared to be the main cause of high optimum feed moisture in this study.

Quadratic effects of screw speed and barrel temperature on ER were also found in this study, although to a slightly lower extent as compared with feed moisture. On one hand, increasing screw speed and barrel temperature will introduce more energy to the material in the barrel, which would enhance the moisture evaporation at the die exit and hence increase ER. On the other hand, increasing screw speed will reduce the residence time of the material (Gogoi and Yam, 1994), which could reduce the energy received by the material in the barrel, and therefore could result in lower ER. Sun and Muthukumarappan (2002) reported similar results when screw speed increased from 180 rpm to 200 rpm and 220 rpm. However, Seker (2005) and Hagenimana *et al.* (2006) found that increasing screw speed steadily enhanced ER. Park *et al.* (1993) and

Hagenimana *et al.* (2006) also reported the quadratic effect of barrel temperature on ER. Sun and Muthukumarappan (2002) stated that when temperature increased from 155 to 185^{0}C, ER steadily decreased.

When the extrusion process carried out at temperatures above 100^{o}C, moisture is superheated inside the extrusion barrel due to internal high temperature and high pressure conditions (Park *et al.*, 1993; Heldman and Hartel, 1997). As the product exits the die nozzle, moisture flash evaporates suddenly due to the pressure drop. As a result, the extruded product is expanded and the characteristic texture of the extrudate is formed with a porous structure. Sufficient amount of moisture for the vaporization is necessary for the expansion of the product. However, too high a feed moisture reduces the shear strength and the energy input to the material, therefore decreases the moisture evaporation at the die exit and results in lower product expansion.

ER is an important quality parameter in products like breakfast cereals and ready to eat snack foods. In products intended for further cooking this may not be important; in fact large expansion ratio which promotes increased porosity may result in softer texture in cooked products. Hence choosing the optimal level depends on the intended product.

3.3.2 Bulk density (BD)

The fitted regression model for BD is shown in Eq. 3.8 (all independent variables in coded values) demonstrating quadratic effects only with the feed moisture.

$$BD = 0.645 - 0.124S - 0.085T - 0.158M + 0.102M^2 \text{ (g/ml)} \qquad (3.8)$$

Figure 3.2 shows the response surface plot of BD vs two independent variables at a time with the third taken at the midpoint level. Feed moisture was again the most significant factor for BD among the three, however, the influence was not as pronounced as with ER. As the feed moisture surface plot concaved at mid moisture content somewhat similarly with both temperature and screw speed indicating a lower bulk density. Park *et al.* (1993) reported similar results. However, Sun and Muthukumarappan (2002) found that with increasing the feed moisture BD increased steadily. Hagenimana *et al.* (2006) reported that BD increased with an increase in feed moisture at low barrel temperature, whereas the opposite effect occurred at high temperature.

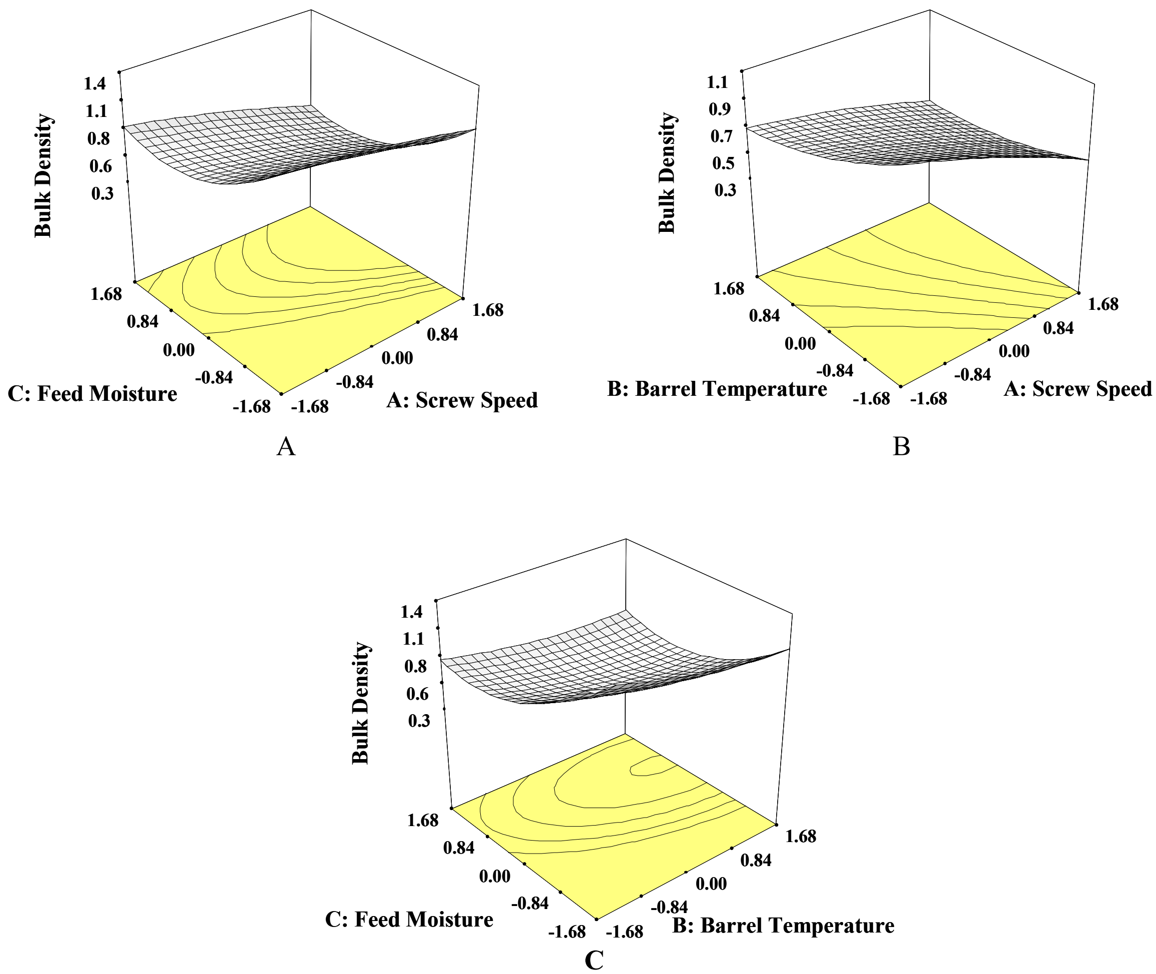


Figure 3.2 Effects of extrusion variables on BD

BD relationship with temperature and screw speed was somewhat flat showing minimal interactions, with both barrel temperature and screw speed contributing to a lower BD. Seker (2005) and Hagenimana *et al.* (2006) reported similar results High screw speed and barrel temperature could result in larger extent of starch gelatinization. Case, Hamann and Schwartz (1992) indicated that with starch gelatinization increasing, the volume of the extrudate increases and the BD decreases. But Sun and

Muthukumarappan (2002) found BD increased significantly with increasing screw speed, and no explanation was provided.

Both ER and BD represent the extent of puffing of the extrudate. Therefore, it might be expected that these two properties would be negatively correlated, with higher ER contributing to lower BD. But Park *et al.* (1993) reported that this is not always the case. The reason could be that ER only considers the expansion in the radial direction, perpendicular to extrudate flow, whereas BD considers the expansion in all directions (Falcone and Phillips, 1988). In this study, a significant inverse relationship (r = - 0.71) was found between ER and BD. Park *et al.* (1993) and Falcone and Phillips (1988) also showed a similar result.

3.3.3 Breaking stress (BS)

The mean values of BS values under different extrusion conditions are listed in Table 3.2. Analysis of variance (Table 3.3) showed that BS was significantly ($P < 0.05$) affected by screw speed, barrel temperature, feed moisture and the quadratic effect of feed moisture. The fitted regression equation for BS is shown in Eq. 3.9 (all independent variables in coded values).

$$BS = 0.645 - 0.124S - 0.085T - 0.158M + 0.102M^2 \quad (\text{N/mm}^2) \qquad (3.9)$$

Figure 3.3 shows the response surface plot of BS vs two independent variables at a time with the third taken at the midpoint level. Like the situations in ER and BD, feed moisture had a quadratic effect on BS. The combined effect of feed moisture and temperature or screw speed was much more severe on the BS than the combination of temperature and screw speed. The latter combination showed a relatively small but perceptible effect demonstrating a steady decrease in the BS as the temperature and screw speed increased. This combination produced a crispier product that was easier to break. The feed moisture relationship with BS was somewhat consistent at different temperature and screw speeds. The BS increased with an increase in moisture content but decreased with an increase in temperature and screw speed. But the curves were somewhat convex upwards suggesting some interactions. One would expect that BS would decrease with an increase in feed moisture. This would be the case normally under low temperature extrusion conditions. Under the conditions tested, high moisture promotes more explosive behavior at the exit die resulting in an increase in the ER. Higher ER results in a larger diameter sample which offers a greater resistance to breaking resulting in an increase in BS which is consistent with the experimental results. Sun and Muthukumarappan (2002) found that the shear force per unit weight of extrudate decreased with the increase of the feed moisture and decrease of barrel temperature, and it increased with increasing screw speed to a certain point and then decreased with the further increase of screw speed.

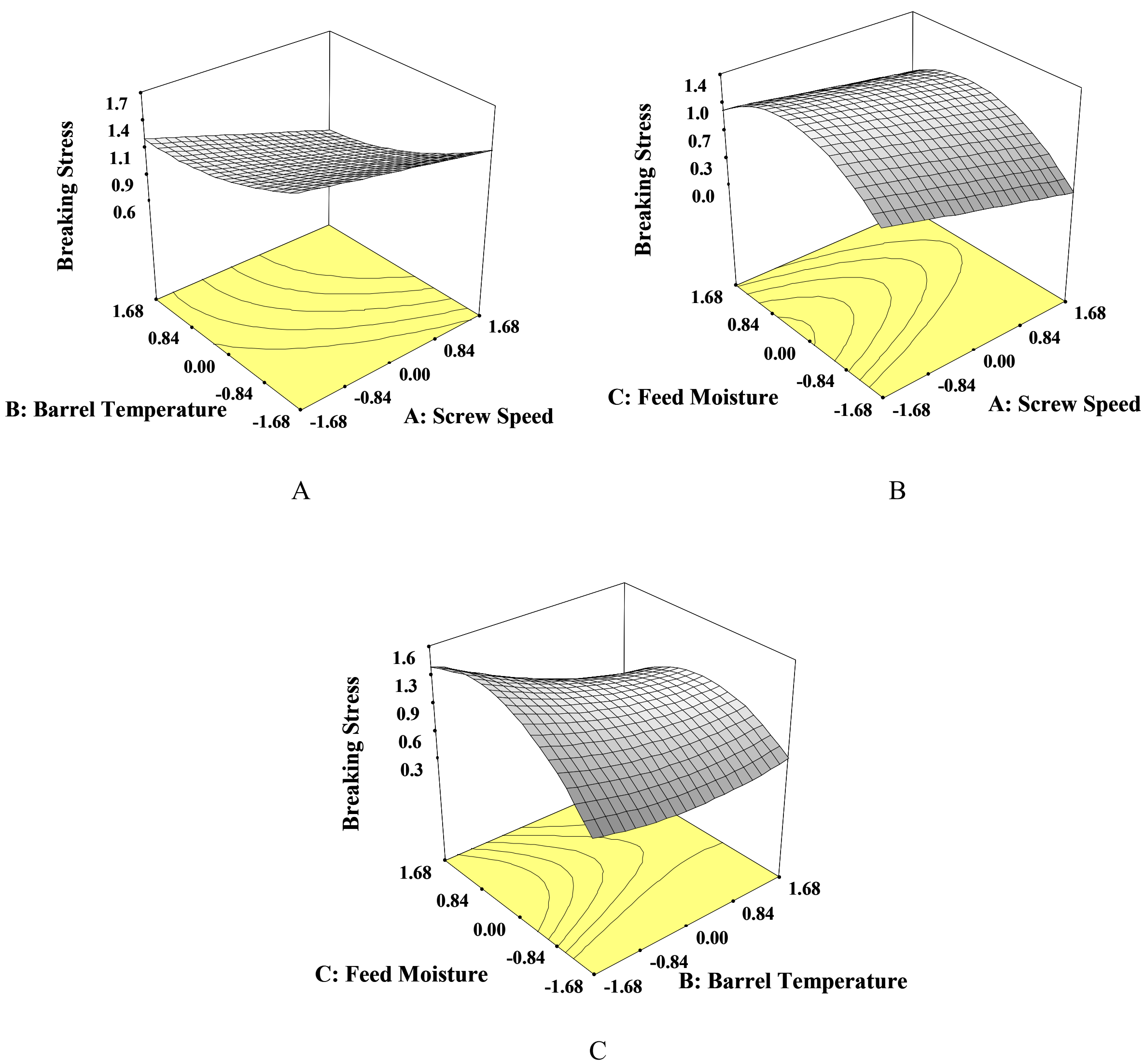


Figure 3.3 Effects of extrusion variables on BS

3.3.4 Water solubility index (WSI)

The WSI values under different extrusion conditions (Table 3.2) and the analysis of variance results (Table 3.3) showed that WSI was significantly ($p < 0.05$) affected by the individual and quadratic effects of screw speed, barrel temperature, feed moisture as well as the interaction effect of barrel temperature and feed moisture. The fitted regression model for WSI is shown in Eq. 3.10 (all independent variables in coded values).

$$WSI = 3.049 + 0.383S + 0.735T + 0.824M + 0.349T*M - 0.299S^2 - 0.512T^2 + 0.823M^2 \quad (\%) \qquad (3.10)$$

Figure 3.4 shows the response surface plot of BS vs. two independent variables at a time with the third taken at the midpoint level. Again the feed moisture was the most dominant factor. Higher screw speed and higher barrel temperatures resulted in increase in the WSI. The surface plot showed a slight convex surface with levels of temperature and screw speed at the higher end yielding the maximum WSI. Hagenimana *et al.* (2006) also reported that with barrel temperature and screw speed increased WSI increased steadily; however, Sekar (2005) reported that screw speed was not significant for WSI.

With the feed moisture the surface response plot relationship with WSI was a bit different when temperature and screw speeds were considered co-variables. In both situations, WSI increased with moisture content, with the higher end feed moisture resulting in a greater quadratic influence on the WSI. The surface plots were concave upwards. The WSI is contributed by the nature of the major components in the feed mix – the carbohydrates and proteins and their status whether they are in their native state or in the gelatinized/denatured state. At lower feed moisture levels, it is possible that there was not enough water for the starch gelatinization and protein denaturation to be completed. This could be the reason for gradual increase in WSI with an increase in moisture content. When feed moisture is higher than a certain critical level, higher feed moisture levels contribute to a greater starch gelatinization and denaturation of protein, resulting in an increased WSI. Hagenimana *et al.* (2006), however, reported that with extruded rice flour when feed moisture increased from 16% to 22%, WSI decreased. The opposite results could be due to the inclusion of higher amounts SPI in the present study. The combination perhaps helped to increase the WSI.

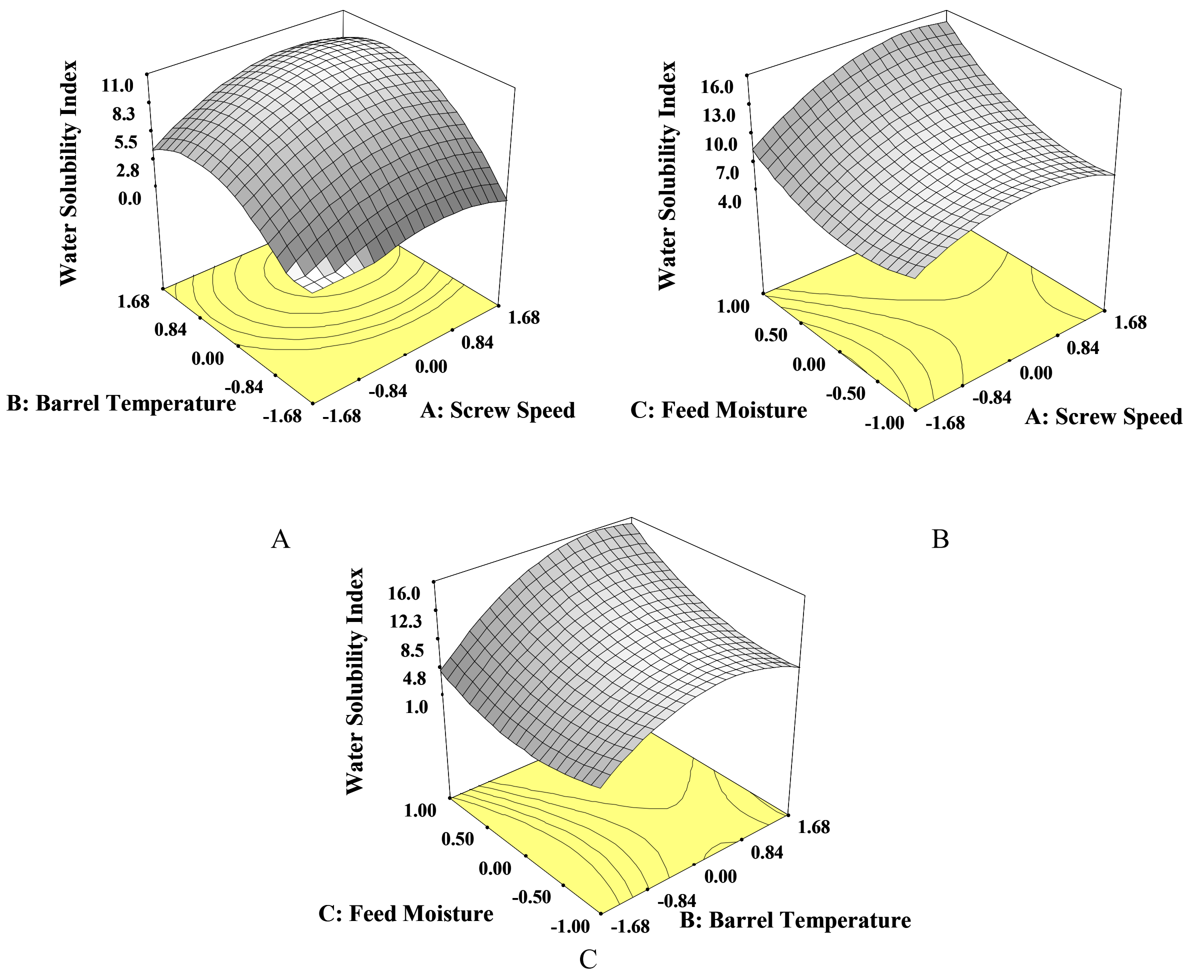


Figure 3.4 Effects of extrusion variables on WSI

3.3.5 Rehydration ratio (RR)

Extruded products are frequently rehydrated prior to consumption like in a breakfast cereal, or used as an ingredient in cooking preparations. The rehydration ratio is an important parameter for such consideration as it will define the ability of how much liquid the product can be absorb.

The rehydration ratio RR values was significantly ($p < 0.05$) affected by screw speed, barrel temperature, feed moisture, and the also the interaction effects of barrel temperature and feed moisture (Tables 3.2 and 3.3). However, there were only linear effects with all variables and no quadratic effects. The fitted regression equation for RR is shown in Eq. 3.11 (all independent variables in coded values).

$$RR = 69.526 + 6.992S + 9.658T + 32.479M + 9.927T * M \quad (\%) \qquad (3.11)$$

Figure 3.5 shows the response surface plot of RR vs. two independent variables at a time with the third taken at the midpoint level. The effects of the process variables on RR were straight forward. RR increased with feed moisture, barrel temperature and screw speed. Each of these also contributed to higher ER and lower BD. Higher ER means higher porosity of the sample and hence greater ease for rehydration. All these conditions are indicative of higher and more rapid expulsion of moisture due to the larger pressure difference and higher temperature at the die. Park *et al.* (1993) and Sun and Muthukumarappan (2002) also found higher barrel temperature to result in increased water absorption. Sun and Muthukumarappan (2002) and Seker (2005), however, did not find any significant effect of screw speed on water absorption of the extrudate. In this study, although significant, screw speed was the least important factor for RR.

Correlation analysis showed that RR was positively related to ER and WSI ($r > 0.71$). The expanded structure of the extrudate could be one of the main forces of the water absorption. RR was found negatively related to BD ($r = -0.56$). Park *et al.* (1993) also reported water absorption was reversely related with BD ($r = -0.66$).

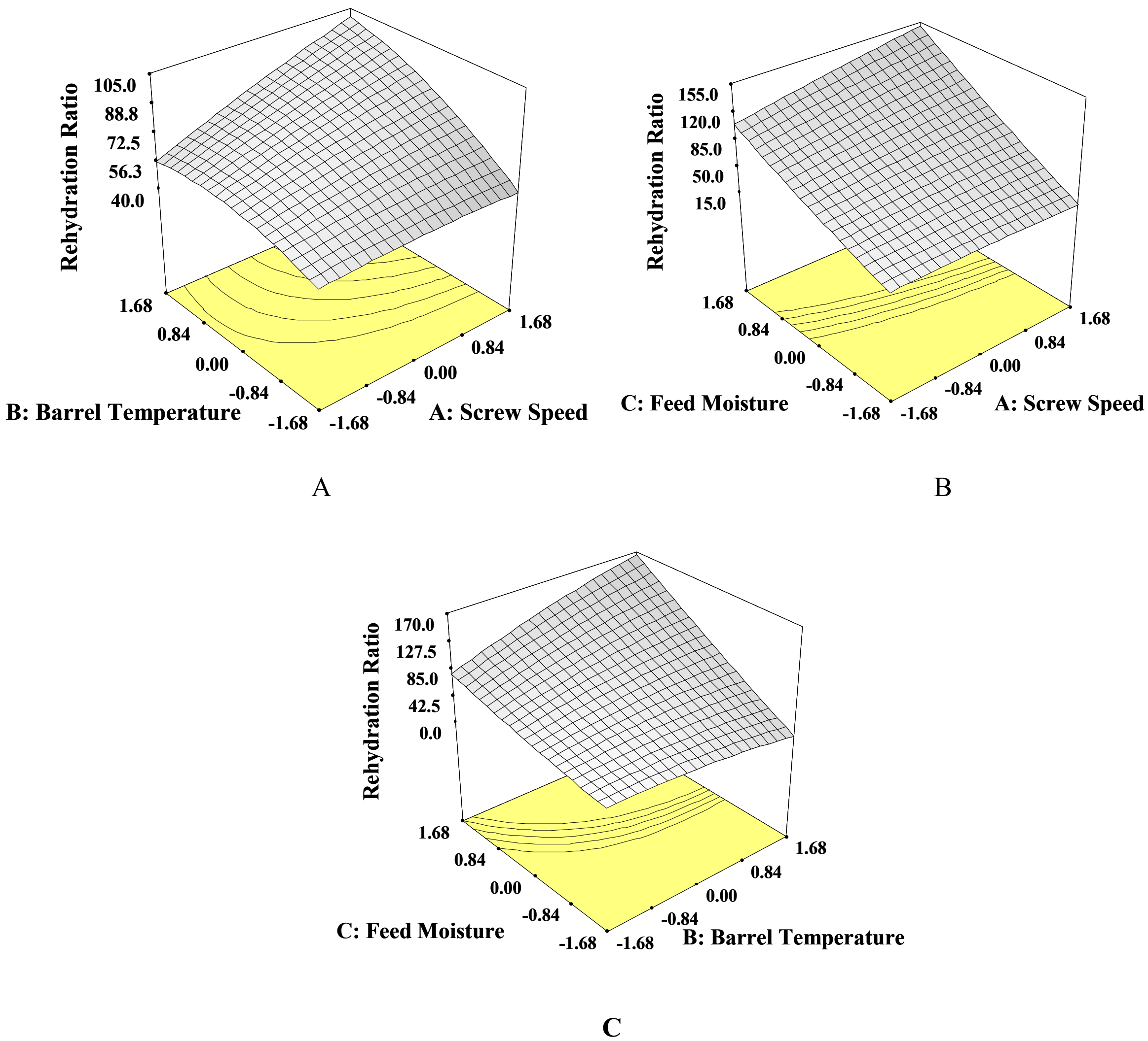


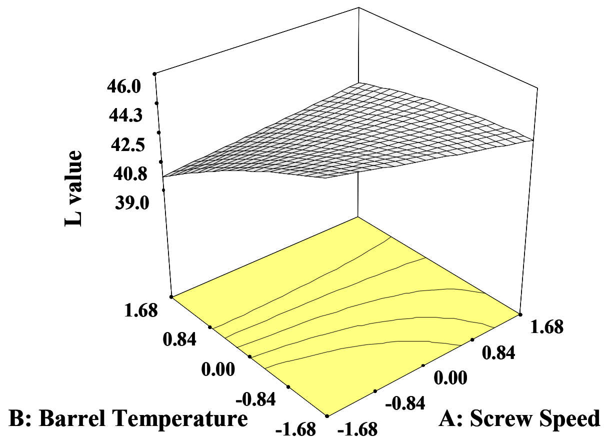
Figure 3.5 Effects of extrusion variables on RR

3.3.6 Color

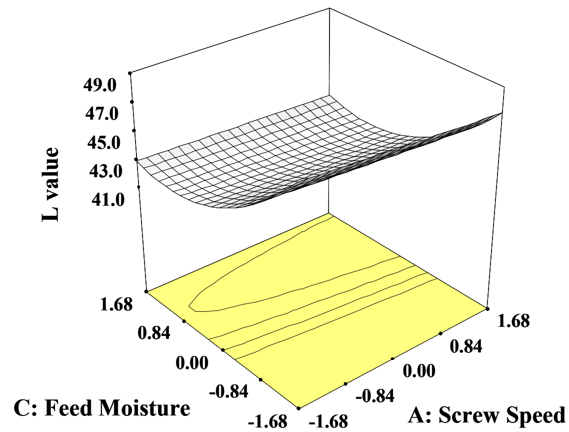
The color coordinates of L*, a* and b* under different extrusion conditions were evaluated (Table 3.2). Analysis of variance results (Table 3.3) showed that L* was significantly ($p < 0.05$) affected by barrel temperature, feed moisture and the quadratic effect of feed moisture. The b value was significantly ($p < 0.05$) affected by feed moisture and its quadratic effect while a value was significantly ($p < 0.05$) affected by only barrel temperature. Only L values were used in the regression analysis, because of the low R^2 of the regression for a and b values. L value regression equation is shown in Eq. 3.12 (all independent variables in coded values).

$$L = 42.715 - 1.137T - 1.503M + 0.872M^2 \quad (3.12)$$

Figure 3.6 shows the response surface plot of L values vs. two independent variables at a time with the third taken at the midpoint level. L value increased with barrel temperature. Sun and Muthukumarappan (2002) found insignificant effect of barrel temperature on L value. Park *et al.* (1993) reported significant interaction effects between feed moisture and barrel temperature. Feed moisture – screw speed and feed moisture – barrel temperature effects were similar. Low feed moisture was associated with higher L value (lighter color) and increasing moisture content had a quadratic effect on L irrespective of screw speed and temperature. Sun and Muthukumarappan (2002) reported that the increase of feed moisture increased L value.



A



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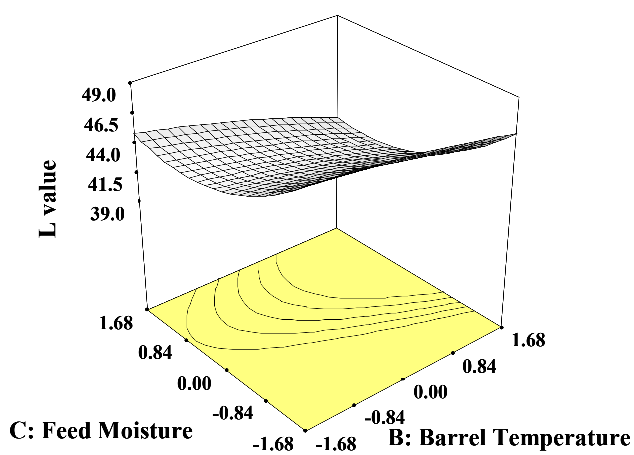

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Figure 3.6 Effects of feed moisture and barrel temperature on L value

3.4 Conclusions

Response surface methodology revealed the significant effects of all three important extrusion parameters (screw speed, feed moisture and barrel temperature) on the physical properties of twin screw extruded SPI and corn flour blends. Within the experimental range, feed moisture was the most important factor for the physical properties of the extrudate.

The effects of feed moisture on most of the properties of the extrudate were found to be quadratic; the effects were linear with some interaction only with RR. Screw speed also had quadratic effects on ER and WSI, while higher screw speeds always resulted in high RR and low BS and BD. Interactive effects of barrel temperature and feed moisture were found on WSI and RR. The effect of barrel temperature on ER was quadratic. High barrel temperature resulted in high L value and low BS and BD.

Some differences were found between the results of the present study and published results, which could be due to the different configurations of the extruder, the experiment range and more importantly feed compositions especially the combination of corn starch and SPI. Some physical properties could be expected and interrelated, but this was not always the case, revealing more studies required to fully understand the complicated extrusion process. Clearly some of the differences in the extrusion behavior was caused by the inclusion of SPI (25% level in the feed mix). Hence if protein dense products are desired, such studies should be extended to higher protein levels to assess their influence on the physical and sensory properties of the extruded product.

Connective statement to Chapter 4

Chapter 3 was the first basic study on extrusion processing in the lab and was intended to understand the relationship between some of the extrusion parameters like feed moisture, screw speed and barrel temperature on the physical properties of extruded corn flour and soy protein isolate (SPI) blends and to appropriately adjust the extrusion coordinates with respect the intended products. In order to know the status about how the materials run within the extruder, a further study on the residence time distribution (RTD) is usually carried out as the first step. Chapter 4 focuses on the RTD of this system with respect to the SPI-corn flour mixture extrusion under the general conditions explored in Chapter 3.

CHAPTER 4 RESIDENCE TIME DISTRIBUTION (RTD) OF SOY PROTEIN ISOLATES (SPI) AND CORN FLOUR FEED MIX IN A TWIN SCREW EXTRUDER

Abstract

The residence time distribution (RTD) of SPI and corn flour mixture was evaluated in a twin screw extruder using a full factorial design of experiments. The mixture was prepared by blending corn flour and SPI in the ratio of 4:1. The effect of different screw speed (75, 100 and 125 rpm), raw material moisture (25, 30 and 35%) and die diameter (3 and 5 mm) was investigated. All factors were found to have a significant ($p<0.05$) effect on the mean resident time and the variance of the residence time distribution (square of standard deviation). Higher screw speed, higher initial moisture content and larger die diameter resulted in a shorter mean residence time. Two conventional flow models were used to represent the RTD patterns in the extruder – the frequency model (F distribution) and the cumulative RTD model (E-distribution). The parameters of these models – the half concentration internal age and particle accumulation rate – were determined by a nonlinear regression. The parameters of these models were responsive to process variables and both, F and E distributions, were well predicted.

NOTATIONS

C	Particle concentration
E(t)	Exit age distribution or RTD
E(θ)	Equivalent of E(t) in a normalized form
F(t)	Cumulative RTD function
t	Time
t_m	Mean residence time
t_v	Mean Variance
θ	Normalized time (t/t,)
σ	Variance
FPRT	Fastest particle residence time

ECT	Total Extrudate Collection Time
B	Particle accumulation rate
M_C	Half-concentration internal age

4.1 Introduction

Extrusion is a simple process but yields complex results due to the large number of influencing process variables involved, such as feed rate, raw material moisture content, barrel temperature, screw speed, screw profile, die shape, die size, barrel length, barrel diameter, etc. The type of raw material and its composition add further complexity. Response variables of the process include the various physical-chemical and sensory characteristics of the product and several process related parameters such residence time distribution (RTD).

RTD is an important parameter which describes the time the material spends in the extruder. The effect of residence time on product transformation is direct since it represents the duration of the extrusion treatment. The product quality depends on the complex chemical and physical changes during the extrusion process, which are highly related to the RTD of the material, in addition to extrusion screw configurations, barrel temperature and feed characteristics. RTD studies provide information about the degree of mixing, severity and uniformity of thermo-chemical and rheological reactions that the material undergoes during its passage through the extruder. RTD itself is influenced by barrel temperature, screw speed, screw configuration, feed composition and other variables (Harper, 1989). Chuang *et al.* (2004) studied the effect of screw profile on RTD and starch gelatinization in rice flour during single screw extrusion cooking and using model systems, Kumar *et al.* (2008) evaluated a laboratory model extruder for RTD using input variables moisture content, screw speed, nozzle diameter and barrel temperature.

RTD in extrusion process are frequently evaluated by adding a colored dye or other traceable components such as salt into the raw material and monitor the color/tracer in the output extrudate (Nwabueze and Iwe, 2008). Abdelrahim *et al.* (1993b) studied the effect of variance by different particle size, starch concentration, flow rate etc. on the RTD of meat and carrot particles in a pilot scale aseptic processing system. Nwabueze and Iwe (2008) found that the total collection time increased with an increase of protein content in the formula. Bounie (1988) compared two heat-stable tracers, zinc oxide and erythrosine, in the Gamma model. Davidson *et al.* (1983) and Fichtali *et al.* (1995) used manganese dioxide as the tracer for the residence time. Lithium tracer was used by Yeh *et al.* (1992); the tracer was injected in two locations, at the feed port and at three quarters of the whole length, and the lithium tracer was measured by a flame ionization spectrophotometer. More recently, Lei *et al.* (2008) used a blue dye as the tracer to study the variables which affect the RTD.

Different base materials have been used for extrusion RTD analyses, such as wheat flour (Yeh *et al.,* 1992) and rice flour (Peng *et al.,* 1994). Kumar *et al.* (2006) compared two different measuring methods using image processing instead of a colorimeter to determine the concentration of tracer in the product and reported that the results were found to be similar.

The residence time distribution during of SPI and corn flour mixtures during twin screw extrusion processing has not been well studied. The objective of this study was therefore to evaluate the RTD of SPI and corn flour mixtures in a co-rotating twin screw extruder as influenced by three independent variables (screw speed, feed moisture and barrel temperature).

4.2 Theory of RTD

Several studies have focused on describing the RTD theory dealing with E and F functions and the different procedures to establish the RTD curves in a wider range of continuous flow systems (Levenspiel, 1972; Yeh *et al.*, 1992, Abdelrahim *et al.*, 1993a,b, Fichtali *et al.*, 1995; Ramaswamy *et al.*, 1995a,b; Iwe and Ngoddy, 2001; Chen, 2001; Nwabueze and Iwe, 2008). Both E and F curves can be obtained through the introduction of tracer particles at the entrance of a continuous flow system and then collecting them at the exit, by introducing the particles as a pulse or stepwise fashion. The following provides a brief description of the RTD theory and the parameters that are important in RTD studies:

Both the mean residence time and its variance (square of the associated standard deviation) are important characteristics of RTD. All values of particle residence times can be normalized by dividing the individual residence times by the mean residence time or can be evaluated with their real time values. The particle mean residence time, t_m, is defined by the following mathematical expression (Levenspiel, 1972):

$$t_m = \frac{\sum_{i=0}^{\infty} t_i C_i \Delta t}{\sum_{i=0}^{\infty} C_i \Delta t} \qquad (4.1)$$

The variance ($t_v = \sigma^2$) indicates the spread of the residence times, and is generally expressed along with the mean residence time as following (Levenspiel, 1972):

$$\sigma^2 = \sum_{i=0}^{\infty} (t_i - t_m)^2 E(t_i) \qquad (4.2)$$

For easier analysis and comparison purposes, normalized residence time is often used instead of real residence time, which is the ratio of the particle (or fluid) residence time (t) to that of the mean residence time of the carrier fluid (t_m) (Yeh *et al.*, 1992; Ramaswamy *et al.*, 1995a,b; Nwabueze and Iwe, 2008):

$$\theta = t/t_m, \qquad (4.3)$$

The E curve gives the output concentration (E) for any non-ideal flow which, in terms of normalized particle concentration as a function of time, can be described as shown below (Levenspiel, 1972):

$$E(t) = C_\theta / C_0 = \frac{C_\theta}{\sum_{\theta=0}^{\infty} C_\theta \Delta t} \qquad (4.4)$$

The F function represents the cumulative particle concentration at the exit stream after a time θ in the system, and is given by the following (Levenspiel, 1972)

$$F(\theta) = \int_0^\theta \mathrm{E}(\theta)\mathrm{d}\theta = \sum_0^\theta \mathrm{C}(\theta)\mathrm{d}(\theta)/\mathrm{C}_0 = \frac{\sum_{i=0}^{\theta} C_i \Delta\theta}{\sum_{i=0}^{\infty} C_i \Delta\theta} \tag{4.5}$$

For the relatively homogeneous flow patterns, which generally follow normal distribution, the above model has been successfully used to describe the RTD; however, for describing the RTD of food particles which shows some deviation from normal distribution, other models, such as a logistic module, are used. As described by Abdelrahim *et al.* (1993a) who studied RTD in a tube flow, the following model can be used.

$$F(\theta) = \frac{U}{\left(1 + e^{-B(\theta - \mathrm{Mc})}\right)} \tag{4.6}$$

The above equation is also known as autocatalytic or the inverse exponential curve (Abdelrahim *et al.*, 1993a). It was successfully used in different conditions involving tube flow of particulates fluids. In this equation, the parameter B represents the particle accumulation rate; U, the upper limit of particle concentration and M the 50% internal age. Figure 4.1 illustrates a typical sigmoid model that represents the above behavior.

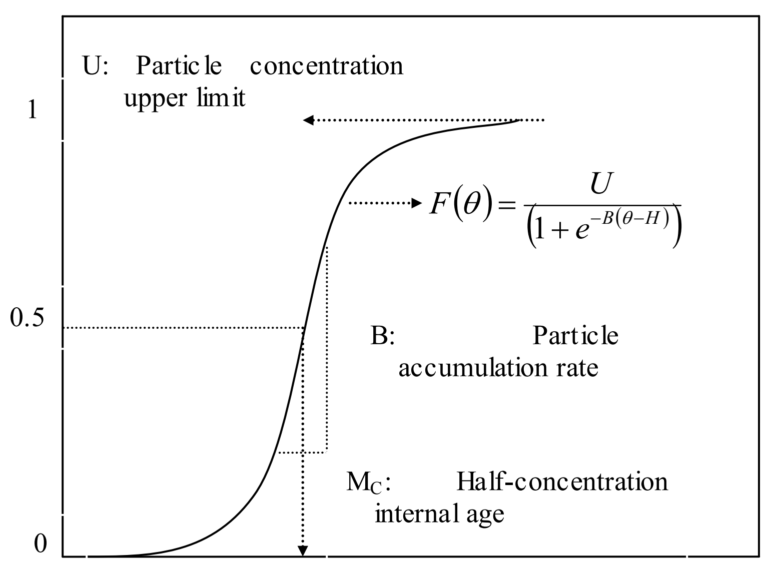


Figure 4.1 Parameters associated with the cumulative residence time distribution function using the generalized logistic model

4.3 Material and methods

4.3.1 Experiment material

Corn flour (Brar Natural Flour Mills, Winnipeg, MB) (composition: 1.7% of lipids, 76.7% of starch and 10% of protein by weight) was purchased from a local market. Soy protein isolate (SPI) was obtained from American Health and Nutrition (Ann Arbor, MI), which contained 90% protein. Sodium erythrosine (Sigma Chemical Co., St. Louis, MO) was selected as the color index. A Minolta colorimeter CM-500d (Minolta Corp., Ramsey, NJ) was used to quantify the red dye.

4.3.2 Extrusion process

Extrusion was performed in a co-rotating twin screw extruder (DS32-II, Jinan Saixin Food Machinery, Shandong, P. R. China), which consisted of three independent zones that controlled temperature in the barrel. The diameter of the screw was 30 mm, the length to diameter ratio of the extruder was 20:1 and the diameter of the hole could be set at 5 mm or 3mm (in different experiments) with a die length of 27 mm. The screw speed and the temperature of the third barrel section (metering section) were adjusted to the required levels by an automatic control system. The extruder was fed manually through a conical hopper and the flights of the screw were kept fully filled in order to avoid accumulation of the material in the hopper. The extruder heating system was controlled automatically and separated into 3 parts, the barrel temperature at the exit zone of the extruder was controlled at 150°C, the middle part was set to 135°C, and the entrance part was set to 110°C.

The moisture contents of the corn flour and SPI were measured before mixing. The corn flour and SPI were mixed by using a Hobart mixer (Hobart Corp, OH) at the ratio of 4:2 (20% SPI). Appropriate amount of water was added to adjust the mixture to the required moisture content (wet basis) and the blends were well mixed for 20 minutes before use.

A red dye, 0.02g of sodium erythrosine (Sigma Chemical Co., St. Louis, MO) and 2g of SPI/corn/water mixture were mixed as the tracer, and after the stable conditions were established, the tracer was introduced and a timer was started. Samples were collected every 10s and the appearance and disappearance of the red color was noted. Samples that did not include the tracer were used as control. The collected samples were dried overnight under mild air flow conditions at room temperature and then finish dried to moisture content of 9-10% (wet basis) by an air convection oven at 45^{o}C and an air flow rate of 0.1 m/s. Sample were ground small enough to pass through a standard sieve (#50) prior to the color determination. The red color was measured using a Minolta colorimeter (CM-500d) as the "a" value and used as a measure of the color concentration.

4.3.3 Experimental design

A full factorial design experiment was used in this study, in which three levels of screw speed (75, 100 and 125 rpm), three levels of feed moisture (25, 30 and 35% w/w) and two levels of die diameter (3 and 5 mm) were employed providing a total of 18 test runs to describe the effect of different factors on the resident time in the extrusion process. Test samples were introduced through the feeder after steady state conditions were established with a control mix without the tracer. Test samples were collected at 10 second intervals after the tracer was introduced into the hoper for each run.

The details of the experiment arrangement, fastest particle residence time (FPRT), extrudate collection time (ECT), mean residence time (t_m) and variance (t_v) were monitored and tabulated as shown in Table 4.1.

Microsoft Excel^{R} Software was used to calculate all the RTD parameters. E(t) was obtained by dividing the output concentration C_t by the total concentration C_0. The total concentration C_0 was the sum of all the C_t of the samples. F(t) was calculated from E(t), (Equation 5) as a cumulative value of E(t). The cumulative concentration up to time t can be obtained by the sum of each C_t before time t and multiplying by 10s. The total concentration of the run was the sum of all C_t multiplied by 10s. The E curve describes the transient output concentration E(t) at different extrusion times t. The F curve represents the cumulative particle concentration trend at the exit stream. The details of the experiment arrangement, fastest particle residence time (FPRT), extrudate collection time (ECT), mean residence time (t_m) and variance (t_v) were computed.

FPRT is the shortest time of the extrudate residence time in the extruder. It was recorded as the time from the beginning when the tracer was introduced to the time when the first sign of red color emerged out the extruder. ECT is the duration between the time of the first appearance of the red tracer dye at the exit until all the tracer material exited through the extruder. FPRT and ECT are tools that are useful for scaling up and for determining the optimal extrusion processing conditions. They can be used to determine the degree of cooking of raw materials and to evaluate the changes in the functionality of feed components, to estimate the extent of inactivation of enzymes or microorganisms. ECT is especially important with respect to the temperature-sensitive materials and various biopolymers yield different attributes to the product based on the FPRT and ECT together with the temperature, pressure and other process parameters. Mean and variance provide the information on how long the feed stays in the extruder, and how uniform the processing influence will be on the product quality.

4.3.4 Calculation of E(t) and F(t)

As described by equation 4.4, the E curve provides a measure of the output concentration E(t) for various extrusion times t. A curve can be obtained by calculating E(t) for various times t and finally drawing the figure.

F curve represents the cumulative particle concentration trend at the exit stream, F(t) can be calculated by equation 4.5 as outlined in the previous section. F curve can be drawn by different F(t) that we get follow equation 4.5.

As listed in equation 4.4, E(t) is obtained by output concentration C_t divided by the total concentration C_0, the concentration C_t is the reading of the a value of the sample coming out at time t minus the control a value. The total concentration C_0 is the sum of all the C_t of the samples.

F(t) is calculated by E(t), as listed in equation 4.5, F(t) is calculated by the concentration from the beginning of the run to time t divided by the total concentration of the run. The concentration up to time t can be obtained as the sum of each C_t before time t multiplied by 10s. The total concentration of the run is the sum of the entire C_t multiplied by 10s.

Table 4.1 Experiment arrangement and results of RTD experiment

Run	**Feed moisture (W/W)**	**Screw speed (rpm)**	**Die diameter (mm)**	**FPRT**	**ECT**	**t_m**	**σ^2**
1	**30**	**75**	**5**	**30**	**100**	**67.42**	**33.37**
2	**30**	**100**	**5**	**20**	**80**	**48.18**	**20.94**
3	**30**	**125**	**5**	**10**	**70**	**39.89**	**13.37**
4	**30**	**75**	**3**	**30**	**120**	**76.00**	**42.64**
5	**30**	**100**	**3**	**20**	**100**	**55.30**	**26.66**
6	**30**	**125**	**3**	**10**	**80**	**43.99**	**16.59**
7	**35**	**75**	**3**	**30**	**90**	**61.51**	**26.58**
8	**35**	**100**	**3**	**20**	**80**	**45.67**	**17.59**
9	**35**	**125**	**3**	**10**	**60**	**35.01**	**9.79**
10	**25**	**75**	**3**	**40**	**100**	**86.64**	**52.30**
11	**25**	**100**	**3**	**20**	**110**	**64.05**	**42.00**
12	**25**	**125**	**3**	**10**	**80**	**54.15**	**25.28**
13	**35**	**75**	**5**	**30**	**80**	**61.14**	**16.46**
14	**35**	**100**	**5**	**20**	**60**	**41.64**	**13.50**
15	**35**	**125**	**5**	**15**	**70**	**34.81**	**12.40**
16	**25**	**75**	**5**	**30**	**100**	**77.40**	**34.82**
17	**25**	**100**	**5**	**20**	**100**	**59.94**	**31.35**
18	**25**	**125**	**5**	**15**	**90**	**44.75**	**21.42**

Table 4.2 ANOVA (P Values) of FPRT, ECT, t_m and mean variance

	FPRT	ECT	t_m	σ^2
Model	0.0140	0.0032	0.0005	0.0003
S	0.3989	0.0009	0.0003	< 0.0001
M	0.0007	0.0011	< 0.0001	< 0.0001
D	1.0000	0.0114	0.0059	0.0006
SM	0.6480	0.0329	0.4405	0.0149
SD	0.6400	0.0434	0.1244	0.0376
MD	0.2500	0.0230	0.8256	0.0082

S: screw speed; M: moisture; D: die diameter

4.4 Results and discussion

4.4.1 Significance of factors on RTD parameters

The fastest particle residence time (FPRT) ranged from 10 to 40 s and the extrudate collection time (ECT) ranged from 60 to 120 s (Table 4.1). On the other hand, mean residence time (t_m) ranged from 35 to 87 s with variance ranging from 13 to 53 s depending on different processing conditions.

Analysis of Variance (ANOVA Table 4.2) showed that the developed models were significant (p<0.05) for all output variables, FPRT, ECT, t_m and t_v. The feed moisture content had a significant (p<0.05) effect on FPRT, while the other two factors (die diameter and screw speed) were not statistically significant (p>0.05). On the other hand, ECT was influenced significantly (p<0.05) by all three factors and their interactions. The mean residence time was also significantly (p<0.05) influenced by all three factors; however, none of their interactions was significant (p>0.05). The mean residence time (t_m) of the SPI and the corn flour mixture was significantly (p<0.05) influenced by the three variables with the moisture having the highest effect, while the interactions were not significant (p>0.05). The variance of the SPI and the corn flour mixture (t_v) was also significantly ($p<0.05$) influenced by the three variables. Screw speed and feed moisture had a higher significance and hence were responsible for the spread of residence time distribution curve of the sample. All the two-way interactions (screw speed/moisture content, screw speed/die diameter, moisture content/die diameter) were significant ($p<0.05$) with ECT and mean variance. ANOVA table is good to test the significance of the influence of different factors on the output variables; however, response plots are necessary to understand the nature of their influence. Since the different factors had an overall influence on process variables, these are described below in the form of different plots taking two factors at a time, keeping the third at selected levels.

4.4.2 Mean Residence Time and its Variance

Based on the RSM design and ANOVA discussed in the previous section, the models used for mean residence time (t_m) and variance (t_v) were significant ($p<0.05$) and explained more than 90% of the experimental variability.

$$t_m = \frac{\sum_{i=0}^{\infty} t_i C_i \Delta t}{\sum_{i=0}^{\infty} C_i \Delta t} \tag{4.7}$$

$$\mathrm{t_v} = \sigma^2 = \sum_{i=0}^{\infty} (t_i - t_m)^2 E(t_i) \tag{4.8}$$

4.4.2.1 Mean Residence Time

Figure 4.2 shows the effect of screw speed and feed moisture content on mean residence time with the diameter of the die at 3 mm (Figure 4.2-A) and 5 mm (Figure 4.2-B) respectively. Figure 4.2 shows that at the same die diameter, t_m decreased as the screw speed increased. With 3mm die diameter, increasing the screw speed from 75 to 125 rpm resulted in t_m decreasing t_m from 87 to 54 s at a feed moisture level of 25%. Same trends were observed when the moisture contents were increased to 30 and 35%. The trends were also similar with the extrusion process using a 5 mm die. Since one of the primary functions of the screw is to push the product forward through the extruder, it can be found that increasing screw speed resulting in a reduction in the mean resident time of the extrudate. Others studies have reported similar observations. For example, De Ruyck (1997) reported the RTD to increase considerably as the screw speed increased. Also, Singh and Rizvi (1998) found that higher screw speed decreased the t_m in the CO_2 injection process. Furthermore, Yeh and Jaw (1998) observed similar results when they employed rice flour in a single screw extruder.

Figure 4.2 also shows that, at both illustrated die diameters, a decrease in moisture content resulted in an increase in the mean resident time. For the 3mm die diameter, a decrease in the moisture content from 35 to 25% caused the t_m to increase from 62 to 87 s at a screw speed of 75 rpm. Similar trends were observed at the higher screw speeds of 100 and 125 rpm. Similar results can also be seen using the 5 mm die system. Moisture is an important factor in the RTD distribution of particles. As noted previously while discussing the ANOVA in Table 4.2, it is the most significant factor with respect to the t_m. Altomare and Ghossi (1986) reported that the moisture content had little effect on the residence time, while Gogoi and Yam (1994) reported that moisture content slightly affected the mean residence time. These results differ from our study, which might have been the result of the use of different raw materials, the proportion of starch to protein ratios or simply different designs of the extruder system. Our study findings matched the ones reported by Nwabueze and Iwe (2008) who found that the residence time in the extruder increased when the feed moisture decreased. Higher moisture content in the sample could have contributed to a higher pressure within the system, which accelerated the forward push of the product with increased screw speed. Higher moisture content also helped to enhance the starch gelatinization and denaturation of proteins, which generally contribute to product swelling, resulting in an increased system pressure. This would cause an increased pressure differential across the die, contributing to a higher flow rate and lower residence time.

4.4.2.2 Variance of t_m

Increasing the screw speed reduce the variance of the mean residence time (t_v) significantly (p<0.05), as reported in Table 4.2. As shown in Figure 4.2, when using a 3 mm diameter die, increasing the screw speed from 75 rpm to 125 rpm caused the variance to decrease from 52 to 25 s at feed moisture of 25%. similar trends were observed at the 30 and 35% moisture contents as well as when the larger 5 mm diameter die was used; however, in terms of magnitude, there were all significantly (p<0.05) different.

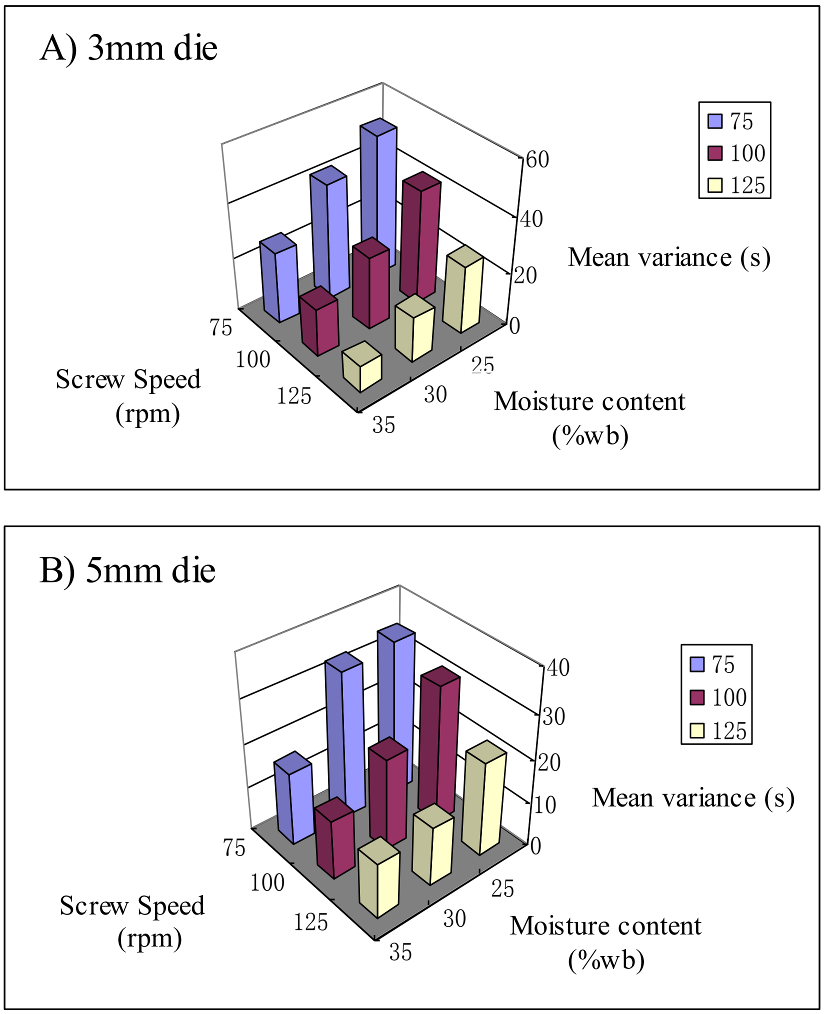
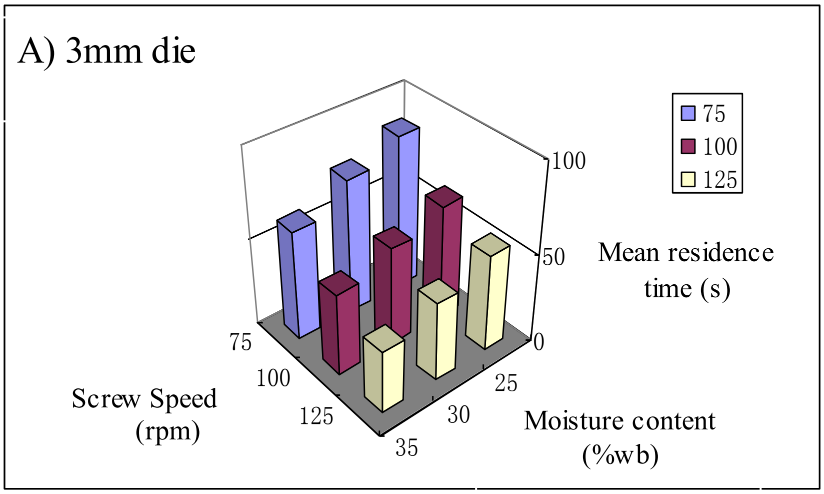


Figure 4.2 Effect of screw speed and moisture to mean residence time(t_m) under different die diameter. a) 3mm die, b) 5mm die

As with the mean residence time, the mean residence time decreased with an increase in the feed moisture content as shown in Figure 4.3. With a 3 mm diameter die, decreasing the moisture content from 35 to 25% caused the mean residence time increase from 62 to 87s at a screw speed of 75 rpm. Similar trends were observed at other screw speeds and the other die diameter. In general, the mean residence time and its variance were related.



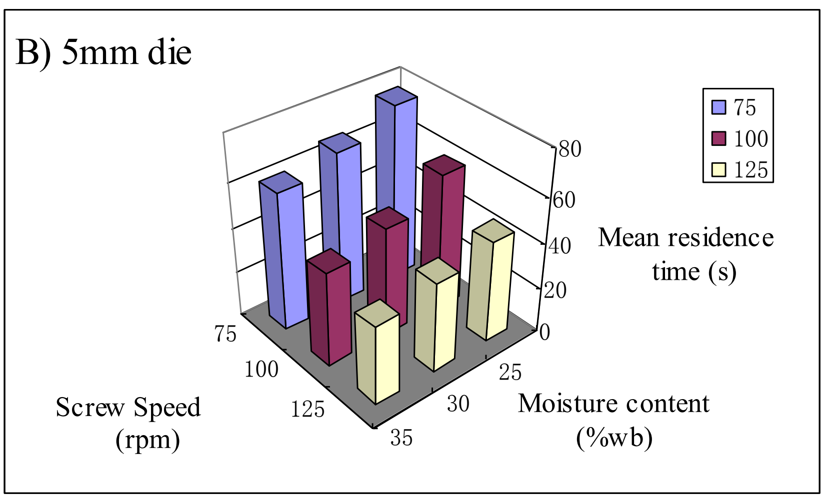


Figure 4.3 Effect of screw speed and moisture to mean variance under different die diameter. a) 3mm die, b) 5mm die

4.4.3 RTD E and F curves

4.4.3.1 Influence of Screw Speed on RTD

Figure 4.4 shows the effect of screw speed on the E-curve and F-curve using various operating conditions. The screw speed was maintained at 75rpm, 100rpm and 125rpm. On the other hand, different moisture contents (25%, 30%, 35% W/W) in the raw material was used in order to check the effect on the resident time distribution at different conditions using the die diameter of 5 mm. Increasing the screw speed reduced the mean residence times significantly (p<0.05) (Table 4.2, Figure 4.2) and shifted the residence time distribution E curve and F curve to the left (Figure 4.4).

Figure 4.4-A shows the resident time distribution for extrusion at 3 different screw speeds using the 5 mm die while the raw material moisture content was controlled at 25%. At the highest screw speed (125 rpm), the sample showed the highest E(t) and narrowest E curve spread, while the FPRT was the shortest within these limits. On the contrary, at 75 rpm sample's highest E(t) was the lowest and it also gave the widest spread of E curve and the longest FPRT. Figure 4.4-B and 4.4-C are E-distributions at the other two moisture contents. Similar results were obtained, as shown in Figure 4.4-A. The same conditions were also tested for E-distribution with the 3 mm die diameter and similar trends were observed, however with some quantitative differences. Unlu and Faller (2002) found that the regular spreads of the distributions decreased with increasing feed rate, but they increased with increasing screw speed. They concluded that the effect of the screw speed is similar to our findings.

Figure 4.4 D-F represent the F curve (accumulation of particles) for different screw speeds (75rpm, 100rpm and 125rpm) for 25% moisture samples with the 5mm diameter die. The 125 rpm sample was the first sample to reach the 100% accumulation, while the75 rpm one spent the longest time to reach the 100% accumulation. Figure 4.4-E and Figure 4.4-F show the effect of different screw speeds on the F curve. Similar trend was observed in Figure 4.4-D. Other 9 trials also tested the effect of different screw speeds on the F(t), which were tested using a 3mm diameter die and different moisture content (25%, 30% and 35%) Similar trend was observed when using a 5mm diameter die.

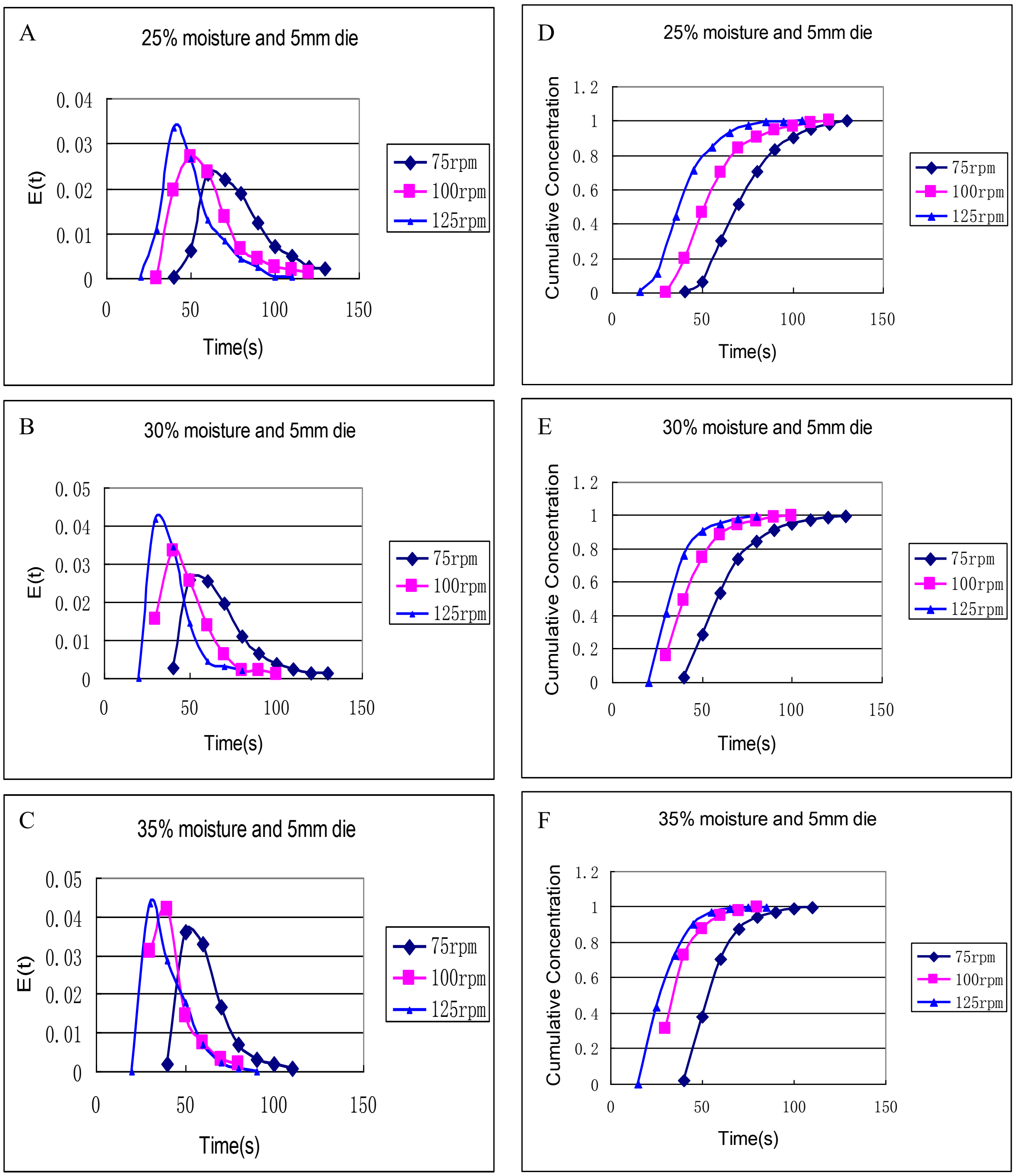


Figure 4.4 Impact of screw speed on E(t) (Fig. A, B, C) and F(t) (Fig. D, E, F)

4.4.3.2 Influence of Feed Moisture on RTD

Figure 4.5 shows the effect of different feed moisture contents on the E-curve and F-curve at different screw speeds with the 3 mm diameter die. Figure 4.5 frames, A, B and C, illustrate the impact of different moisture contents on the E-distribution while frames D, E, and F show the effect on F-distribution.

As shown in Figure 4.5-A, the test sample with 35% moisture content had the fastest FPRT and the narrowest spread distribution at a screw speed of 75 rpm. Moisture content was found to be significant (p<0.05) for FPRT, ECT, t_m and mean variance (Table 4.2). Higher moisture content decreased the FPRT and mean variance. In Figures 4.5-B and 4.5-C, show similar effects at 100 and 125 rpm. While the qualitative nature is similar the peak E(t) value increased with increasing screw speed and so is the spread. The other 9 tests with 5 mm diameter die also showed similar qualitative results (not shown).

Figure 4.5-D is the F curve (accumulation of particles) with at 75 rpm and 3 mm die for the sample with the three moisture contents 25, 30, and 35%. As with the E-distribution, the 35% moisture sample had the shortest time to reach the 100% accumulation, while the 25% moisture sample spent the longest time to go out of the extruder. Figure 4.5-E and 4.5-F demonstrate similar trends at the other rotation speeds. Increasing the moisture content in the sample resulted in a more rapid accumulation of the particles and a higher peak of the normalized output concentration, and in shorter residence time of the sample in the extruder.

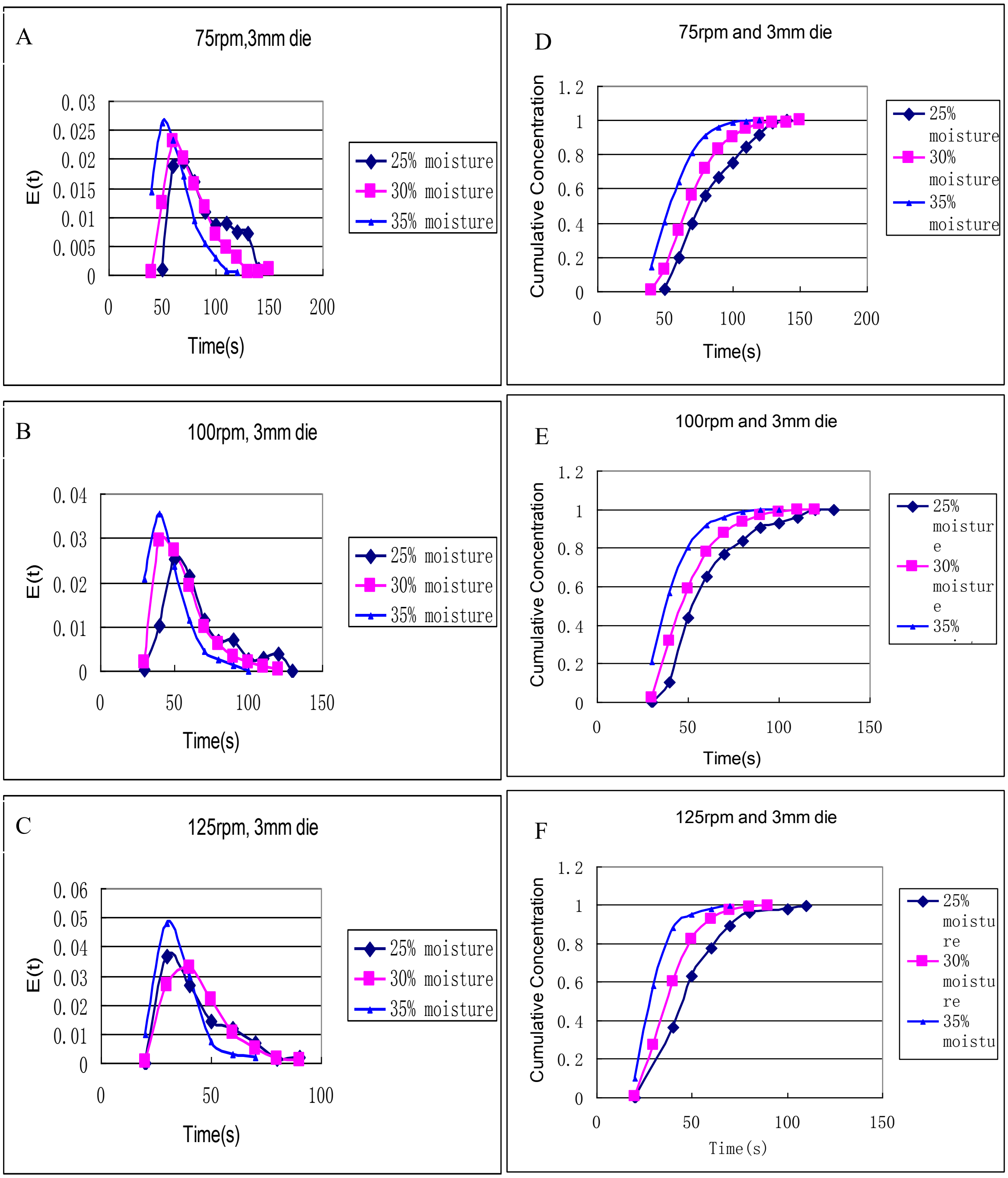


Figure 4.5 Impacts of moisture content on E(t) (Fig. A, B, C) and F(t) (Fig. D, E, F)

4.4.3.3 Influence of Die diameter on RTD

Figure 4.6 shows the effect of die diameter on the E and F curves at different feed moisture contents but at the intermediate screw speed of 100 rpm operating conditions. Figure 4.6-A is the E-curve under the extrusion processing condition of 100 rpm screw speed and 25% feed moisture content. The E(t) curve of the product with 5 mm die demonstrated a slightly higher peak and a decreased spread than the one with 3mm die. Figure 4.6-D and 4.6-E show similar curves at the other two moisture contents. Figures 4.6-D, 4.6-E and 4.6-F show that the cumulative concentration curve of 5 mm die sample reached the peak value slightly faster than the 3 mm die sample. In general, increasing the die diameter resulted in a slightly faster completion of the extrusion process and a tighter spread in RTD.

4.4.4 Modeling of E and F Distribution

The autocatalytic or the inverse exponential model (Equation 4.6) was fitted to the experimental values (Equation 4.5) and the associated RTD parameters (B and M values) were determined. The regression details are shown in Table 4.3 and the associated R^2 was higher than 0.98 for each case. E(t) curves are obtained by differentiating the F(t)-curves. The predicted F and E curves are compared with their experimental counterparts in Figures 4.7 and 4.8 demonstrating an excellent fit for the F-distribution and a slightly larger spread, but nevertheless a high R^2 of 0.91 for the E-distribution, both demonstrating good distribution of points around the diagonal line.

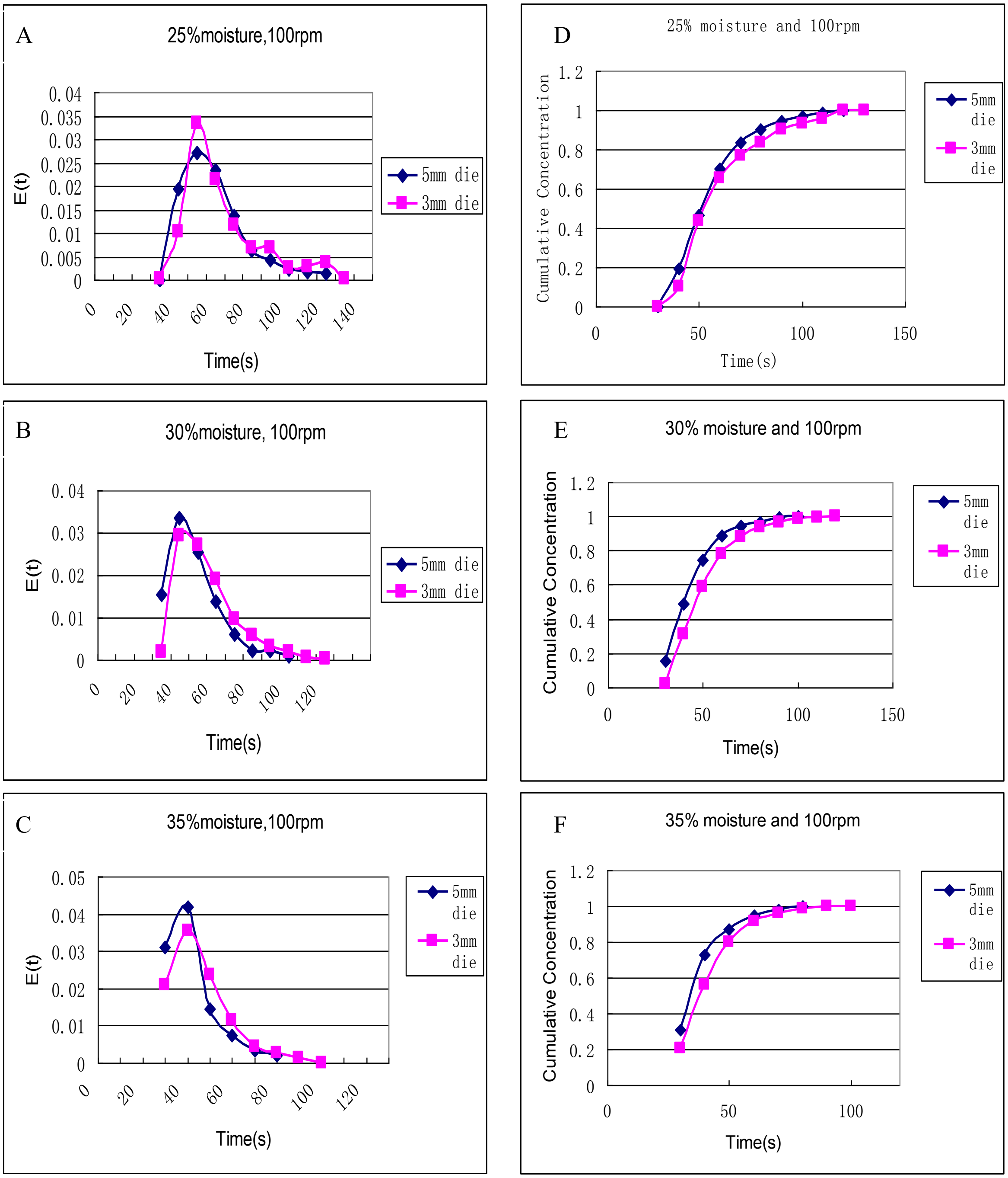


Figure 4.6 Impacts of die diameter on E(t) (Fig. A, B, C) and F(t) (Fig. D, E, F)

Table 4.3 B, Mc value and the comparison (R^2) of F(θ) and the predicted F

Run	Feed moisture (W/W)	Screw speed (rpm)	Die diameter (mm)	B	M_C	R^2
1	30	75	5	7.4695	0.8769	0.990
2	30	100	5	6.3614	0.8498	0.995
3	30	125	5	5.7698	0.8126	0.989
4	30	75	3	6.8986	0.8965	0.994
5	30	100	3	6.6975	0.8596	0.989
6	30	125	3	6.4445	0.8484	0.994
7	35	75	3	6.3942	0.887	0.996
8	35	100	3	6.2179	0.8477	0.996
9	35	125	3	6.0911	0.8163	0.997
10	25	75	3	6.0620	0.9106	0.981
11	25	100	3	5.8278	0.8873	0.983
12	25	125	3	5.8021	0.8393	0.994
13	35	75	5	7.0227	0.8813	0.991
14	35	100	5	6.9421	0.8228	0.991
15	35	125	5	5.8913	0.8051	0.985
16	25	75	5	7.8078	0.9013	0.994
17	25	100	5	7.0583	0.8638	0.993
18	25	125	5	5.9893	0.8419	0.994

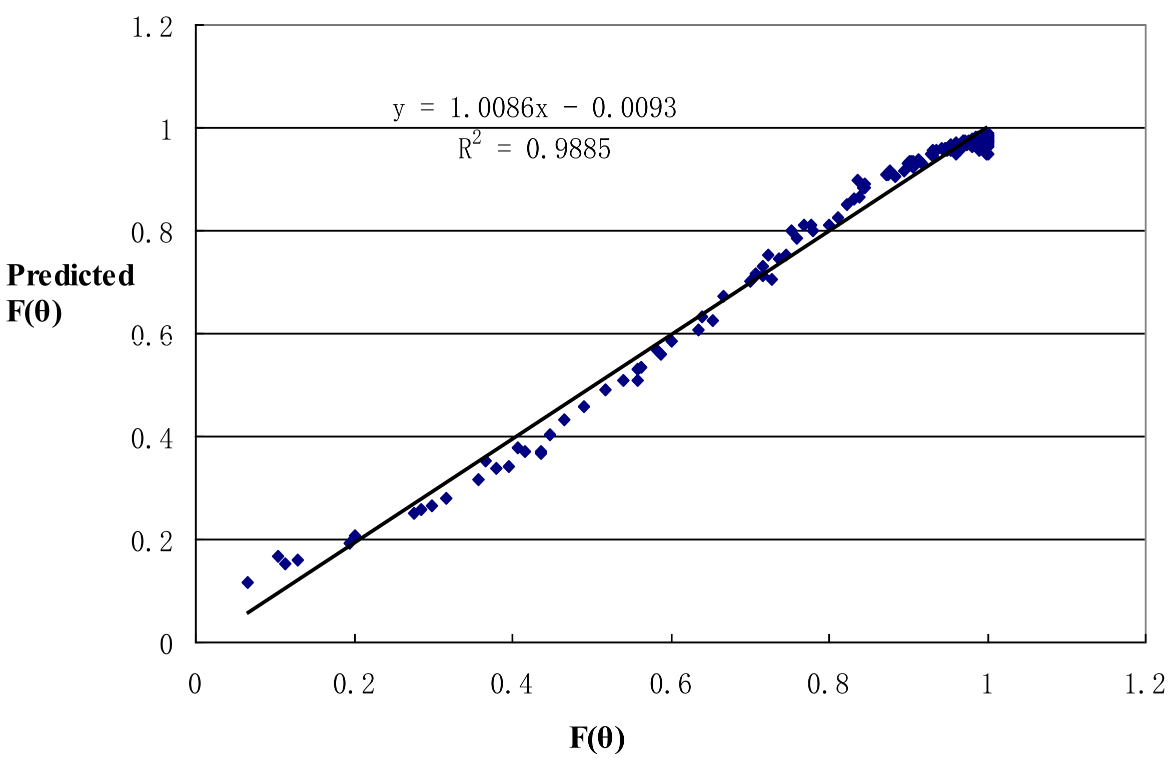


Figure 4.7 Comparison of F(θ) and Predicted F for various extrusion conditions

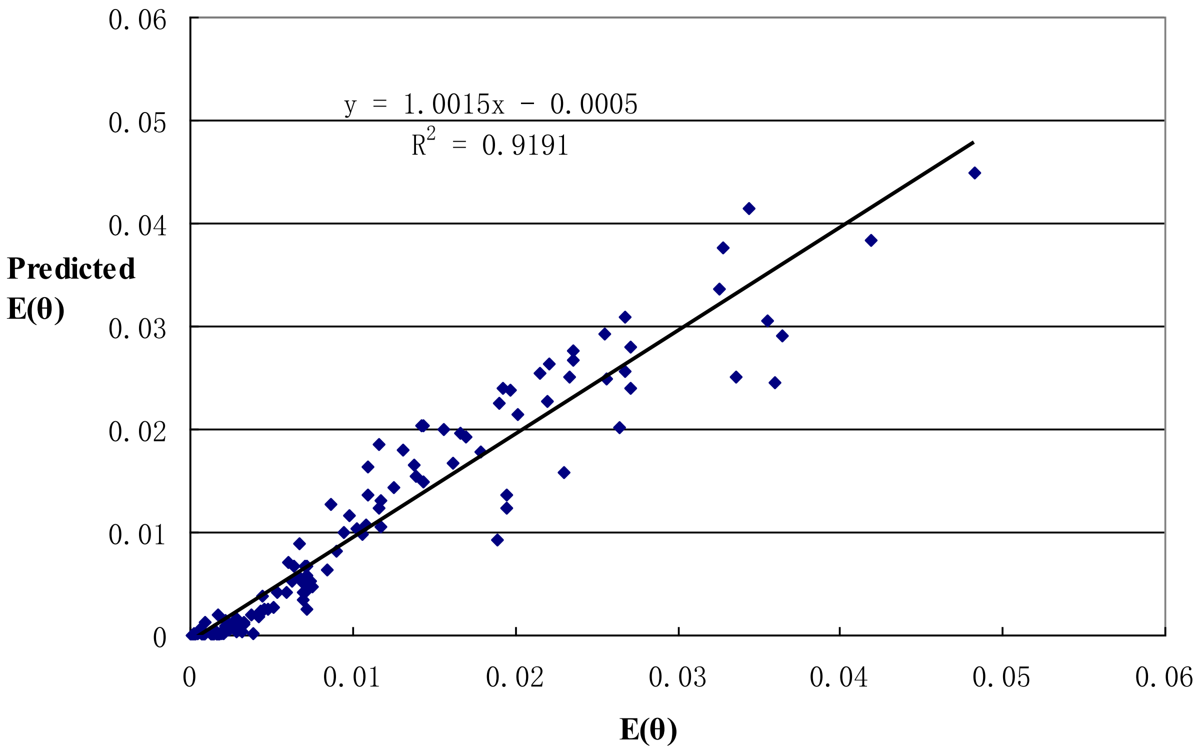


Figure 4.8 Comparison of E(θ) and Predicted E under different extrusion conditions

4.4.5 B and M_C value of the predicted F

From the previous results it was evident that the autocatalytic model accurately described the residence time distribution of the particles through the extrusion process. The two parameters, particle accumulation rate (B) and the half-concentration internal age (M_C) could be adequately used to predict the F and E distributions (Figures 4.7 and 4.8). Both B and M_C are dependent on extrusion process variables – screw speed, feed moisture content and die diameter. The conditions listed in Table 4.3 are from the factorial design. Table 4.4 shows the ANOVA of the B and Mc, the result indicating that that two factors, screw speed (S) and die diameter (D), were significant for both B and Mc, while the feed moisture content (M) was significant with M_C. The interactions of screw speed/die diameter, moisture content/die diameter were also significant for B ($p<0.05$). The fitted regression models for B and M_C are shown below as the model equations (all independent variables in coded values), where as the S stands for screw speed, W stands for moisture content, D stands for diameter of the die.

Table 4.3 listed the B and M_C values which associated with the predicted F value (expression 4.6), software *Prism 5 for Windows* was used to evaluate all the B and M_C values. Table 4.4 presents the ANOVA of the B and M_C, the result shows that two factors, screw speed (S) and die diameter (D), are significant affects both B and Mc, whereas moisture significant affects Mc. The interactions of screw speed/die diameter, moisture content/die diameter significant affect the B ($p<0.05$).

The fitted regression models for B and M_C are shown as the following equations (all independent variables in coded values), and the S stands for screw speed, M stands for raw material moisture content, D stands for diameter of the die.

$$\begin{aligned} B = {} & 6.486 + 0.456S + 0.032S^2 - 0.061W + 0.121W^2 \\ & + 0.215D + 0.054SM - 0.013S^2M + 0.121SM^2 - 0.109S^2M^2 \\ & + 0.276SD + 0.054S^2D + 0.312MD - 0.289M^2D \end{aligned} \tag{4.9}$$

$$\begin{aligned} \mathrm{Mc} = {} & 0.8582 + 0.034S + 0.0031S^2 + 0.0158M + 0.0009M^2 \\ & + 0.0076D + 0.0021SM + 0.0046S^2M + 0.0046SM^2\, 0.0005S^2M^2 \\ & - 0.0019SD + 0.0021S^2D + 0.0026MD + 0.0032M^2D \end{aligned} \tag{4.10}$$

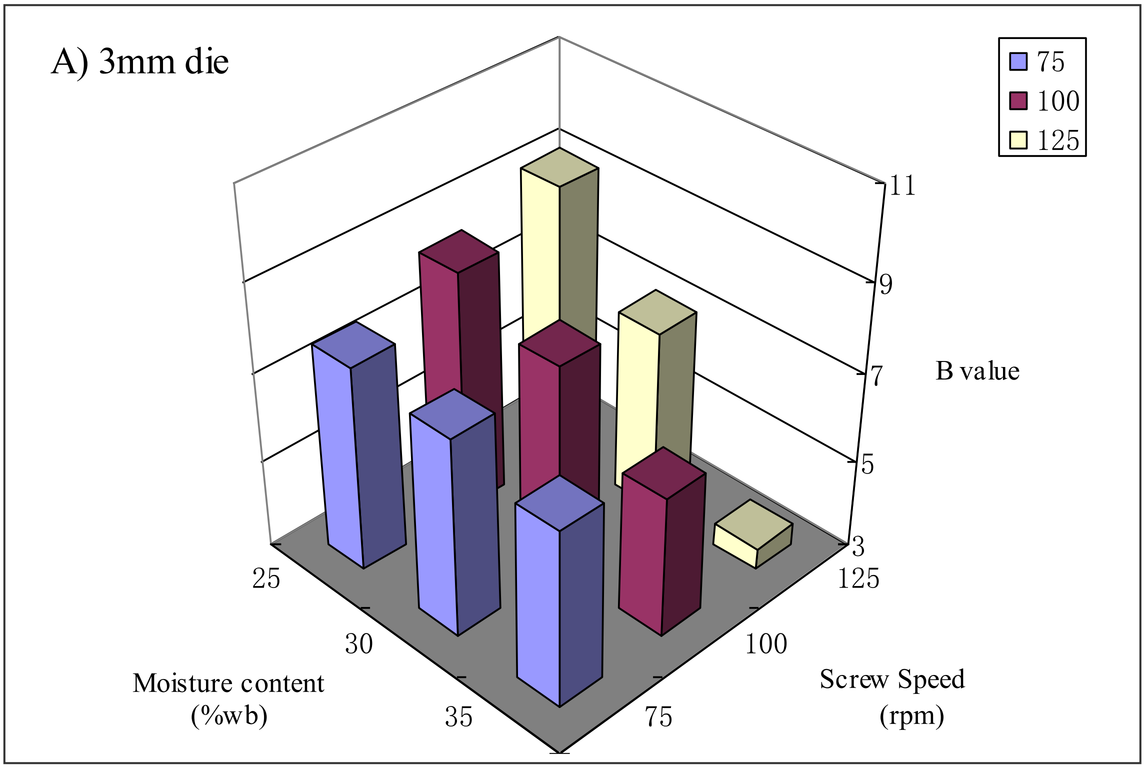
Figure 4.9 shows 3-D bar graphs of model predicted B values as a function of screw speed and feed moisture content for the two die diameters. The particle accumulation rate (B values) decreased slightly as the feed moisture content increased at the screw speed of 75 rpm. However the trend was reversed if the screw speed was increased to 100 and 125 rpm. Similarly, the B values increased slightly with screw speed at the 25% feed moisture, but the trend was reversed when the moisture content increased to 35% demonstrating some interaction effects. The trends were similar at the two die diameters, but the accumulation rates were faster at the larger die diameter.

Figure 4.10 shows 3-D bar graphs of model predicted M_C values as a function of screw speed and feed moisture content for the two die diameters. The trends were more clearly defined with respect to the half concentration internal age (M_C). With both die diameters, M_C values clearly increased with an increase in screw speed. While Mc values slightly increased with increase in moisture content with the 5 mm die, it was not well defined with the 3 mm die.

Table 4.4 ANOVA of B and M_C of the predicted F

	B			M_C		
Source	Sum of Squares	DF	p-Value	Sum of Squares	DF	Prob > F
Model	6.023	13	0.0179	0.017	13	0.0114
S	2.685	2	0.0038	0.013	2	0.0009
M	0.132	2	NS	2.8E-03	2	0.0149
D	0.835	1	0.0123	1.0E-03	1	0.0311
S*M	0.159	4	NS	2.9E-04	4	NS
S*D	1.126	2	0.0185	4.7E-05	2	NS
M*D	1.087	2	0.0197	1.1E-04	2	NS
Residual	0.177	4		3.9E-04	4	
Cor Total	6.201	17		0.017	17	
C.V.	3.246			1.157		
R-Squared	0.971			0.977		
Adj R-Squared	0.878			0.904		
Pred R-Squared	0.421			0.543		
Adeq precision	10.983			12.015		

S: screw speed; M: moisture; D: die diameter



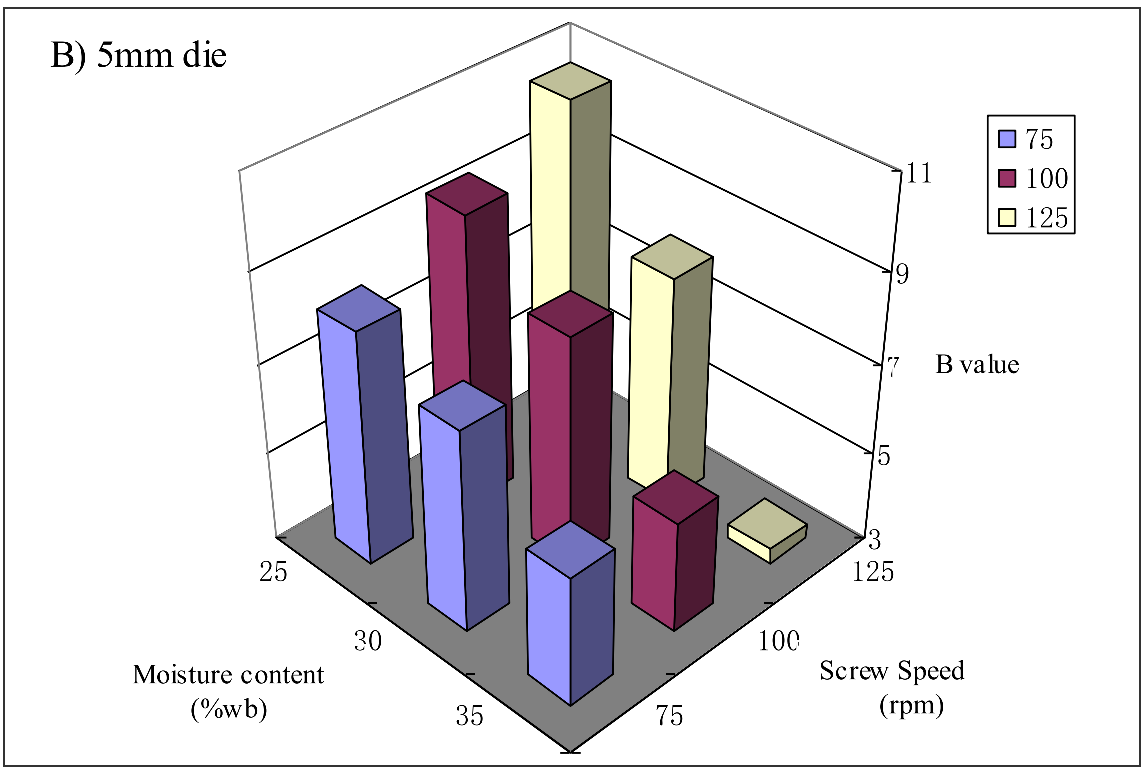


Figure 4.9: Model predicted effect of screw speed and moisture on B value at different moisture content and rpm with a) the 3mm diameter or b) the 5 mm diameter die

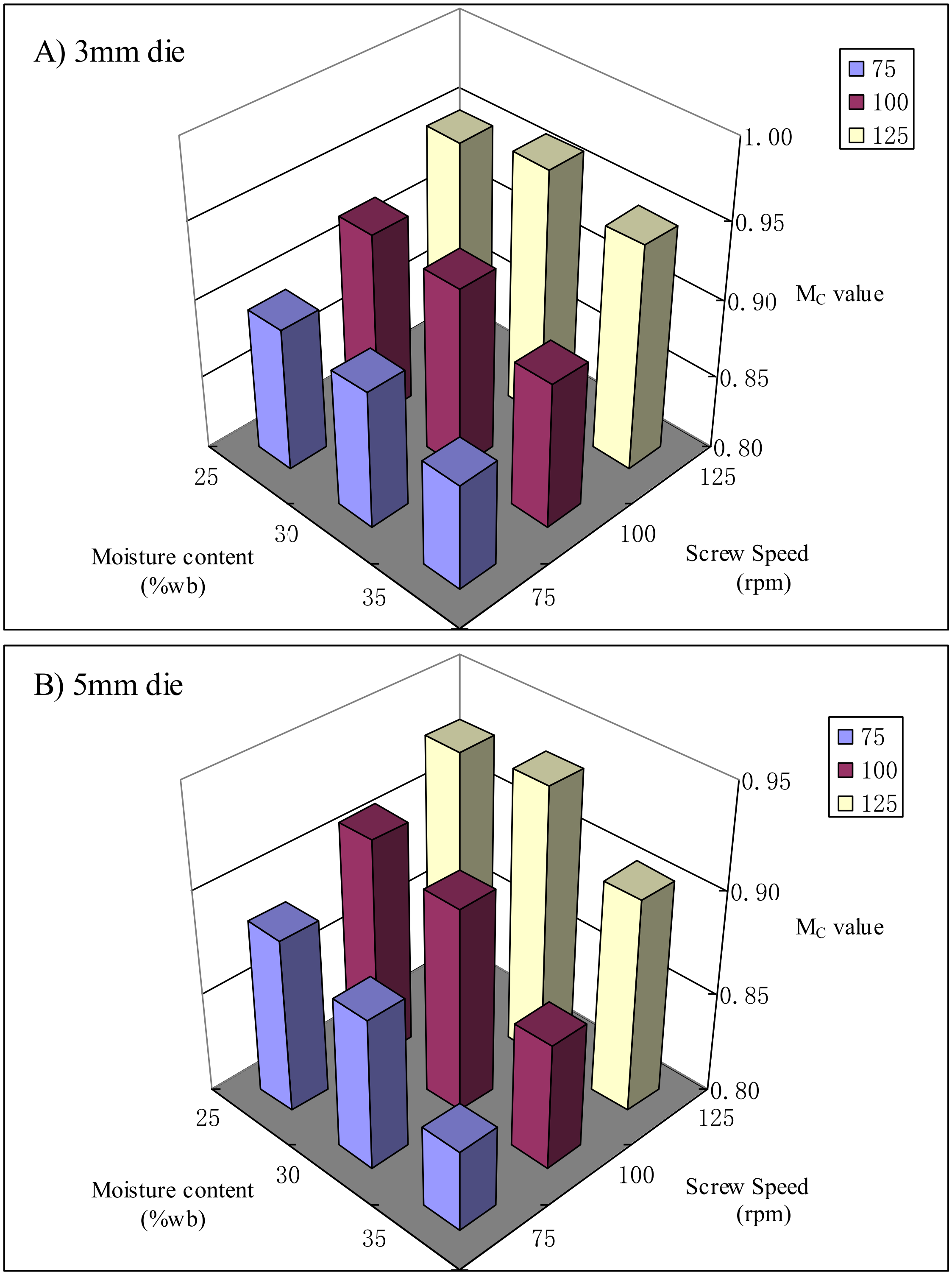


Figure 4.10 Model predicted effects of screw speed and moisture on M_C value at different moisture content and rpm for the a) 3mm diameter or b) 5 mm diameter die.

4.5 Conclusions

RTD parameters associated with SPI and corn flour were influenced by machine screw speed, die diameter and raw material moisture content. Mean residence time decreased with increasing the screw speed, moisture content and die diameter. The distribution of the RTD was wider when the screw speed or the moisture content was lower or the die diameter was smaller. An expression of F curve, which originally was not used in the extrusion process, has been demonstrated to well fit the situation of SPI-corn flour mixture extrusion process.

Three parameters greatly affected the RTD of the SPI-corn flour mixture. They all significantly ($p<0.05$) affected the ECT, t_m and variance. Only the material moisture content significantly affected the FPRT. All the interactions within the three parameters were significant ($p<0.05$) on the variance and ECT. On the other hand, the interaction of screw speed and die diameter was significant to the t_m. All the parameters were significantly ($p<0.05$) affected by the rate of the particle accumulation and half-concentration internal age, while the interactions of screw speed/moisture content, screw speed/die diameter and moisture content/die diameter were significant (($p<0.05$) on affected the magnitude of M_C. Both B and Mc related to the process variables through an RSM model.

Connective statement to Chapter 5

Chapter 4 focused on the resident time distribution (RTD) in the lab extruder, it helped to give clues on how the SPI-corn flour mixture will run within the extruder, and together with the previous exploratory study in Chapter 3, the basic information about our extrusion system was clarified. In order to determine the extrusion performance for high protein content formulations, a final study on the physical properties of the products with high protein content was employed. In the study focused in Chapter 5, the protein content of the SPI corn flour mixture was increased up to 66.7% to fine tune the extrusion coordinates. Within these high limits, optimization of the process based on the physical properties was included to predict the optimal processing conditions.

CHAPTER 5 OPTIMIZATION OF EXTRUSION PROCESS FOR PREPARATION OF PROTEIN DENSE SOY-CORN BASED FORMULATIONS

Abstract

Protein rich extruded products were prepared from different blends of soy protein isolate and corn flour using a twin screw extruder and the extrusion effects on the physical properties of the extruded product were evaluated as related to the different process variables: protein content (32.2-66.6%), feed moisture content (31.6-48.4%) and processing temperature (126.4-193.6°C). A central composite rotatable design (CCRD) and model generated response surfaces plot that served to evaluate the significance of independent and interaction effects of extrusion process variables on the product's various physical properties (breaking stress, bulk density, expansion ratio, water solubility index, rehydration rate and color). Second order polynomial regression equations were developed to relate the product responses to process variables as well as to obtain the response surfaces plots. The independent variables had significant ($p \leq 0.05$) effects on physical properties of extruded products: (i) higher SPI and feed moisture contents increased the breaking stress and bulk density, but decreased the expansion ratio, water solubility index, and rehydration rate, (ii) higher SPI content decreased the color L value, whereas higher feed moisture content increased it, (iii) higher temperatures increased breaking stress, expansion ratio, rehydration rate and L value, but decreased the bulk density and water solubility index.

5.1 Introduction

Since the consumer always demands healthy and nutritious foods, finding nutritional products is a food producer's unchangeable target. Degradation in the nutritional quality of finished food products arising from thermal processing of foods is a challenging matter under most traditional cooking methods. Extrusion cooking is a high temperature short time (HTST) cooking technique which provides thermal and shear energy to a food material to induce desirable physical and chemical changes. HTST extrusion process can minimize heat degradation of food nutrients, while improving digestibility by gelatinizing starch and denaturing protein (Harper, 1981) as compared to traditional thermal processed foods. The extrusion cooking technique is preferable to others in terms of process continuity with high productivity and significant nutrient retention (Guy, 2001; Singh *et al.*, 2007). The functional properties of the food ingredients are modified during the extrusion processing process (Asp and Bjorck, 1989); extrusion also destroys or inactivates the anti-nutritional or toxic compounds (i.e. trypsin inhibitors, hemagglutinins, and gossypol etc.), undesirable enzymes such as lipoxigenases, peroxidases, lipoxidases and lipases, microorganisms and other food-borne pests (Harper, 1981).

Extrusion of corn flour products has been extensively studied (Chinnaswamy and Hanna, 1998; Gomez and Aguilera, 1984); however, when such products are made exclusively from corn ingredient, they often lack macro-nutrients like proteins. There has been a general interest in enriching the nutrient content of extruded foods by adding other nutritional ingredients to the feed mix used in the extrusion process (Guy, 2001; Konstance *et al.,* 1998). Incorporating soy protein isolates (SPI) into corn flour can significantly increase it protein content and quality characteristics of the extruded product (Harper, 1989). With the development of the soybean processing industry, the soy crop has proven to be a low cost, and widely available source of superior quality protein. The use of SPI in extrusion products provides a high quality protein source, rich in lysine and bland in flavor, while reducing the flatulence factors and reducing sugars associated with whole soy flour. Thus SPI, widely used as a functional ingredient in the food industry, is believed to contribute to the overall improvement in extrusion product quality (Konstance *et al.,* 1998).

Showing a high potential to improve the nutritional profile of starch-based extruded food products (Sun and Muthukumarappan, 2002), SPI also has been credited with several potential health benefits. People with high (*vs.* low) soybean intakes have lower rates of coronary heart disease, breast cancer and osteoporosis (Liu, 2004; Sun and

Muthukumarappan, 2002). Indeed, a statement that "including 25 g of soy protein per day in a diet low in saturated fat and cholesterol may reduce the risk of heart disease by lowering blood cholesterol levels" was approved by the US Food and Drug Administration (FDA, 1999). To uphold this soy protein health claim a single serving of the food must contain a minimum of 6.25 g of soy protein (FDA, 1999). Meeting the FDA soy protein threshold necessary to make such health claims can be achieved by producing SPI-enriched food products (i.e. extruded soy-corn blend food products) for all three meals and for snacks.

A number of studies have attempted to meet these goals. Konstance *et al.* (1998) produced extruded products combining corn meal with soy flakes, soy protein concentrate and soy oil. Faller *et al.* (1999) developed acceptable extruded snack products containing soy protein, and evaluated the influence of soy protein type, soy content, and moisture content. Muhungu *et al.* (1999) extruded corn flour and soy protein to investigate the influence of barrel temperature, moisture content, and relative residence time on extruder response and isoflavone profile. Sun and Muthukumarappan (2002) examined the effects of defatted soy flour content, feed moisture, screw speed and temperature on the functionality of soy-based extruded products. Seker (2005) evaluated the expansion ratio, bulk density and water solubility index of extruded products of SPI-modified corn starch mixtures. The effects of moisture content, screw speed and soybean content on the textural qualities of soybean-corn starch extruded products were studied by Li *et al.* (2005).

In our previous study (Chapter 3), the effects of feed moisture, screw speed and barrel temperature on physical properties of extruded corn flour-SPI (20%) blends were studied to understand the influence of extrusion process on the product and to identify working range of parameters. Within the framework of this study, processing conditions (*e.g.*, extrusion temperature, feed moisture and screw speed) were analyzed for constraint based optimization to provide end-products with a wide range of physical quality parameters (e.g., expansion ratio, bulk density, breaking stress, water solubility index, rehydration ratio and color).While the study showed very promising results producing high value products, the protein content was limited to a maximum of about 25%. This was because of the experimental design and somewhat broader objectives.

The previous experimental design was refined to include much higher levels of protein content (32.2-66.6%) so that the product could be used as a protein supplement. In order to limit the experimental variables to 3, only two of the three factors from the previous study (moisture content and temperature) were employed in this study so that the same 20 run CCRD design could be employed. While the study may look similar, the influencing parameters and their ranges selected were quite different (temperature and feed moisture content in the 126.4-193.6°C and 31.6-48.4% range as compared with the earlier 140-180°C, and 18-38%, respectively) making the study and the product appreciably different. The overall objectives were to evaluate the effects of processing parameters (protein content, feed moisture content, and extrusion processing temperature) on the quality of extruded protein rich products and assess their optimization scenarios.

5.2 Materials and Methods

5.2.1 Materials

Corn flour from Brar Natural Flour Mills (Winnipeg, MB) was purchased from a local market. The composition of the flour was: 1.7% lipids, 76.7% carbohydrate, 10% protein, 12% moisture. Soy protein isolate containing (90% protein, 5% carbohydrates and 5% moisture) was obtained from American Health and Nutrition (Ann Arbor, MI), while soy flour (composition: Protein content 40%, lipids 22%, carbohydrates 33.5%, moisture 4.5%) was purchased from Soyador (Quebec, Canada). The purpose of adding soy flour to the mixture was to provide some natural fat for lubrication of the extruder during the process. To a small extent some fat (1.7%) also came through corn flour. The moisture contents of all the flours were measured before mixing.

5.2.2 Extrusion process

A co-rotating twin screw extruder (DS32-II, Jinan Saixin Food Machinery, Shandong, P. R. China) was used in all extrusion processes. The barrel was equipped with four independent temperature controlled zones. The first zone (feeding part) temperature was controlled at 110°C, second and third zones (mixing part) were controlled at 135°C and 150°C, the temperature of the fourth barrel zone (metering section) was adjusted to the required levels as one of the variables. The diameter of the screw was 30 mm. The length to diameter ratio of the extruder barrel was 20:1. The diameter of the hole in the die was 5 mm with a die length of 27 mm. A constant screw speed of 100 rpm was selected based on previous experimental results, and to limit the number of process variables to three. The extruder was fed automatically through a conical hopper, keeping the flights of the screw fully filled and avoiding accumulation of the material in the hopper.

After stable conditions were established, extruded products were collected and cut into 35 mm long cylindrical specimens and dried at 55°C for 120 min by an air convection oven, at an air flow rate of 0.1 m/s, and then further dried to a moisture content of 9-10% (wet basis) by an air convection oven operating at 45°C and an air flow rate of 0.1 m/s. Dried samples were stored in air-tight plastic containers at room temperature until analysis.

5.2.3 Experimental design.

In a previous experiment (Chapter 3), different variables including screw speed, moisture content and barrel temperature were tested, in order to get a protein enriched product by maintaining a 20% SPI level in the feed mixture. In this study, protein content was used as one of the prime variable and two other independent variables (barrel temperature and moisture content) were selected and investigated using a central composite rotatable design (CCRD) (Draper, 1982). Protein content on a dry matter basis was varied from 32.2-66.6%, feed moisture content (wb) from 31.6-48.4%, and extrusion barrel temperature (metering section) from 126.4-193.6°C. Overall, 20 experimental runs were made, each with 8 (2^3) factorial points (three level for each variables), six star corner points (two for each variable) and 6 centre points to meet the statistical design requirements. The CCRD experiment ranges for the 3 independent variables were selected based on preliminary tests (Table 5.1).

An Excel worksheet was used to record the quantities of the SPI, soy flour, corn-flour and moisture (based on a mass balance approach). The details of the different test run with coded (and real) values of the process variables as well as the amount of ingredients added for a 2.0 kg batch (excluding moisture) are shown in Table 5.1. The flours were mixed in a Hobart mixer (Hobart Food Equipment Group Canada, North York, ON) operating at a medium speed. Predetermined amount of water was added to adjust the mixture to the desired moisture content, according to the experimental design. The wetted blends were mixed for 20 min in the Hobart mixer before use.

Table 5.1 Experimental (CCRD) design with coded and (actual) values for protein content (P), moisture content (M), and processing temperature (T), along details of the quantities of soy protein isolate (SPI), soy flour, corn flour and water added for each 2.0 kg batch of ingredients.

Run	P (%)	M(wb%)	T (°C)	SPI (g)	Soy flour (g)	Corn flour (g)	Water (g)
1	-1 (40)	-1 (35)	-1 (140)	633	67	1300	790
2	1 (60)	-1 (35)	-1 (140)	1095	108	797	842
3	-1 (40)	1 (45)	-1 (140)	633	67	1300	1298
4	1 (60)	1 (45)	-1 (140)	1095	108	797	1359
5	-1 (40)	-1 (35)	1 (180)	633	67	1300	790
6	1 (60)	-1 (35)	1 (180)	1095	108	797	842
7	-1 (40)	1 (45)	1 (180)	633	67	1300	1298
8	1 (60)	1 (45)	1 (180)	1095	108	797	1359
9	-1.68(33.2)	0 (40)	0 (160)	478	53	1469	1004
10	1.68 (66.6)	0 (40)	0 (160)	1252	122	626	1098
11	0 (50)	-1.68(31.6)	0 (160)	862	87	1051	676
12	0 (50)	1.68 (48.4)	0 (160)	862	87	1051	1547
13	0 (50)	0 (40)	-1.68(126.4)	862	87	1051	1051
14	0 (50)	0 (40)	1.68 (193.6)	862	87	1051	1051
15	0 (50)	0 (40)	0 (160)	862	87	1051	1051
16	0 (50)	0 (40)	0 (160)	862	87	1051	1051
17	0 (50)	0 (40)	0 (160)	862	87	1051	1051
18	0 (50)	0 (40)	0 (160)	862	87	1051	1051
19	0 (50)	0 (40)	0 (160)	862	87	1051	1051
20	0 (50)	0 (40)	0 (160)	862	87	1051	1051

In order to develop the RSM models, coded values (P_{cv}, T_{cv}, M_{cv}, respectively) were derived from the numerical values of the independent variables protein content (P, as %), barrel temperature (T, as ^{o}C), and feed moisture (M, as %):

$$P_{cv} = \frac{(P - 50) \times 1.682}{10} \tag{5.1}$$

$$T_{cv} = \frac{(T - 160) \times 1.682}{20} \tag{5.2}$$

$$M_{cv} = \frac{(M - 35) \times 1.682}{10} \tag{5.3}$$

5.2.4 Physical properties

Expansion ratio (ER), Bulk density (BD), Breaking stress (BS), Water solubility index (WSI), Rehydration ratio (RR), Color are selected as the physical parameters tested in this experiment.

Breaking stress

Breaking stress (BS) was measured in a 3-point bend test (Zasypkin and Lee, 1998) using the TA.XT Plus Texture Analyzer (Texture Technologies Corp., Scarsdale, NY/Stable Micro Systems, Godalming, Surrey, UK.) equipped with a 50 N load cell. The extruded product was placed on two rounded stands (bridge) 30 mm apart. A rounded plunger was made to push the sample at the middle of the bridges at 5 mm/min until breakage occurred. BS was determined as the breaking force per unit cross section area (N/mm^2). Eight measurements were made on each product (separate samples) and their mean value was used.

Bulk density

Bulk density (ρ, g/ml) was measured using the displacement method (Seker, 2005). Extruded products strands were cut into roughly 25 mm (1 inch) sections (≈ 15 g) and weighed (M_{ext}, g). Each strand was then placed in a graduated cylinder, to which a certain volume (V_{om}, ml) of yellow millet particles were added, and the final volume was measured (V_{ym}, ml). BD was calculated as:

$$\rho = \frac{M_{ext}}{V_{ym} - V_{om}} \quad \text{(g/ml)} \tag{5.4}$$

Expansion ratio

Expansion ratio (ER) **is** defined as the ratio of the diameter of the extrudate to the diameter of the die (Jyothi *et al.*, 2009). In order to determine the ER, 20 randomly selected segments of each sample were measured using vernier caliper and the mean value was used.

Water solubility index

The water solubility index (WSI) of the extrudate was determined following the method described by Anderson *et al.* (1969) with little modification. The extruded products were ground and about 2 g (W_{os}) of the ground extrudate was placed into a centrifuge tube. Distilled water (25 ml) at 30°C was added with the sample. After 10 min standing with intermittent shaking every 2 min, the sample was centrifuged at 5000 rpm for 15 min (Sorvall GLC-2B General Laboratory Centrifuge, Du Point Instrument). The supernatant was decanted into a Petri dish and dried at 105°C overnight. The weight of the dry solid (W_{ds}) was determined and the WSI (%) calculated:

$$WSI = \frac{W_{ds}}{W_{os}} \times 100 \tag{5.5}$$

Rehydration ratio

An air dried sample (≈20 g, 9-10% moisture wb) of extrudate were weighed (M_1) and placed into 500 ml of water at 30°C for 15 min. The water was then drained and the rehydrated sample was weighed (M_2). The rehydration ratio (*RR,* %) was defined as:

$$RR = \frac{M_2 - M_1}{M_1} \times 100 \tag{5.6}$$

Color

Color measurements were done using a Minolta colorimeter CM-500d using an aperture of 1.2 cm diameter. The instrument was calibrated with a standard white tile (L = 77.58, a = -0.27, b = -26.63). The color was reported in terms of Hunter L, a and b values. Eight measurements on each sample were taken and the mean value was used.

Regression modelling and statistical analysis

The second order polynomial equation fitted with coded variables was:

$$Y = B_o + \sum_{i=1}^{n} B_i X_i + \sum_{i=1}^{n} B_{ii} X_i^2 + \sum_{i=1}^{n-1} \sum_{j=i+1}^{n} B_{ij} X_i X_j \qquad (5.7)$$

where, Y represents the experimental responses (BS, BD, ER, WSI, RR and color), B_o, B_i, B_{ii} and B_{ij} are constants and regression coefficients of the model, while X_i and X_j are independent extrusion processing variables, n is number of processing variables (here n = 3) The whole model includes linear, quadratic and cross-product terms.

Experimental data were analyzed using Design-Expert 6.0 (Stat-Ease Inc. Minneapolis, MN) and Microsoft Excel 2003 Version. Design-Expert was used to solve the second order polynomial regression equation and Excel was used to investigate the effects of three independent input variables of SPI content, feed moisture content and extrusion processing temperature on the physical properties (BS, BD, ER, WSI, RR and color) of extruded products. Analysis of variance (ANOVA) was employed to justify the model goodness of fit as well as significant effects of independent variables on responses.

5.3 Results and Discussion

The experiment arrangements (test run number) and data on the physical properties of breaking stress (BS), bulk density (BD), Expansion ratio (ER), water solubility index (WSI), rehydration rate (RR) and color (L, a, b values) of extruded products are shown in Table 5.2. The table also indicates the approximate proximate composition (on dry weight basis) of the product obtained after the extrusion process. Second-order polynomial regression equations were established by considering significant factors on the basis of t (t-test)> 2.5 at probability level p< 0.05 and coefficients obtained are presented in Table 5.3. The established equations show empirical relationship between physical properties (BS, BD, ER, WSI, RR and Color) and the coded value of independent variables of total protein content (P), feed moisture content (M) and barrel temperature (T). An analysis of variance (ANOVA) was performed to analyze the impacts of independent variables of soy protein isolate content, feed moisture content and temperature on the physical properties of BS, BD, ER, WSI, RR and the color parameter L of extruded product

The ANOVA study results showed that total protein content significantly affected the WSI, ER and L. Feed moisture content (M) significantly ($p<0.05$) affected the RR, BD and BS. Barrel temperature (T) significantly ($p<0.05$) affected WSI, RR, BD, BS and ER. All the three parameters showed the quadratic effect ($p<0.05$) to WSI, BS, BD and ER. Interactive effects of total protein content and feed moisture were found on RR, BS, BD and L ($p<0.05$). Interactive effects ($p<0.05$) of total protein content and barrel temperature were found on RR, BS, BD and ER. Interactive effects ($p<0.05$) of moisture content and barrel temperature were found on WSI, RR, BS, BD and ER.

Table 5.2 Experimental results for each test run (details in Table 5.1) as means (and standard deviation) of physical properties of soy protein isolate- corn starch blend extrusions and approximate proximate composition (in dry matter) and the moisture content of the extudate of the resulting product

Run	BS (N/mm2)	BD (g/ml)	ER	WSI (%)	RR (%)	L	a	b	Protein (%db)	Carbohydrate (%db)	Moisture (wb%)
1	0.641(0.017)	0.771(0.037)	1.31(0.048)	3.6(0.127)	58(2.1)	87.01(3.035)	0.12(0.004)	23.85(0.842)	40.1	57.9	30.7
2	0.246(0.033)	0.864(0.039)	1.43(0.078)	4.7(0.238)	195(9.5)	75.17(4.053)	1.54(0.083)	28.77(1.549)	60.0	38.0	29.0
3	0.179(0.012)	0.528(0.027)	1.35(0.053)	5.5(0.321)	49(3.8)	83.75(3.168)	1.79(0.068)	31.52(1.135)	40.1	57.9	32.0
4	0.141(0.027)	0.723(0.098)	1.45(0.204)	6.8(0.763)	95(10.3)	78.47(9.016)	3.39(0.667)	32.68(2.457)	60.0	38.0	34.4
5	0.523(0.032)	0.613(0.036)	1.37(0.076)	4.9(0.291)	157(8.5)	84.89(5.033)	0.18(0.011)	25.11(1.495)	40.1	57.9	23.6
6	0.458(0.059)	0.423(0.075)	1.69(0.219)	5.1(0.641)	141(12.8)	74.21(8.789)	3.47(0.218)	35.36(3.276)	60.0	38.0	24.0
7	0.332(0.009)	0.656(0.019)	1.28(0.021)	3.1(0.046)	205(4.4)	82.87(1.188)	1.57(0.023)	32.15(0.463)	40.1	57.9	26.7
8	0.773(0.051)	0.514(0.028)	1.51(0.094)	3.8(0.195)	96(7.1)	83.03(4.470)	1.39(0.077)	24.79(1.018)	60.0	38.0	27.5
9	0.431(0.015)	0.662(0.016)	1.41(0.046)	2.9(0.101)	132(7.2)	87.37(3.041)	1.05(0.037)	34.72(1.209)	33.2	64.8	30.5
10	0.282(0.021)	0.641(0.055)	1.59(0.107)	4.6(0.308)	122(5.5)	75.69(5.064)	2.97(0.219)	29.29(1.948)	66.6	31.4	29.2
11	0.586(0.028)	0.669(0.034)	1.45(0.054)	3.8(0.138)	131(5.6)	84.24(3.259)	0.52(0.019)	25.17(0.912)	50.0	48.0	24.2
12	0.216(0.007)	0.541(0.018)	1.49(0.033)	4.5(0.108)	96(2.7)	84.38(2.248)	0.55(0.013)	23.09(0.555)	50.0	48.0	33.7
13	0.237(0.010)	0.791(0.035)	1.25(0.066)	5.6(0.202)	60(2.7)	84.38(3.197)	0.79(0.035)	23.73(1.028)	50.0	48.0	31.8
14	0.726(0.020)	0.452(0.023)	1.51(0.041)	3.9(0.109)	195(5.2)	80.13(2.247)	1.52(0.043)	29.07(0.815)	50.0	48.0	23.8
15	0.684(0.031)	0.475(0.021)	1.74(0.067)	3.4(0.182)	107(5.5)	81.81(4.338)	1.43(0.077)	25.34(1.985)	50.0	48.0	26.8
16	0.816(0.038)	0.478(0.019)	1.73(0.061)	2.9(0.127)	129(5.9)	80.13(3.233)	1.86(0.075)	28.97(1.175)	50.0	48.0	30.3
17	0.671(0.062)	0.466(0.058)	1.77(0.098)	3.4(0.221)	139(10.3)	80.03(5.194)	0.34(0.022)	24.15(1.540)	50.0	48.0	28.5
18	0.721(0.053)	0.495(0.036)	1.75(0.105)	2.7(0.163)	139(10.7)	80.19(4.821)	0.55(0.043)	24.79(1.487)	50.0	48.0	27.9
19	0.828(0.041)	0.476(0.028)	1.69(0.093)	3.1(0.171)	139(7.4)	79.68(4.371)	0.68(0.057)	25.85(1.423)	50.0	48.0	28.2
20	0.818(0.034)	0.479(0.101)	1.76(0.114)	3.3(0.214)	129(8.4)	81.21(5.197)	1.05(0.046)	25.65(1.661)	50.0	48.0	28.0

BS = Breaking stress, BD= Bulk density, ER= Expansion ratio, WSI= water solubility index, RR=Rehydration ratio, L, a, b (color)

Table 5.3 Regression equations for physical properties of soy protein isolate-corn starch blend extrusion (taking significant parameters on the basis of t > 2.5 at probability level $p \leq 0.05$). BS = Breaking stress, BD= Bulk density, ER= Expansion ratio, WSI= water solubility index, RR = Rehydration ratio, L= Lightness (L, a, b color space)

Physical properties	Equations (in coded values)
BS (N/mm^2)	Y_{BS}=0.756-0.078P+0.125T-0.137P^2-0.122M^2-0.093T^2+0.108P*M+0.101P*T+0.086M*T
BD (g/ml)	Y_{BD}=0.478-0.034M-0.092T+0.062P^2+0.045M^2+0.051T^2+0.019P*M-0.078P*T+0.065M*T
ER	Y_{ER} = 1.74+0.079P+0.055T-0.087P^2-0.097M^2-0.129T^2+0.041P*T-0.041M*T
WSI (%)	Y_{WSI} = 0.031+0.005P-0.05T +0.003P^2+0.004M^2+0.007T^2 -0.009M*T
RR (%)	Y_{RR} = 125.7-12.1M + 31.4T-23P*T-38.5P*T+14.1M*T
L value (color)	Y_L = 81.332 -3.462P + 2.175P*M

P: Protein content; M: Moisture content ; T: Barrel temperature

Various physical properties have been studied in different extruded products. Jyothi *et al.* (2009) studied the physical properties including bulk density, true density, porosity, and expansion ratio; water absorption index, water solubility index, oil absorption index, in the single extruder to process tuber starch. Rocha-Guzman *et al.* (2008) studied water absorption index (WAI), water absorption capacity (WAC), oil absorption capacity (OAC), and emulsifying capacity (EC) in the extrusion process of bean cultivars flour. Őzer *et al.* (2004) studied the physical properties (bulk density, expansion, and porosity) of a nutritionally balanced extruded snack food by the RSM method. The influence of process variables on physical properties have been shown to be generally significant in all these studies.

Table 5.2 also provides some data on the resulting product in the form of proximate composition and the actual moisture content of the products. These are predicted values based on the dry ingredients and the moisture content of the product as it exited from the extruder. It can be seen that the extruded product has a dry basis protein content in the range 40 to 60%, carbohydrate in 31 to 65% range and moisture content in the 22-35% range. While they provide a protein and carbohydrate rich product, the extruded product is also too high in moisture content to provide adequate stability. The water activity in most cases was higher than 0.85 and hence the drying of extruded product for few hours was necessary to produce a low moisture shelf stable product.

5.3.1 Breaking stress (BS)

The values of breaking stress (BS) of extruded products under experimental conditions are presented in Table 5.2. The highest value of BS was 0.828 N/mm^2 while extrusion was done at 160°C with 50% protein content and 40% feed moisture. The lowest value of BS was 0.141 N/mm^2 while extrusion processing at 140°C with 40% protein content and 45% feed moisture. According to the BS, barrel temperature was the most significant affecting parameter (Table 5.4), response surfaces plot for protein content *vs.* moisture for temperatures of 140°C, 160°C and 180°C are shown in Figure 5.1 in subplots A, B and C, respectively. The results show that with an increase in temperature, the BS values increased, especially for the high protein and high moisture content product. The lowest BS appeared when the barrel temperature was the lowest (Figure 5.1-A), the moisture and protein content are at the highest level; the highest BS appeared in Figure 5.1-C, when the barrel temperature is the highest, the moisture and protein content are also at the highest level.

Barrel temperature and material moisture content significantly ($p \leq 0.05$) affected the BS (Table 5.4), and all the quadratic effects including protein content, moisture content and barrel temperature, and interaction effects including protein content and moisture content, protein content and temperature content, moisture content and temperature significantly affected BS. Lack of fit was not significant relative to the pure error, which meant the model was well fitted. The regression equation for the empirical relationship between BS (Y_{BS}) and the independent extrusion processing variables in coded form is shown in Table 5.3.

Sun and Muthukumarappan (2002) revealed that the shear force per unit weight of extrudate decreased with an increase in the feed moisture and a decrease in barrel temperature. It may be expected that high moisture content in the blends with high temperature extrusion processing expands the products due to release of superheated steam. This phenomenon helps to make hollowed and low density products that decrease the breaking stress of the extruded products. But our study revealed the reverse findings regarding to high moisture, high protein content and high barrel temperature of the process. One reason can be the high density product naturally offers high breaking stress. A high extrusion processing temperature with a high screw speed provides a high level of thermal and mechanical energy simultaneously, which possibly leads to excessive structural damage and breakdown, and hence density increases slightly (Guha *et al.,* 1997). Another reason can be the air cell membrane of the extruded products became harder due to high soy protein isolate content. Harper (1981) showed that shear strength of the extruded products increased with an increase in the protein content and processing temperature. The ANOVA study demonstrated that, between the three parameters, barrel temperature was the one that affected the BS appreciably, and with a combination effect of the three parameters, the result was even more intense.

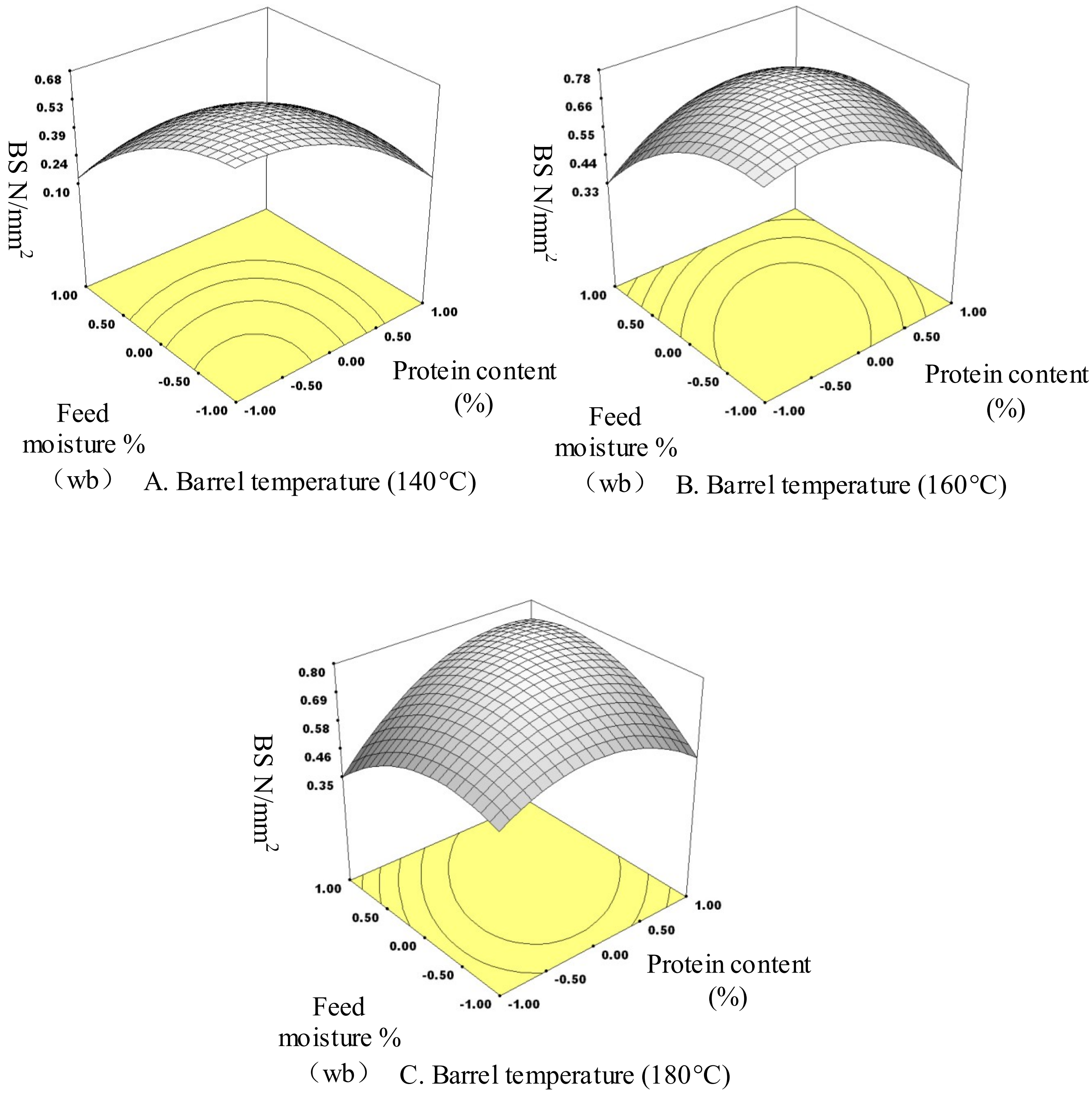


Figure 5.1 Response surfaces plot of breaking stress (BS) of soy protein isolate-corn starch blend extrusion for the effect of soy protein content and moisture content under different Barrel temperature

Table 5.4 ANOVA of physical properties（BS, ER, L）of soy protein isolate- corn starch blend extrusion

	BS			ER			L		
Source	Coefficient Estimate	Sum of Squares	p-Value	Coefficient Estimate	Sum of Squares	p-Value	Coefficient Estimate	Sum of Squares	p-Value
Model	0.756	1.049	< 0.0001	1.740	0.564	< 0.0001	81.332	223.827	< 0.0001
P	-0.023	0.007	0.2614	0.079	0.084	< 0.0001	-3.462	163.706	< 0.0001
M	-0.078	0.083	0.0021	-0.010	0.001	0.4152	0.764	7.979	0.0653
T	0.125	0.212	< 0.0001	0.055	0.041	0.0012	-0.479	3.139	0.2290
P^2	-0.137	0.272	< 0.0001	-0.087	0.108	< 0.0001			
M^2	-0.122	0.213	< 0.0001	-0.097	0.136	< 0.0001			
T^2	-0.093	0.125	0.0005	-0.129	0.240	< 0.0001			
P*M	0.108	0.093	0.0014	-0.014	0.002	0.4121	2.175	37.845	0.0007
P*T	0.101	0.082	0.0022	0.041	0.014	0.0280	0.825	5.445	0.1203
M*T	0.086	0.060	0.0058	-0.041	0.014	0.0280	0.845	5.712	0.1124
Lack of Fit		0.023	0.5629		0.017	0.0718		22.261	0.0666
Pure Error		0.026			0.004			3.346	
R-Squared		0.955			0.965			0.897	
Adj R-Squared		0.915			0.933			0.850	
Pred R-Squared		0.805			0.772			0.712	
Adeq Precision		13.280			14.229			16.769	

P : Protein content; M: Moisture content ; T: Barrel temperature

5.3.2 Bulk density (BD)

The experimental values of bulk density of extruded products under different designed extrusion conditions are presented in Table 5.2. BD ranged from 0.423 to 0.864 g/ml for the extruded products. The response surfaces plot [Figure 5.2 (A: 140°C, B: 160°C, C: 180°C)] represent the feed moisture and protein content effect on BD under different temperatures. Results show that at the low barrel temperature (140°C), increasing protein content and decreasing moisture content resulted in a higher BD. At high barrel temperature (180°C), increasing protein content lead to decrease the BD. At middle barrel temperature (160°C), the minimum BD area appeared in the middle part of the figure.

Soy protein isolate (SPI) constructs small uniform pores in the extruded products after being squeezed out of the die as soy protein isolate can work as high quality emulsifier between hydrophilic materials and hydrophobic materials by exposing the hydrophilic groups and hydrophobic groups to their respective phases (Li *et al.*, 2005). The thickness of the wall of the pores become thinner when the amounts of soy protein isolate increase and the soy protein isolate absorbs high amounts of water. Thus, it is logical to expect an increasing bulk density of the extruded products with increasing protein and feed moisture contents. Starch gelatinization during extrusion processing has a big influence on bulk density of extruded products. The low processing temperature decreases the extent of gelatinization of the extruded products, which leads to low swelling and low volume of extruded products, therefore, bulk density of extruded products increases with the deceasing of extrusion processing temperature.

The regression equation for bulk density (Y_{DB}) is shown in Table 5.3. ANOVA of bulk density is shown in Table 5.5. ANOVA shows that both barrel temperature and moisture content are significantly affect BD in linear and quadratic forms. Protein content significantly affected BD through a quadratic model. All the interaction effect among protein content, barrel temperature and moisture content were significantly for BD. This explains that temperature provides a curvilinear effect on extrudate bulk density and its quadratic effect dominates at high temperature (Guha *et al.,* 1997).

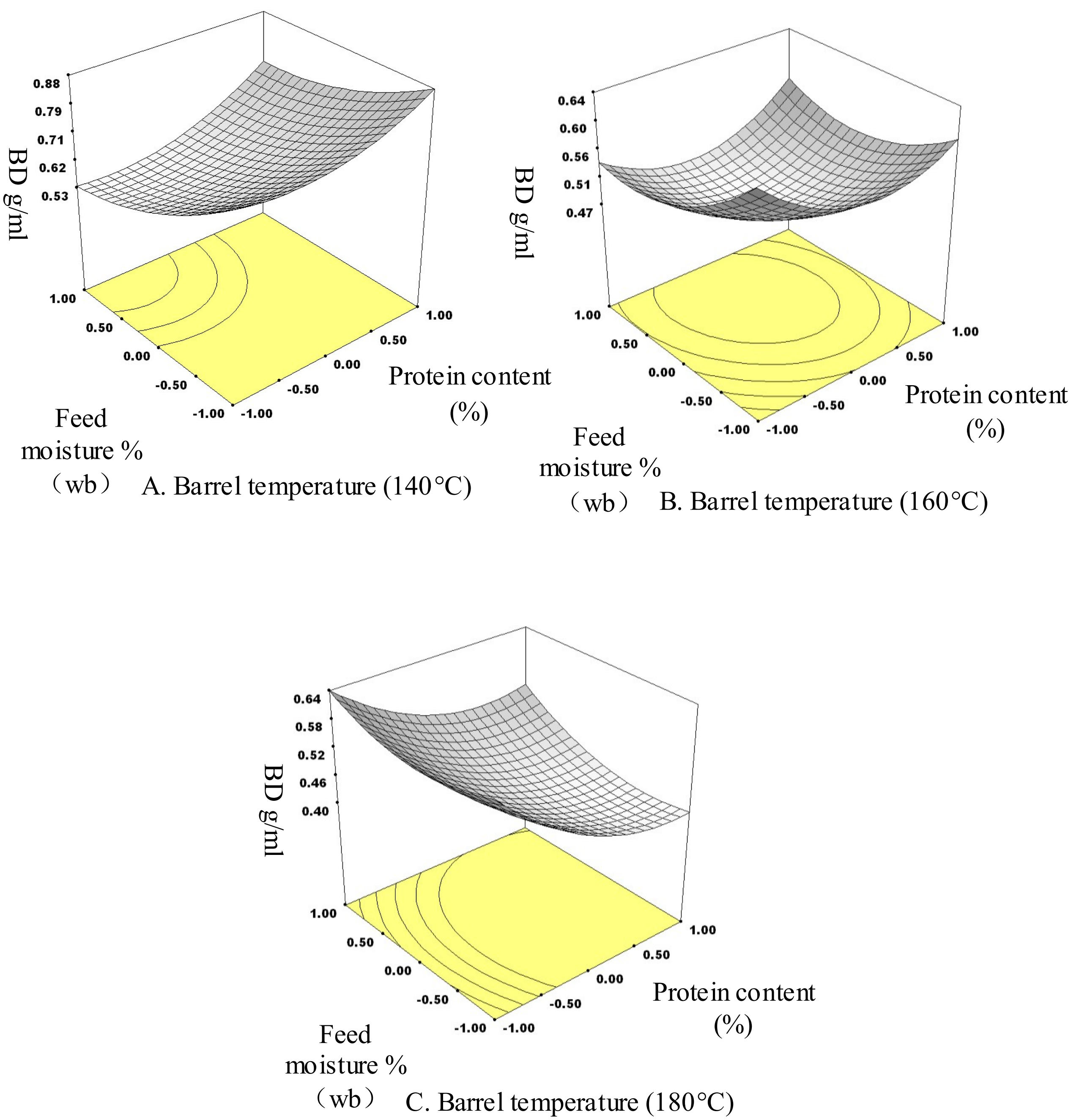


Figure 5.2 Response surfaces plot of bulk density (BD) of soy protein isolate-corn starch blend extrusion for the effect of soy protein content and moisture content

Table 5.5 ANOVA of physical properties（WSI, RR, BD）of soy protein isolate- corn starch blend extrusion

	WSI			RR			BD		
Source	Coefficient Estimate	Sum of Squares	p-Value	Coefficient Estimate	Sum of Squares	p-Value	Coefficient Estimate	Sum of Squares	p-Value
Model	0.031	2.2E-03	< 0.0001	125.7	33210.7	< 0.0001	0.478	0.317	< 0.0001
P	0.005	2.8E-04	0.0004	3.0	120.4	0.4258	-0.006	0.000	0.1407
M	0.002	3.2E-05	0.1079	-12.1	1999.0	0.0052	-0.034	0.016	< 0.0001
T	-0.005	3.2E-04	0.0002	31.4	13440.8	< 0.0001	-0.092	0.114	< 0.0001
P^2	0.003	1.3E-04	0.0047				0.062	0.055	< 0.0001
M^2	0.004	2.9E-04	0.0003				0.045	0.029	< 0.0001
T^2	0.007	6.2E-04	< 0.0001				0.051	0.037	< 0.0001
P*M	0.001	6.1E-06	0.4550	-23.0	4218.2	0.0003	0.019	0.003	0.0027
P*T	-0.002	2.8E-05	0.1268	-38.5	11850.3	< 0.0001	-0.078	0.048	< 0.0001
M*T	-0.009	6.3E-04	< 0.0001	14.1	1582.0	0.0106	0.065	0.034	< 0.0001
Lack of Fit		6.0E-05	0.3460		1539.7	0.4239		0.001	0.1247
Pure Error		4.1E-05			775.5			0.000	
R-Squared		0.956			0.935			0.994	
Adj R-Squared		0.916			0.905			0.989	
Pred R-Squared		0.775			0.819			0.964	
Adeq Precision		16.934			21.259			49.290	

P : Protein content; M: Moisture content ; T: Barrel temperature

5.3.3 Expansion ratio (ER)

Expansion ratio (ER) indicates the extent of puffing of extruded products. The experimental values of expansion ratio of extruded products under different designed extrusion conditions are shown in Table 5.2. The values of expansion ratio (ER) varied from 1.25 to 1.77 on the basis of combinations of extrusion process variables. The highest expansion ratio was 1.77 for extrusion processing at 160°C with 50 % protein content and 40% feed moisture content; on the other hand, the lowest expansion ratio was 1.25 for extrusion was processing at 126.4°C with the same protein and feed moisture contents.

The response surfaces plot presented [Figure 5.3 (A:140°C, B:160°C, C:180°C)] showed that the expansion ratio of extruded products increased with an increasing of feed moisture content and after reaching a maximum the ER decreased with further increasing in moisture content. Similar trends were also observed with protein content and barrel temperature.

The regression equation for the relationship between expansion ratio (Y_{ER}) and independent variables in terms of coded variables is showed in Table 5.3 and the analysis of variance (ANOVA) of the models is presented in Table 5.4. The analysis of variance showed that all the quadratic effects from protein content, feed moisture content and barrel temperature were significant with ER. The interaction effect between protein content and barrel temperature, moisture content and barrel temperature were significant with ER. Protein content was the most significant factor affecting ER. Moisture content was less significant than temperature. This result is some different with our prior study (Chapter 3) in which feed moisture content showed more significant effect to ER than barrel temperature. The difference could be because the protein content was lower and maintained constant at 20%. In this study, protein content is much higher, and the combination of protein and moisture content became highly related with the process temperature and the protein content, so the barrel temperature became more significant.

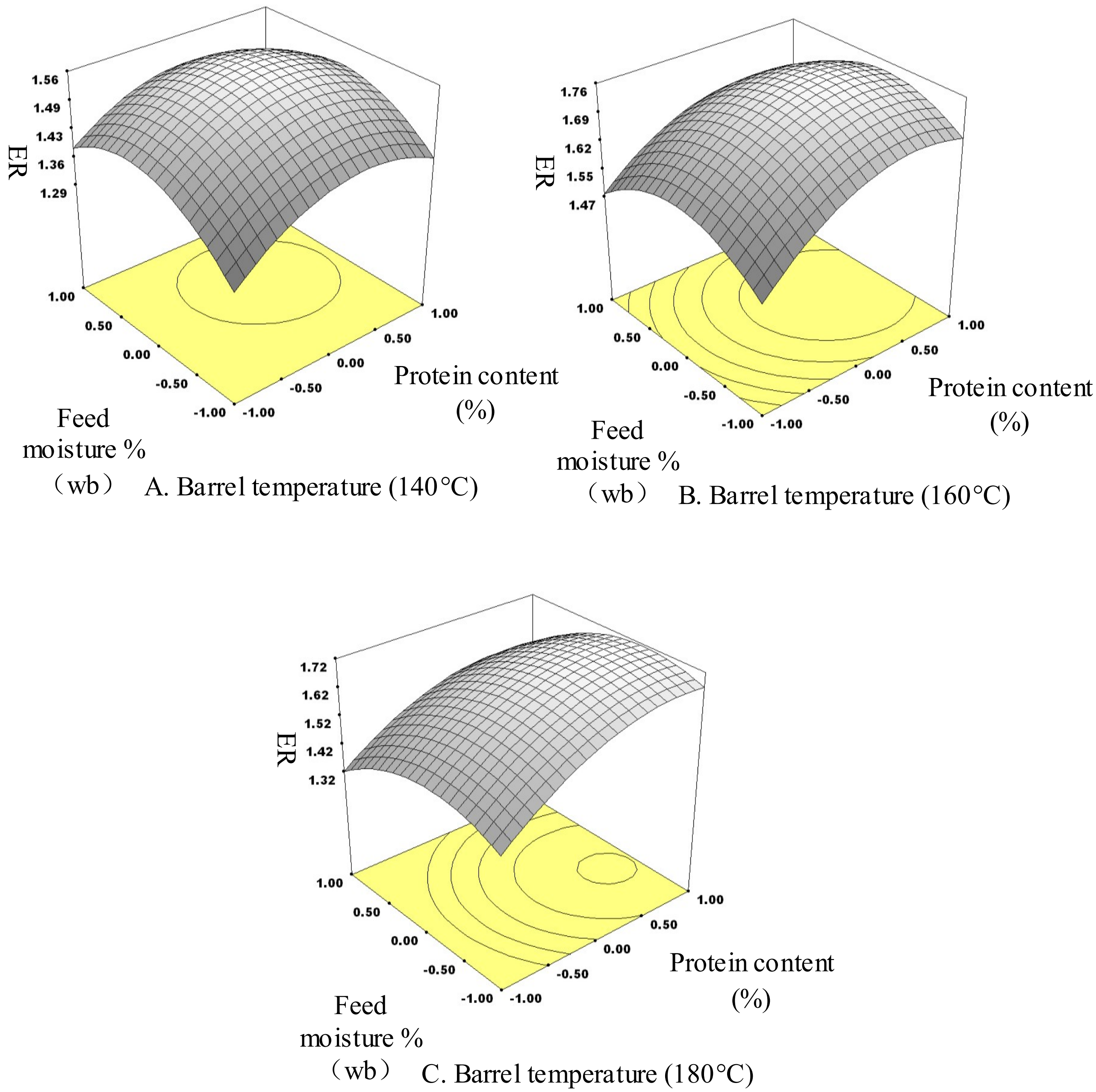


Figure 5.3 Response surfaces plot of expansion ratio (ER) of soy protein isolate-corn starch blend extrusion for the effect of soy protein content and moisture content

5.3.4 Water solubility index (WSI)

The results of water solubility index (WSI) for different experimental conditions are showed in Table 5.2. The water solubility index (WSI) of extruded products ranged between 2.7% (protein content 50%, moisture content 40% (wb), barrel temperature160°C) and 6.8% (protein content 60%, moisture content 45% (wb), barrel temperature140°C).

The response surfaces plot of water solubility index are shown in Figure 5.4 [A: 140°C, B: 160°C, C: 180°C]. Under lower barrel temperature (140°C), the WSI continued to decrease as the feed moisture and protein content decreased. With higher barrel temperature (180°C) the WSI increased with a decrease in the feed moisture and with an increase in the protein content. When the barrel temperature is in the middle (160°C), WSI showed the lowest value in the middle part.

ANOVA of the regression model for water solubility index of extruded products is given in Table 5.5. The model was significant ($p<0.05$). Again barrel temperature and protein content significantly affected the WSI through a linear model. All three factors were significant for WSI with quadratic model, and the interaction between barrel temperature and moisture content was also significant ($p<0.05$). The regression equation, showing empirical relationship between water solubility index (Y_{WSI}) and the processing variables are shown in Table 5.2.

In our previous study (Chapter 3), with 20% SPI content in the extrudate, moisture content was the most significant factor that affected the WSI, and a higher moisture content resulted in a higher WSI. The reason for this might be at lower feed moisture levels, it is possible that there was not enough water for the starch gelatinization and protein denaturation to be completed. This could be the reason for gradual increase in WSI with an increase in moisture content. In this study, when the barrel temperature is at the lower level (140°C), the trend between moisture content and WSI is the same as the previous study, but when the barrel temperature was increased to a higher level (180°C), the WSI decreased with the increasing of the moisture content. Hagenimana *et al.* (2006) reported that with extruded rice flour when feed moisture increased from 16% to 22%, WSI decreased. Gomez and Aguilera (1984) concluded that low feed moisture content of extrudate reduced starch gelatinization and shear degradation of starch, which reduced the physical breakdown of the granules. This may be a possible reason why water solubility index of extruded products increased with the decreasing of feed moisture content. Cumming *et al.* (1973) concluded that the high temperature in the extrusion processing caused most of the water soluble protein to break into small subunits, or become insoluble, and /or be redistributed. This phenomenon may cause to decrease the water solubility index with increasing temperature and soy protein isolate. Different results indicate that the key factor that affects the WSI is how much of the water soluble protein turns into water insoluble. In this study it has been shown that, at the lower temperature condition, increasing moisture content will increase the starch gelatinization and the protein denaturation, but when the temperature is higher to 180°C, the water soluble protein begin to break into insoluble subunits hence the adding moisture content resulted the decrease of the WSI.

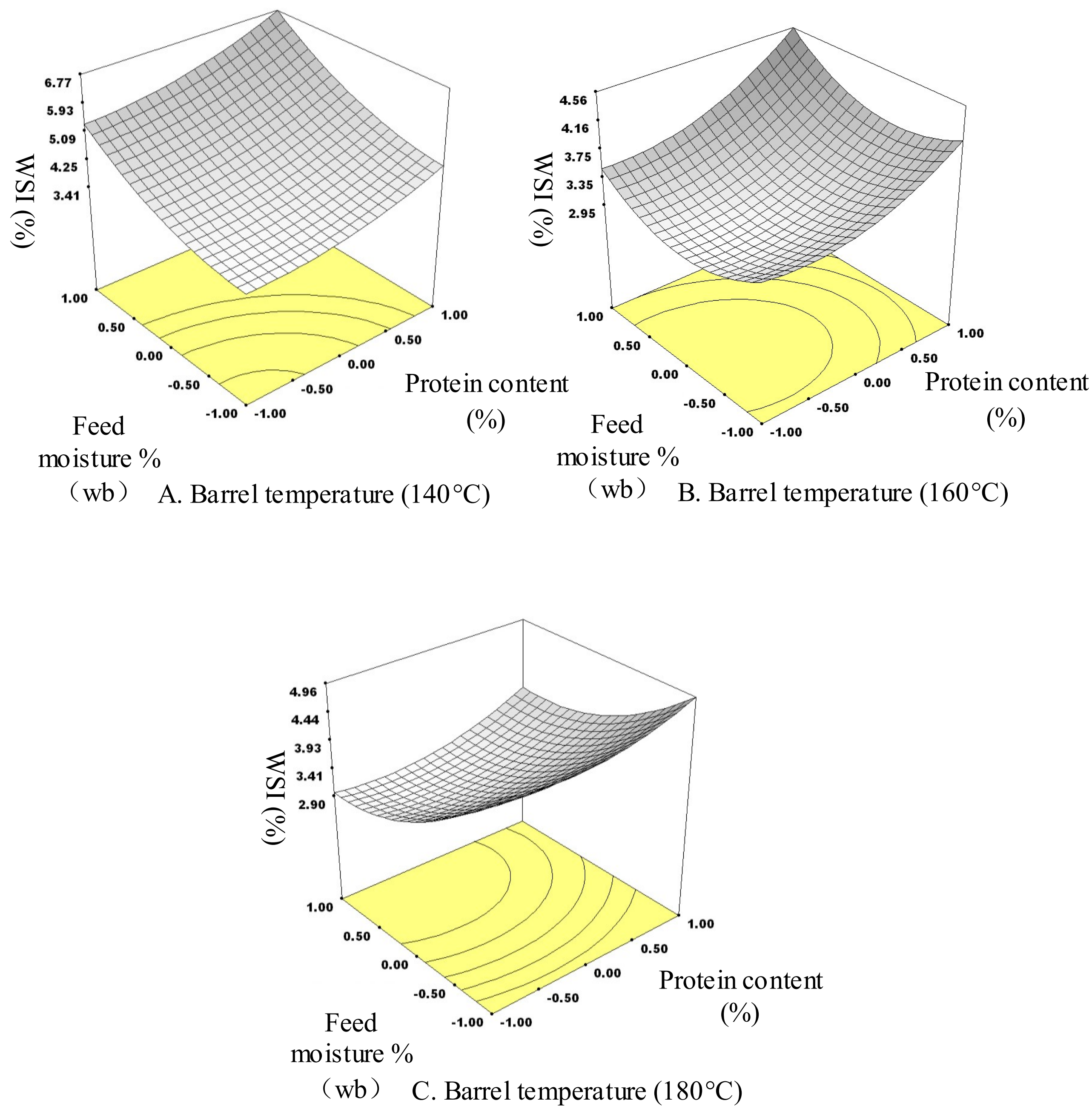


Figure 5.4 Response surfaces plot of water solubility index (WSI) of soy protein isolate-corn starch blend extrusion for the effect of soy protein isolate and moisture content

5.3.5 Rehydration rate (RR)

Rehydration rates of products extruded at different experimental conditions are listed in Table 5.2. The values of RR varied from 49% to 205%. The regression equation for the relationship between rehydration rate (Y_{RR}) and independent variables is shown in Table 5.3. Analysis of variance (ANOVA) of the regression model for rehydration rate of extrudate is showed in Table 5.5. The model gave good prediction correlation between experimental and prediction value of RR ($p< 0.05$). Barrel temperature and material moisture content were significant for RR, temperature was the most significant factor and all interaction among protein content, material moisture content and barrel temperature were also significant.

The response surfaces plot of rehydration rates of extruded products are presented in Figure 5.5 (A:140°C, B:160°C, C:180°C). These figures showed RR have more complicated trend under different extrusion conditions. Under lower barrel temperature (140°C), higher protein content resulted in higher RR. When the temperature was increased to 160°C, an increase protein content at high moisture content (45%) slightly decreased the RR, but increase in protein content at up to a high low moisture content (35%) increased the RR. With barrel temperature at 180°C, RR decreased with an increase in protein content.

Harper (1981) reported that low moisture content in feed decreased the trypsin inhibitor and increased rehydration rate of extruded products. Increasing feed moisture content may lead to retain high moisture content inside the extruded products and consequently decrease the rehydration rate. The high processing temperature probably creates more open spaces and air cells in the product structure due to high temperature generates high thermal energy, which increases the level of superheated steam during extrusion processing. This may impart to imbibe more water when rehydrating the extruded products, subsequently, rehydration rate of extrudate increases with the increasing of extrusion processing temperature. Protein content in feed decreases the starch molecular degradation, an increases in protein content with relative decrease in starch content may influence the extent of starch gelatinization during extrusion processing leading to a decrease in relative water absorption (Yagci and Gogus, 2008). This phenomenon may cause the decrease of rehydration rate of extrudate with the increase in soy protein isolate content in feed blends.

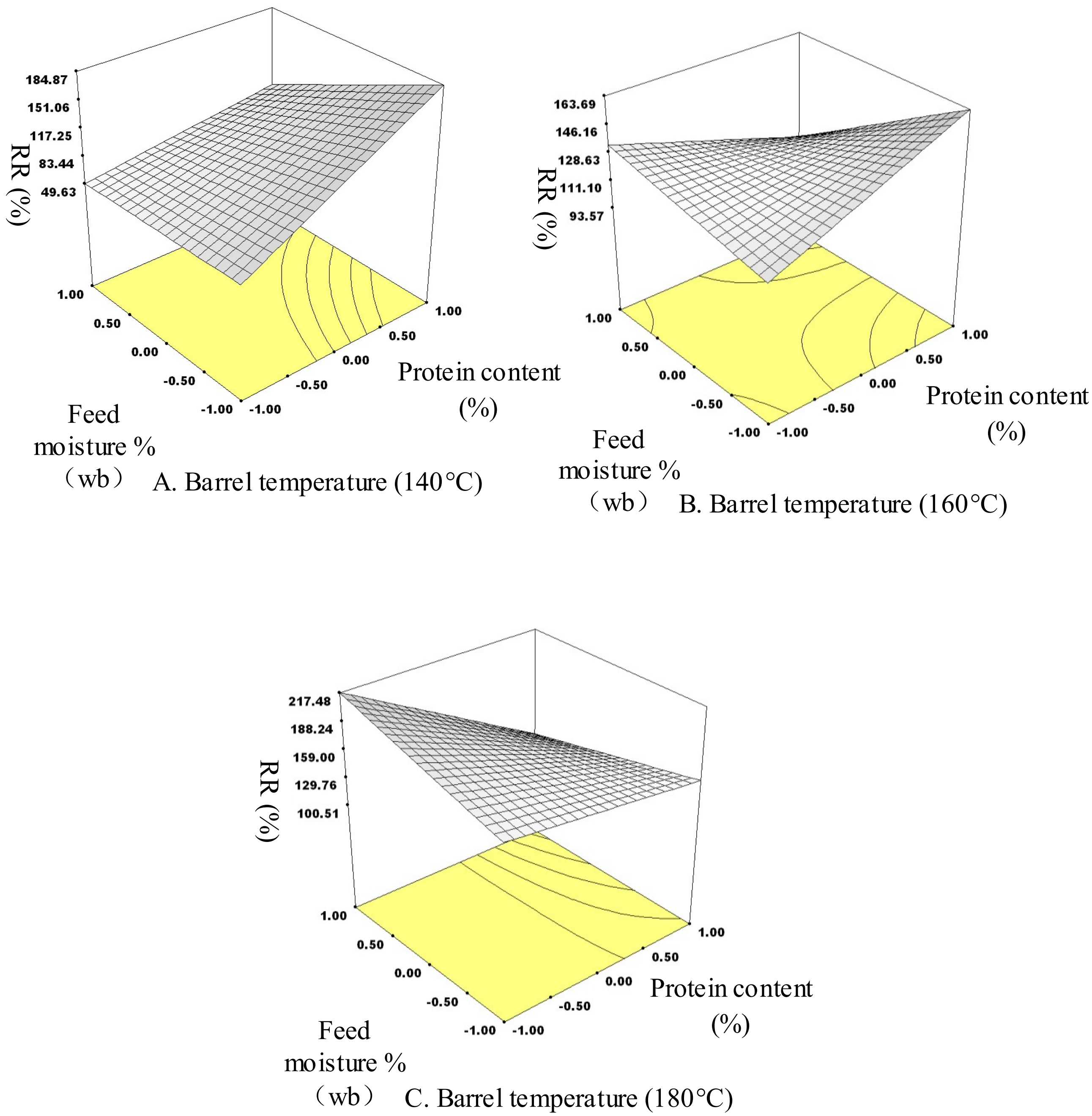


Figure 5.5 Response surfaces plot of rehydration ratio (RR) of soy protein isolate-corn starch blend extrusion for the effect of soy protein content and moisture content

5.3.6 Color

Color is one of the most vital attributes of any food product due to consumer acceptability considerations. The color of extruded products was measured in terms of Hunter L (lightness), a (redness), and b (yellowness) values. The values of L, a, and b under different designed experimental conditions are given in Table 5.2. The results indicated that the L values of extruded products varied between 74.2 and 87.4, a-values ranged from 0.12 to 3.47 and b values varied from 23.1 to 35.4. L value increased with decreasing soy protein isolate content and increasing processing temperature. The a-value increased with increasing protein content and process temperature. The highest b value was found while processing was done at 180°C with 60% soy protein isolate and 35% moisture content. Sun and Muthukumarappan (2002) found similar results in soy-based extruded products.

The regression equation for the relationship between L value (Y_L) and independent variables in terms of coded variables is presented in Table 5.3, 2FI model was selected according to Design Expert 6.0, the "a" and "b" value did not yield any significant model, so only L was selected as the color variable. ANOVA (Table 5.4) showed that linear term of protein content, and interaction between protein content and moisture content significantly affected the L value. The response surfaces plot of L value of extruded products are presented in Figure 5.6. They showed that the protein content had a negative impact on L value. But L value increased with deceasing feed moisture when the protein content was at low, and L value also increased with increasing processing temperature. This may happened due to Maillard reaction between amino groups and carbonyl groups, which leads to browning in the extruded products. Low feed moisture and high processing temperature are good candidates for the Maillard reaction (Singh *et al*., 2007).

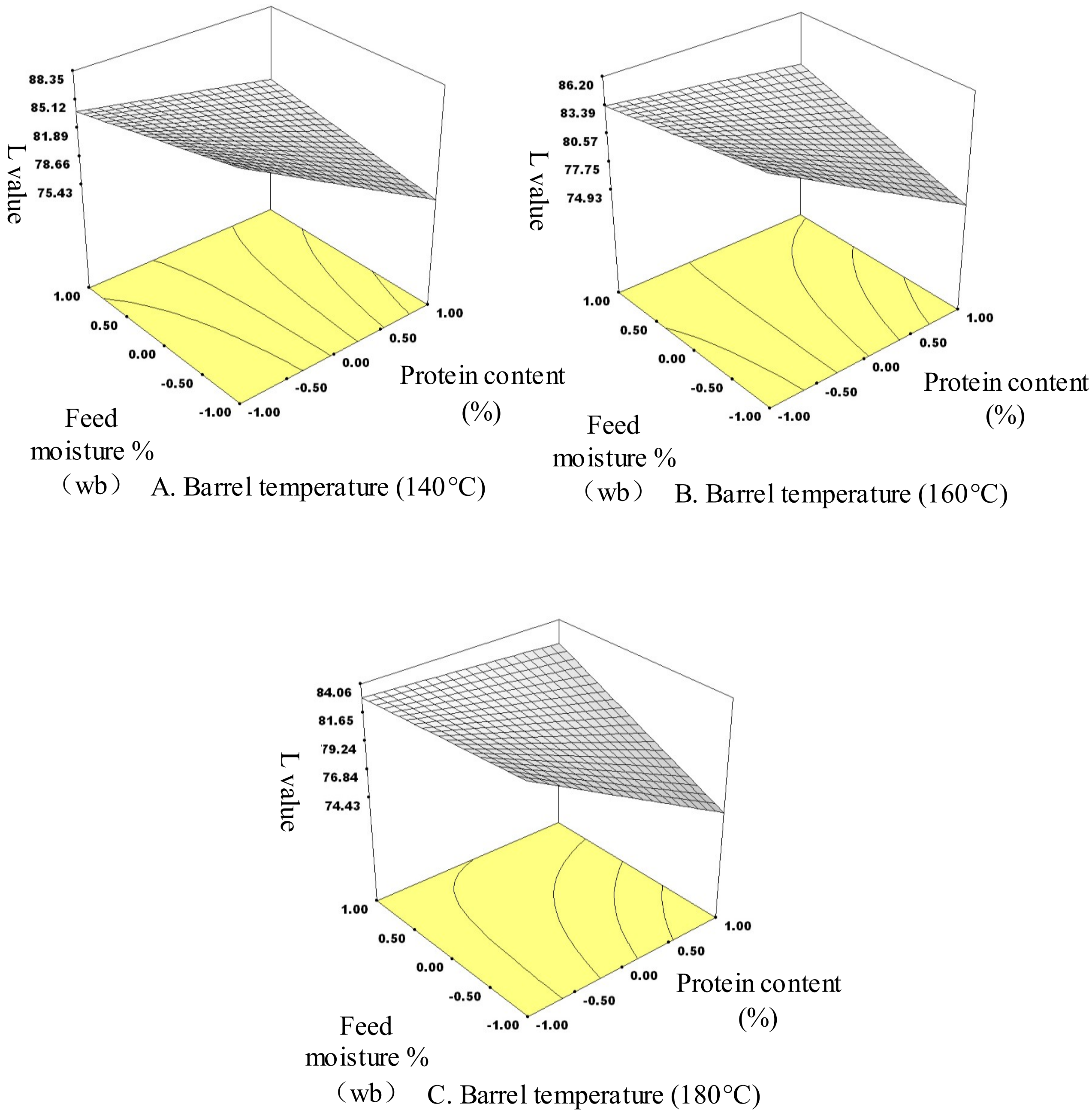


Figure 5.6 Response surfaces plot of color (L value) of soy protein isolate-corn starch blend extrusion for the effect of soy protein content and moisture content

5.3.7 Optimum extrusion conditions and characterization of response surfaces of physical properties

In this study, the optimization was applied within the experimental range of total protein content, raw material moisture content and barrel temperature for selected dependent variables to be maximized or minimized either independently or in combination. Second-order polynomial models obtained in this study were utilized for each response in order to determine the specified optimum drying condition. Different thematic scenarios based on economical and industrial constraints were considered. After finding the best solution, a graphical method was applied for mapping the optimum conditions range.

Two types of extruded products were considered in terms of recognizing the importance of different physical properties. The first one was extruded cereal flakes or chunk type of product which are normally soaked in milk prior to consumption. The second type of products were those that can be directly consumed as a snack food. Different physical properties were chosen for the two types to optimize the extrusion condition with Design Expert software.

For the products of the first type that can be consumed with milk, WSI, RR, BD were used to optimise the process condition. Table 5.6 provides typical optimum conditions for this type of products. For the products which may served directly as a snack food, BS, BD and ER were used to optimise the process condition, and optimum conditions are shown in Table 5.7.

For the first product type, various response constraints were considered. As can be seen from Table 5.6, BD was first minimized while other parameters were permitted to remain within the experimental range (Run 1). The results show for this condition, 0.42 (g/ml) BD, 3.51 (%) WSI and 135 (%) RR obtained under the coded operation condition 0.61 (54% real) for protein content, -0.14 (34%, wb real) for raw material moisture content and 0.74 (169C real) for barrel temperature with the maximum desirability value of 1.0. In Run (2), WSI was maximised while keeping other variables in the range. In this constraint, the desirability was 0.99, and in the run (3), RR was selected as the middle level (125) between the RR range of 100 and 150, a desirability of 1 was obtained under this condition. Run (4) is a combination of maximising WSI and minimising BD, with a compromised desirability of 0.74. Run (5) is a combination of average RR and minimising BD, with a desirability of 1.00. Run (6) is a combination of average RR and Maximum WSI with a desirability of 0.82, and run (7) are all mixed results and give lower desirability 0.62. One can hypothesize the reason for each of the constraint depending on the situation. BD minimised to avoid very porous product which will soak quickly and become soggy. Likewise RR was averaged to make a balance of milk uptake and crunchy feeling. These two factors combined and provided a high desirability index.

In the second set of samples, certain other response constraints were considered. As can be seen in the Table 5.7, BS was minimised while other parameters were allowed to be in the experimental range (Run 1). The results show for this condition, 0.12 (N/mm^2) BS, 1.44 ER and 79.7 (L value) were obtained under the coded operation condition 0.97 (56% real) for protein content, 1.0 (41%, wb real) for raw material moisture content and -0.98 (148C real) for barrel temperature with a high desirability of 1. In Run (2), ER was maximised while keeping other variables in the range. In this constraint, again the desirability was 1, and in the Run (3), L was maximised and again a desirability of 1 could be obtained. Run (4) is a combination of maximising ER and minimising BS, a compromising situation with a low desirability of 0.67. In Run (5) L was maximised and BS was minimised, with a slightly higher desirability index of 0.86. Run (6) maximised ER and L with an achieved desirability of 0.75 and Run (7) gave an even lower desirability of 0.63.

The above two are just hypothetical scenarios. Other possibilities exist like incorporation in to fruit mixes, in cooking preparations, soups, ice creams, etc. The properties that are important in the product must first be considered prior to evaluating the process for optimization.

Table 5.6 Results of optimization by desirability function based on products served in liquid

Run	constraints	Protein content, P (%)	Moisture content, w(db%)	Temperature, T (°C)	BD (g/ml)	WSI (%)	RR (%)	Desirability
1	Min BD							
		0.61	-0.16	0.74	0.42	3.51	135.84	1
		0.49	0.10	0.91	0.42	3.38	137.55	1
		0.30	-0.16	0.82	0.42	3.38	144.09	1
2	Max WSI							
		1.00	1.00	-1.00	0.72	6.77	86.62	0.99
		1.00	0.97	-1.00	0.72	6.71	88.23	0.98
		1.00	0.76	-1.00	0.72	6.32	98.18	0.88
3	Average RR							
		0.43	-0.14	-0.39	0.55	3.62	125.01	1
		-0.23	-0.53	-0.11	0.53	3.09	124.99	1
		-0.02	0.45	0.12	0.47	3.17	124.99	1
4	Min BD +Max WSI							
		1.00	-1.00	1.00	0.41	4.96	142.51	0.74
		1.00	-0.98	1.00	0.41	4.92	142.04	0.74
		-0.21	1.00	-1.00	0.55	5.60	64.21	0.71
5	Min BD +Average RR							
		0.95	-0.08	0.95	0.41	3.84	125.00	1.00
		0.93	-0.02	0.87	0.42	3.74	125.00	1.00
		0.97	-0.07	0.85	0.42	3.80	125.00	1.00
6	Max WSI + Average RR							
		1.00	0.22	-1.00	0.74	5.47	124.99	0.82
		0.97	0.20	-1.00	0.73	5.41	124.99	0.81
		1.00	0.20	-0.75	0.68	4.95	124.99	0.74
7	Max WSI + Average RR+Min BD							
		1.00	0.17	-0.51	0.63	4.52	124.99	0.62
		1.00	0.17	-0.50	0.63	4.51	124.99	0.62
		1.00	0.18	-0.55	0.63	4.58	124.99	0.62

Table 5.7 Results of optimization by desirability function based on products directly served.

Run	Constraints	Protein content P	Moisture content M(db)	Temperature T	BS (N/mm^2)	ER	L	Desirability
1	Min BS							
		0.97	1.00	-0.98	0.12	1.44	79.70	1
		0.87	1.00	-1.00	0.14	1.45	79.88	1
		-0.97	1.00	-1.00	0.14	1.39	83.78	1
2	Max ER							
		0.52	-0.18	0.28	0.74	1.77	79.14	1
		0.56	-0.10	0.29	0.74	1.77	79.16	1
		0.58	-0.17	0.33	0.74	1.77	78.92	1
3	Max L							
		-0.95	-0.94	-0.95	0.68	1.33	87.78	1
		-0.98	-0.67	-0.98	0.66	1.37	87.44	1
		-0.86	-0.97	-0.98	0.68	1.33	87.39	1
4	Min BS + Max ER							
		1.00	0.60	-0.72	0.32	1.57	79.01	0.67
		1.00	0.59	-0.74	0.32	1.56	78.98	0.67
		1.00	-1.00	-0.06	0.44	1.65	74.98	0.66
5	Min BS + Max L							
		-1.00	0.98	-1.00	0.14	1.38	83.89	0.86
		-1.00	0.99	-0.98	0.14	1.39	83.86	0.86
		-1.00	0.96	-1.00	0.15	1.39	83.94	0.85
6	Max ER + Max L							
		-0.54	-0.11	-0.04	0.74	1.67	83.30	0.75
7	Min BS + Max ER + Max L							
		-0.71	1.00	-0.57	0.32	1.51	83.14	0.63
		-0.71	1.00	-0.58	0.31	1.51	83.14	0.63
		-0.71	1.00	-0.61	0.31	1.50	83.14	0.63

5.4 Conclusions

Extrusion processing variables consisting of protein content, feed moisture content and processing temperature significantly influenced the physical properties (BS, BD, ER, WSI, RR and color) of the extruded products. BS and BD increased with increasing protein content, but at higher protein content in blend resulted in decreasing ER, WSI, RR and L value. Higher feed moisture played a very important role to increase BS, BD and L value and decrease WSI and RR. ER showed a maximum cap which considered with the various process variables. Higher extrusion processing temperature showed a dominant effect to increase BS, ER, RR and L value and decrease BD and WSI. The optimum extrusion processing temperature for BS, BD, ER, WSI, RR and color need to be looked at with respect to the intended type of product and physical property desired. This type of study will be useful in identifying desirable operating conditions for targeted extruded products.

Connective statement to Chapter 6

Chapter 5 studied the physical properties of high protein content extruded products, and it also optimized the processing variable to get a selected best products. All these products are dried products, the moisture content of the extruded products are really high and showed a high water activity when it just came out of the extruder, which is not stable, in order to get dry products, we used an air convection oven to dry the products under certain drying condition. During the drying process, we found the drying behaviors of various products are not the same, and the final moisture content of the products are also not exactly the same. Thus a study of the different parameter effect to the drying behavior shows the importance. In Chapter 6, the drying behaviors related to different processing parameters are studied.

CHAPTER 6 DRYING CHARACTERISTICS OF EXTRUDED CORN-SOY PROTEIN ISOLATE FORMULATIONS

Abstract

Different blends of soy protein isolate and corn flour were prepared based on a CCRD design, with variables of protein content (32.2-66.6%), feed moisture content (31.6-48.4%) and processing temperature (126.4-193.6°C). Samples extruded under different conditions were air-dried at 55°C in a convection oven with the air flow rate controlled at 0.1 m/s. The moisture content and water activity of test sample were evaluated every 15 minutes during the course of drying, and the data were used to characterize the drying curve as well as the associated moisture sorption isotherms. A model based on the CCRD design was developed for predicting the moisture ratio of the products.

6.1 Introduction

Extrusion cooking is widely used in food industry; it offers continuous processing while maintaining significant nutrient levels (Guy, 2001). Extrusion of corn flour products is one important category in the extrusion products. However, the nutrition profiles of this kind of products are generally associated with low proportion of protein. Hence soy protein isolate (SPI) commonly is added to starchy sources to enrich the protein content and modify the physiochemical properties (Konstance *et al.,* 1998). The use of SPI has been of increased interest, primarily attributed to its high nutritional value, steady supply, and low cost compared to other sources of protein. Furthermore, soy proteins are widely used in food applications due to their functionality and health benefits (Liu, 1997; Riaz, 2006). Generally, incorporation of soy protein can significantly affect the mechanical, physico-chemical and microstructure properties of foods. Therefore, the modification of these properties could play a significant role on drying behavior and characteristics of protein rich extruded products.

Selection of drying process parameters impacts both cost efficiency and product quality (Keey, 1992). The effect of drying condition on product quality (Geankoplis 1978), color (Chua and Chou, (2004), density (Talla *et al.*, 2004) and texture (Ahrne *et al.*, 2003; Lewicki and Jakubczyk, 2004) have been well studied. In addition to drying condition, initial composition of the material before the extrusion process and extrusion conditions also influence the final properties of extruded products. Nalesnik *et al.*, (2007) studied the combined effect of extrusion and drying conditions on color and texture properties of whey protein concentrates and isolates, and indicated that drying temperature and time were the critical factors in determining desired texture and color of the product. .

In protein rich extruded products, the interaction of protein with carbohydrate significantly affects their functional properties (Onwulata *et al.*, 2003) and subsequently their post-extrusion drying behavior. Depending on the extrusion conditions, soy protein enriched starch formulations will have their own unique properties under different drying conditions. Therefore it is important to find evaluate drying conditions for better shelf life stability and quality of extrution-dried products. The moisture content of extruded products is too high before drying step for achieving shelf stability, and therefore, post-extrusion drying is the final step in production of shelf stable extruded products. In previous chapters (Chapters 3 and 5) physical characteristics of extruded corn flour-SPI blends in low (20% SPI to 80% corn flour ratio) and high protein (33.2%-66.7%) compositions were studied. In both cases, the water activities of extruded samples were found to be more than 0.9. The maximum water activity value for shelf stability of the dried products is 0.75 at room temperature. Therefore, it is necessary to find drying conditions that would reduce the water activity of extruded samples to safe levels in order to provide shelf-stability. Further, the quality and stability of such products are expected to be dependent on the many input variables caused by the extrusion process.

Therefore, in this chapter, the drying characteristics of the extruded products were analyzed and related to extrusion process variables.

6.2 Materials and Method

6.2.1 Materials

Corn flour from Brar Natural Flour Mills (Winnipeg, MB) was purchased locally, which contained 1.7% of lipids, 76.7% of starch, 10% of protein and 12% moisture. Soy protein isolate (SPI) was received from American Health and Nutrition (Ann Arbor, MI), which contained 90% protein, 5% carbohydrates and 5% moisture. Soy flour was bought from Soyador (Quebec, Canada) with protein content of 40%, lipid 22%, carbohydrate 33.5% and moisture 4.5%.

6.2.2 Experimental design

In this study 20 experimental conditions were employed according to a central composite rotatable design (CCRD) using Design Expert Software (Version 6.0 State-Ease, Inc., Minneapolis, MN) with three variables and five levels for each variable (Khuri 1989). Experiments were performed in a random order including six central replicate points to minimize the effect of experimental errors. The independent variables were protein isolate (32.2-66.6%), feed moisture content (31.6-48.4%) and extrusion temperature (126.4-193.6°C). The experimental designs for actual and coded variables are indicated in Table 6.1. A second order polynomial model (Eq. 6.1) which included all the linear, quadratic and interaction terms was used to estimate the predictive responses.

$$Y = \beta_O + \sum_{i=1}^{n} \beta_i x_i + \sum_{i=1}^{n} \beta_{ii} x_i^2 + \sum_{i=1}^{n} \sum_{j=i+1}^{n+1} \beta_{ij} x_i x_j \quad (6.1)$$

Where Y represents response variable, β_0 is the interception coefficient, β_i, coefficient of the linear effect, β_{ii} the coefficient of quadratic effect and β_{ij}, the coefficient of interaction effect. Where x_i and x_{ij} denote the coded levels of variable X_i and X_j investigated in experiments. The variable X_i was coded as x_i according to Equation (6.2):

$$x_i = \frac{X_i - X_o}{\Delta X_i} \quad (6.2)$$

Where x_i is (dimensionless) coded value of the variable X_i, X_o is the real value of X_i at the center point (zero) level, and the ΔX_i is the step change value.

6.2.3 Raw material blend preparation for extrusion

The quantities of the SPI, corn-flour and moisture were determined as detailed in Chapter 5. The details of the different test runs with coded (and real) values of the process variables as well as the amount of ingredients added for a 2.0 kg batch of ingredients (excluding moisture) are shown in Table 6.1. The flours were mixed by using a Hobart mixer (Hobart Food Equipment Group Canada, North York, ON) operating at a medium speed. Appropriate amounts of water were added to adjust the mixture to the required moisture content, according to the experimental design. The wetted blends were mixed for 20 min in the Hobart mixer before use.

Table 6.1 Experimental (CCRD) design with coded and (actual) values for protein content (P), moisture content (M), and processing temperature (T), along details of the quantities of soy protein isolate (SPI), soy flour, corn flour and water added for each 2.0 kg batch of ingredients.

Run	P (%)	M (wb%)	T (^oC)	SPI (g)	soy flour (g)	Corn flour (g)	water (g)
1	-1 (40)	-1 (35)	-1 (140)	633	67	1300	790
2	1 (60)	-1 (35)	-1 (140)	1095	108	797	842
3	-1 (40)	1 (45)	-1 (140)	633	67	1300	1298
4	1 (60)	1 (45)	-1 (140)	1095	108	797	1359
5	-1 (40)	-1 (35)	1 (180)	633	67	1300	790
6	1 (60)	-1 (35)	1 (180)	1095	108	797	842
7	-1 (40)	1 (45)	1 (180)	633	67	1300	1298
8	1 (60)	1 (45)	1 (180)	1095	108	797	1359
9	-1.68(33.2)	0 (40)	0 (160)	478	53	1469	1004
10	1.68 (66.6)	0 (40)	0 (160)	1252	122	626	1098
11	0 (50)	-1.68(31.6)	0 (160)	862	87	1051	676
12	0 (50)	1.68 (48.4)	0 (160)	862	87	1051	1547
13	0 (50)	0 (40)	-1.68(126.4)	862	87	1051	1051
14	0 (50)	0 (40)	1.68 (193.6)	862	87	1051	1051
15	0 (50)	0 (40)	0 (160)	862	87	1051	1051
16	0 (50)	0 (40)	0 (160)	862	87	1051	1051
17	0 (50)	0 (40)	0 (160)	862	87	1051	1051
18	0 (50)	0 (40)	0 (160)	862	87	1051	1051
19	0 (50)	0 (40)	0 (160)	862	87	1051	1051
20	0 (50)	0 (40)	0 (160)	862	87	1051	1051

6.2.4 Extrusion process

A co-rotating twin screw extruder (DS32-II, Jinan Saixin Food Machinery, Shandong, P. R. China) was used in all extrusion processes. Details are given in Chapters 3, 4 and 5. The diameter of the screw was 30 mm. The length to diameter ratio of the extruder barrel was 20:1. The diameter of the hole in the die was 5 mm with a die length of 27 mm. A constant screw speed of 100 rpm was selected based on previous experimental results, and to limit the number of process variables to three. The extruder was fed automatically through a conical hopper, keeping the flights of the screw fully filled and avoiding accumulation of the material in the hopper.

After stable conditions were established, extruded products were collected and cut into 35 mm long sections and air-dried at 55°C and ~15% relative humidity in a convection oven with the air flow rate controlled at 0.1 m/s. Since age numbers of extruded samples were involved, drying was limited to one set of simple air drying parameters. The sample weight was continuously recorded at 15 min intervals during drying period to monitor the weight loss. From a second batch subjected to the same condition, test samples were withdrawn every 15 minutes and sealed in air-tight plastic containers for additional tests. The drying process was stopped after the equilibrium condition reached.

6.2.5 Determination of moisture content

The moisture content was measured gravimetricaly by the AOAC-984.25 (AOAC 1995) method: samples were put into a glass pan and dried in a conventional oven (Fisher Scientific Isotemp Oven, Asheville, North Carolina) at 105 ± 1°C until constant weight. The moisture content (wet basis) was calculated as follows:

$$M(wb\%) = 100 \text{ x } (W_o - W_d)/(W_o - W_p) \quad (6.3)$$

W_o = Weight of the sample with the pan before dried

W_d = Weight of the sample with the pan after dried
W_p = Weight of the pan

The moisture content M (dry basis) was obtained as follow:

$$M(db) = M(wb)/[1-M(wb)] \quad (6.4)$$

6.2.6 Determination of water activity

The water activity was measured by a water activity anylizer (ROTRONIC HygroLab 3, Rotronic Instrument Corp., NY), at room temperature 25°C, samples were put into the chamber of the water activity anylizer until the signal of equilibrium appeared, water activity value was recorded.

The water activity of food greatly affects the growth of microorganisms and it is the principal factor which is linked to the microbial growth. A water activity level of 0.75 is normally considered to be a safe level, because most of bacteria will not grow and cause problems below this level (Ramaswamy and Marcotte, 2006)

6.2.7 Determination of moisture ratio (MR)

Moisture ratio is the percentage of free moisture left in the products, it can be described as following:

$$MR = (Mt-Me)/ (Mi-Me) \quad (6.5)$$

Where:
Mt = moisture at time t
Me = moisture at equivalent condition
Mi = moisture at initial time

6.3 Results and Discussion

6.3.1 Moisture content and water activity results after the extrusion process

The experimental arrangement (test run number), data on moisture content, water activity after the extrusion process (0 minute drying) are shown in Table 6.2. Some of the moisture in the extrudate was evaporated when the extrudate exit the extruder, this can be seen by the comparison of moisture content in the raw material and the extrudate (Figure 6.1). For example in run 1, the moisture content in the raw material is 35% on wet basis, which is equal to 53.8% on dry basis, compared with the extrudate moisture content which is 44.3% on dry basis, the difference before and after extrusion process caused by the moisture evaporation when the extrudate exited the die. Although some of the moisture in the products is evaporated, the water activity is still high in the extruded products, all of the products have water activity more than 0.9, which can offer a very good growth environment to microorganisms. This situation means the products must have some further treatment to get a shelf stable condition, in this study, further drying is selected.

The regression equations of moisture content of the extruded products before drying is as following:

$$Y = 40.2-0.04*P+4.24*M-5.40*T \quad (6.6)$$

Where: P for protein content; M for moisture content; T for barrel temperature

Table 6.2 experimental arrangement and moisture content, water activity after extrusion and sample moisture content, drying time when the sample a_w reach 0.75

Run	Protein content, P (%)	Moisture content, M (%wb)	Temperature, T (°C)	Extrudate Moisture content (db) (0min)	Extrudate Water activity (0min)	Drying time at a_w(0.75) (min)	Moisture content at a_w(0.75) (db)
1	-1 (40)	-1 (35)	-1 (140)	44.3	0.969	137	13.62
2	1 (60)	-1 (35)	-1 (140)	40.8	0.966	127	17.57
3	-1 (40)	1 (45)	-1 (140)	47.1	0.964	151	21.49
4	1 (60)	1 (45)	-1 (140)	52.4	0.97	124	24.24
5	-1 (40)	-1 (35)	1 (180)	30.9	0.94	121	12.84
6	1 (60)	-1 (35)	1 (180)	31.6	0.945	65	18.95
7	-1 (40)	1 (45)	1 (180)	36.4	0.966	167	17.33
8	1 (60)	1 (45)	1 (180)	37.9	0.968	110	21.06
9	-1.68 (33.2)	0 (40)	0 (160)	43.9	0.963	142	14.34
10	1.68 (66.6)	0 (40)	0 (160)	41.2	0.971	108	20.96
11	0 (50)	-1.68 (31.6)	0 (160)	31.9	0.955	99	15.65
12	0 (50)	1.68 (48.4)	0 (160)	50.8	0.969	139	21.32
13	0 (50)	0 (40)	-1.68 (126.4)	46.6	0.979	136	20.92
14	0 (50)	0 (40)	1.68 (193.6)	31.2	0.938	97	17.61
15	0 (50)	0 (40)	0 (160)	36.6	0.959	135	19.89
16	0 (50)	0 (40)	0 (160)	43.5	0.969	120	17.87
17	0 (50)	0 (40)	0 (160)	39.9	0.962	130	19.95
18	0 (50)	0 (40)	0 (160)	38.7	0.952	126	18.30
19	0 (50)	0 (40)	0 (160)	39.3	0.95	122	18.14
20	0 (50)	0 (40)	0 (160)	38.9	0.951	125	18.22

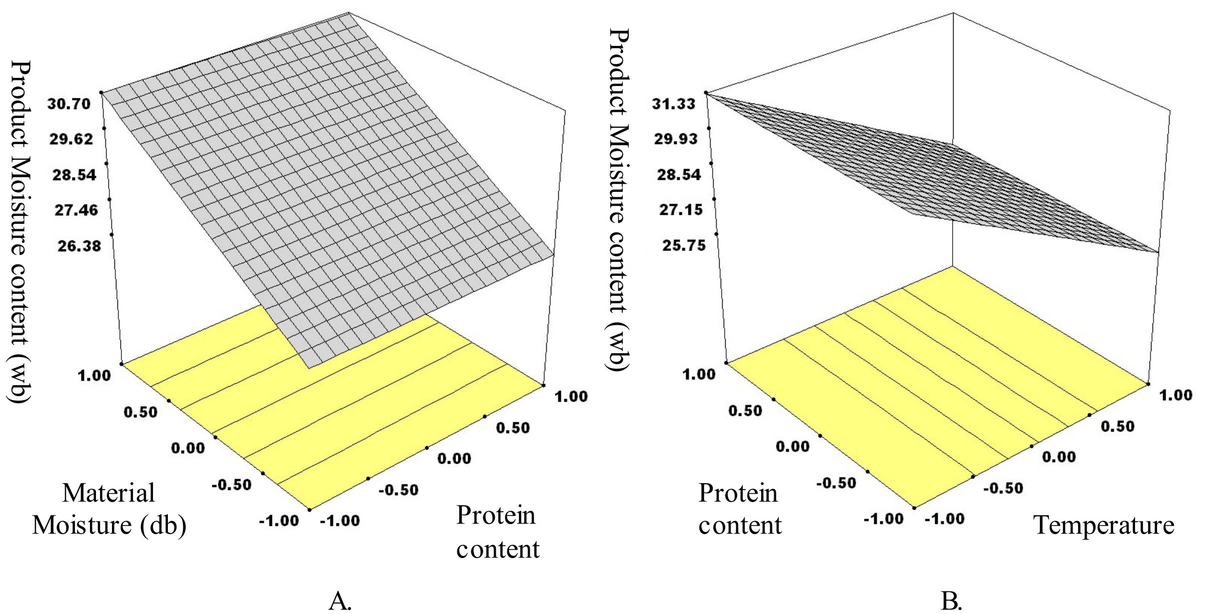


Figure 6.1: Response surface plot of product moisture content (wb) after extrusion of soy protein isolate-corn starch blend extrusion for the effect of soy protein content and moisture content in the raw material

6.3.2 Impact of different variable to the moisture content and the water activity of the sample during the drying process

Moisture content and water activity of the product is affected by different processing variables. Figure 6.2 is the moisture content of the extrudate during the drying process. Figure 6.2-A shows the different protein content affecting the moisture content when the raw material have a moisture content of 40%(wb) and processing barrel temperature 160°C, and according to the experiment results, moisture content decreased with the increasing of drying time, and the product with lowest protein content (33.2%) experiencing moisture loss faster then those with a higher protein content. Figure 6.2-B is the moisture content effect to the drying process, the results shows a regular decreasing of moisture content in the extruded products during the drying process and higher initial moisture content will result a higher moisture product after a given treatment time. Figure 6.2-C is the processing barrel temperature effect to the drying process, again, the results shows that higher processing temperature will result lower product moisture content at the same drying time.

Figure 6.3 is the water activity of the extrudate during the drying process. Figure 6.3-A shows different protein content affecting the a_w of the products during the drying process. The results show that the highest protein content (66.7%) products have a rapid decreasing in a_w after the drying time of 75 min, it gives a much lower a_w than the other two products (33.2% and 50% protein content) at 120 min drying time. Higher initial moisture content lead to higher a_w is found in Figure 6.3-B, and Figure 6.3-C shows that highest processing temperature (180°C) will cause low a_w in the products, and lower processing temperature 140°C and 160°C does not show big different in a_w reducing.

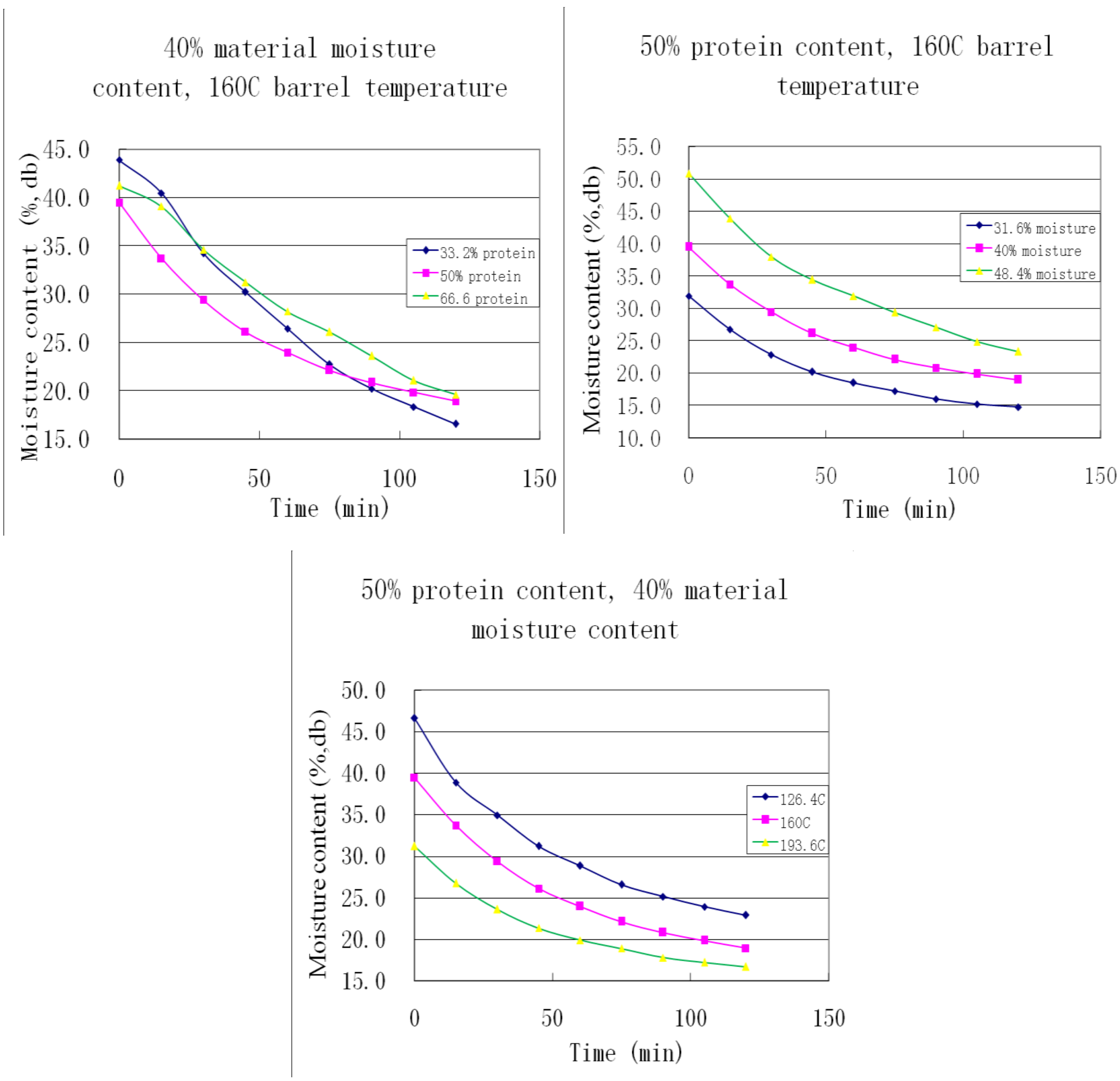


Figure 6.2 Impact of different variable to the moisture content of the sample during the drying process (actual value)

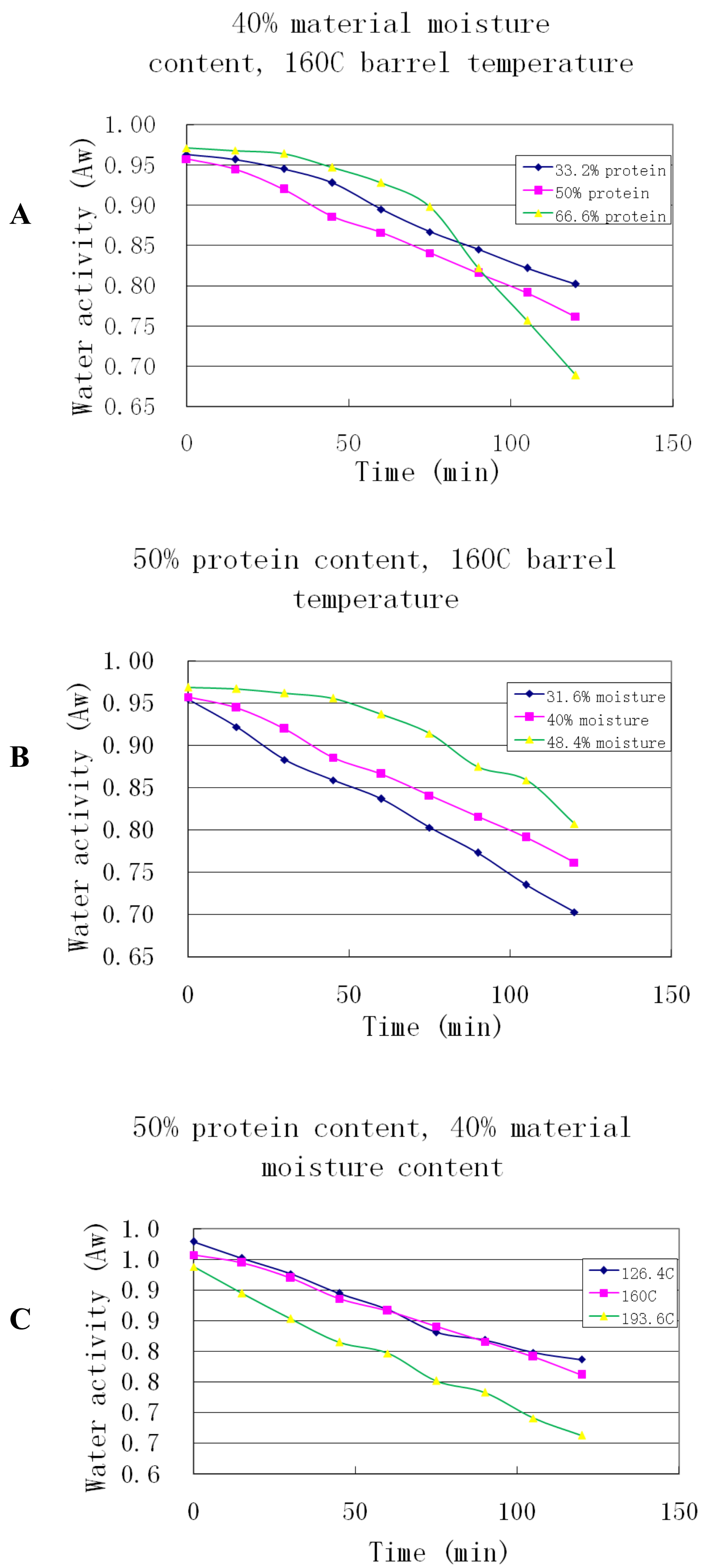


Figure 6.3 Impact of different variable to the water activity of the sample during the drying process (actual value)

6.3.3 Drying time to reach water activity 0.75

Based on experimental drying conditions and the removal of moisture, drying time required achieve the water activity level of 0.75 were determined, as listed in Table 6.2, the longest drying time appeared at run 7 with 40 percent of protein content, 45 percent of material moisture content and 180°C barrel temperature, and the moisture content at a_w 0.75 in that run is only 17.3% on dry basis. The shortest drying time for reaching the a_w 0.75 appeared in run 6 with 60 percent of protein, 35 percent of moisture and 180°C barrel temperature, the moisture content is 19% dry basis.

Figure 6.4 shows the response surface plot of time when of the extruded products' water activity reach 0.75 during the drying process. Figure 6.4-A and Figure 6.4-B clearly show that with the increase of protein content in the products, the drying time of reaching a_w 0.75 is decreased, and Figure 6.4-B and C show that with the increasing of barrel temperature, the drying time is also decreased. Figure 6.4-A, C shows that the increasing of raw material moisture content will result in increasing of drying time.

The extruded products' drying behavior are affected by different variables, the moisture content of the extruded products' a_w reach 0.75 are different, which is an important processing factor to the industry. Figure 6.5 is the response surface plot of product moisture content when the extruded products water activity reach 0.75 during the drying process. Figure 6.5-A, B shows that the increasing of protein content in the product will result a final higher moisture content in the products when the a_w reach 0.75. The increasing of moisture content in the raw material is also increased the final moisture content, especially in higher protein content product.

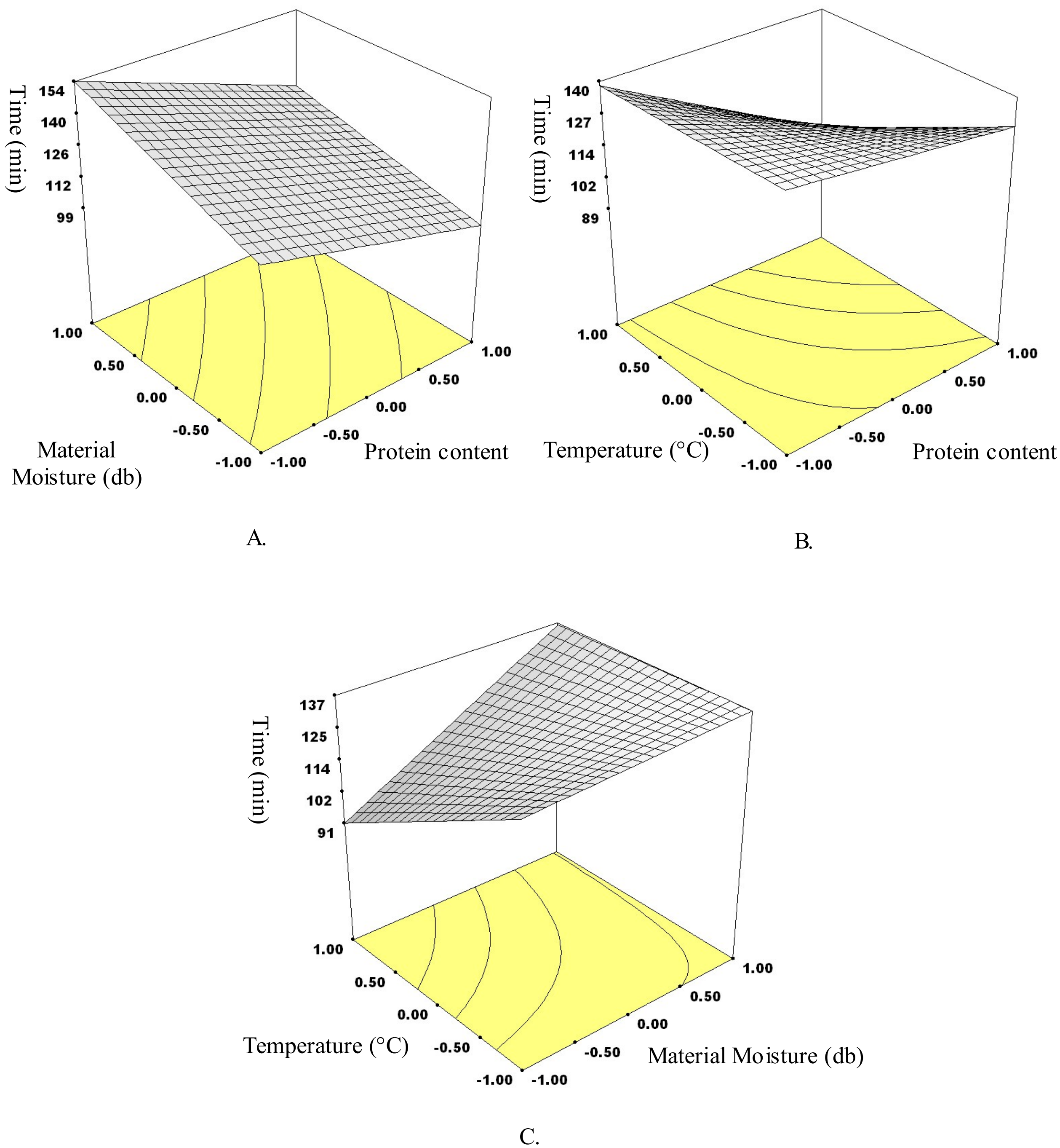


Figure 6.4 Response surface plot of time when of the extruded products water activity reach 0.75 during the drying process

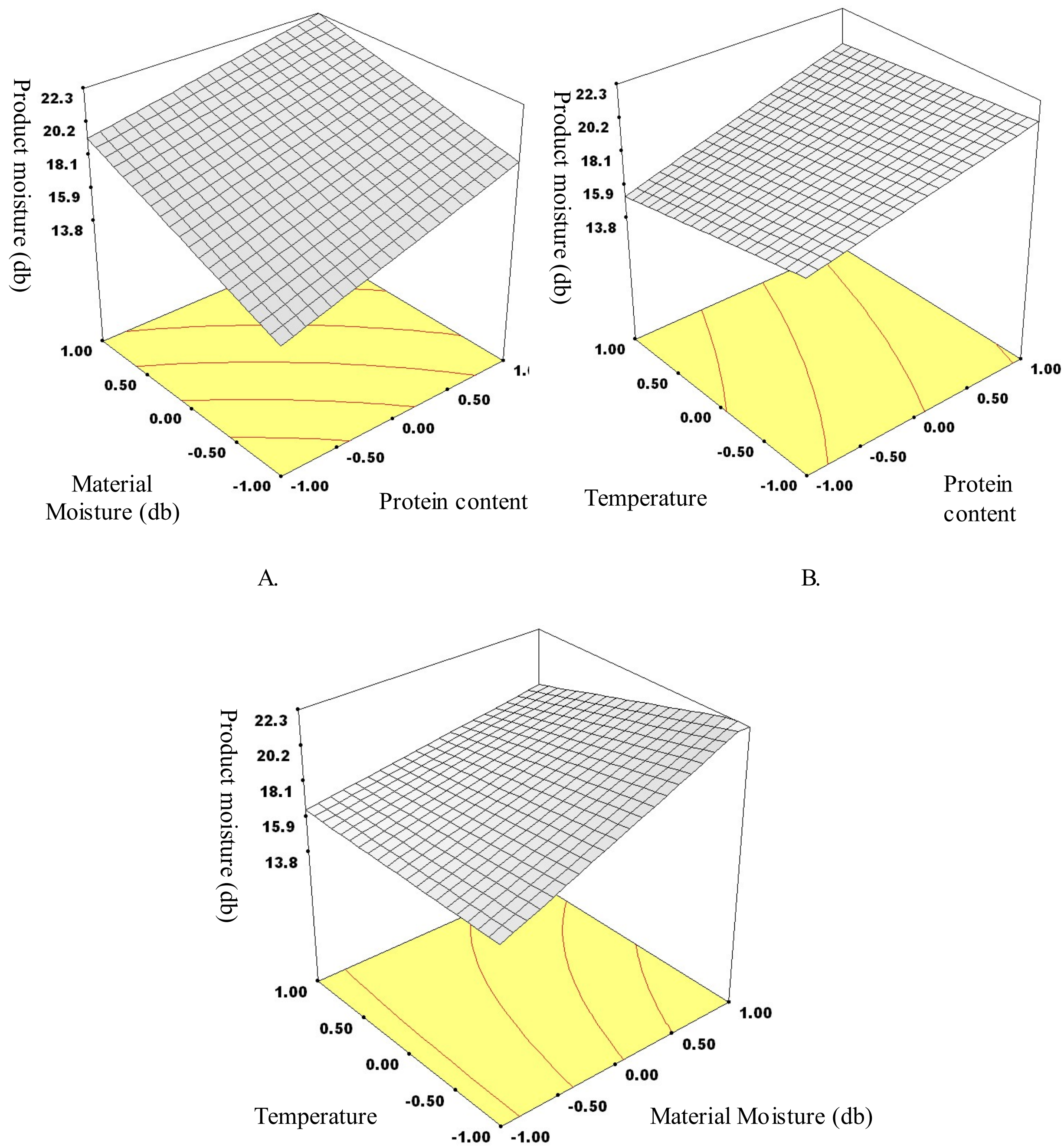


Figure 6.5 Response surface plot of product moisture content when of the extruded products water activity reach 0.75 during the drying process

ANOVA for the condition when the water activities of the sample reach 0.75 are listed in Table 6.3. Total model gives good prediction correlation between experimental and prediction value both of the time and moisture content when a_w reach 0.75($p<0.05$). All the barrel temperature, moisture content and protein content are significantly affecting the drying time and moisture content when a_w reach 0.75 in linear level. The interaction between barrel temperature and protein content, raw material moisture content and barrel temperature are significantly to the drying time when the a_w reach 0.75. The interaction between raw material moisture content and barrel temperature is significantly affected the extruded products moisture content when a_w reach 0.75.

Table 6.3 ANOVA for the condition when the water activities of the sample reach 0.75

	Time when a_w at 0.75			Moisture content when a_w at 0.75		
Wb	Coefficient Estimate	Sum of Squares	Prob > F	Coefficient Estimate	Sum of Squares	Prob > F
Model	124	8282.1	< 0.0001	18.51	146.79	< 0.0001
P	-15.2	3138.8	< 0.0001	2.03	56.08	< 0.0001
M	12.4	2102.2	< 0.0001	2.25	68.89	< 0.0001
T	-10.4	1467	0.0001	−0.90	11.10	0.0028
PM	-2.3	40.6	0.3812	−0.45	1.59	0.1874
PT	-9.5	717.8	0.0022	0.39	1.24	0.2418
MT	10.1	815.6	0.0013	−0.99	7.90	0.0085
Residual		641.6			10.69	
Lack of Fit		498	0.205		6.32	0.5741
R-Squared		0.93			0.93	
Adj R-Squared		0.89			0.90	
Pred R-Squared		0.78			0.84	
Adeq Precision		22.68			19.29	

6.3.4 Modeling of Moisture Ratio (MR)

Software Design-Expert 6.0 was used to make the ANOVA and the predicted equation for moisture ratio. Three variables (Protein content, moisture content and processing barrel temperature) were used as the Numeric Factors and drying time was used as category factor (0, 15, 30, 45, 60, 75, 90 minutes) in the analysis. This procedure is slightly different from the conventional drying model which is generally based on a diffusion model. However, the purpose is same – to be able to model moisture loss with time. Table 6.4-A is the ANOVA result for the MR, moisture content and drying time are significantly affected the MR at linear level, protein and moisture content are significantly affect MR at quadratic level, the interaction between PM, PT and MT are significantly affected the MR. The lack of fit not significant indicate the model is good and the high "Pred R-Squared" 0.92 indicate the model can be well predict the result. Table 6.4-B is the final equations of MR in terms of actual factors at different drying time.

Microsoft Excel was used to find the predicted moisture ratio for the drying process. Figure 6.6 shows the comparison of model predicted Moisture Ratio with the Actual value of Moisture Ratio at middle point (protein content 50%, raw material moisture content 40% and barrel temperature 160°C). MR is decreased with the increasing of drying time, and the predicted MR is very close to the actual MR value.

Figure 6.7 shows the model predicted and calculated values of Moisture Ratio for the whole experimental range with an R^2 value higher than 0.95. The points are also well distributed around the diagonal line except at the high end. The slope coefficient is 0.9978 indicating almost perfect fit within the overall range of experiments.

Table 6.4-A ANOVA for the Protein content (P), Moisture content (M), temperature (T), drying time (t) model

Source	Sum of Squares	DF	Mean Square	F Value	Prob > F
Model	11.47	33	0.348	76.64	< 0.0001
P	0.00	1	0.003	0.69	0.4064
M	0.04	1	0.039	8.66	0.0040
T	0.01	1	0.005	1.20	0.2755
t	10.78	6	1.796	395.90	< 0.0001
P2	0.31	1	0.307	67.62	< 0.0001
M2	0.04	1	0.038	8.47	0.0044
T2	0.01	1	0.010	2.18	0.1426
PM	0.16	1	0.164	36.11	< 0.0001
PT	0.07	1	0.068	15.05	0.0002
Pt	0.00	6	0.001	0.18	0.9811
MT	0.06	1	0.057	12.60	0.0006
Mt	0.02	6	0.004	0.84	0.5434
Tt	0.00	6	0.001	0.15	0.9879
Residual	0.48	106	0.005		
Lack of Fit	0.37	71	0.005	1.60	0.0632
Pure Error	0.11	35	0.003		
Cor Total	11.95	139			
Std. Dev.	0.07		R-Squared	0.96	
Mean	0.51		Adj R-Squared	0.95	
C.V.	13.33		Pred R-Squared	0.92	
PRESS	0.90		Adeq Precision	32.00	

P: Protein content, M: Moisture content, T : temperature, t : drying time

Table 6.4-B Regression equations for extruded products moisture ratio at different drying time.

Drying Time	Final Equation of Moisture Ratio in Terms of Actual Factors
0 minute	$Y_{MR} = 0.942 + 0.055*P^2 + 0.020*M^2 + 0.010*T^2 - 0.054*PM + 0.035*PT - 0.032*MT$
15 minute	$Y_{MR} = 0.712 + 0.004*P + 0.004*M + 0.003*T + 0.055*P^2 + 0.020*M^2 + 0.010*T^2 - 0.054*PM + 0.035*PT - 0.032*MT$
30 minute	$Y_{MR} = 0.528 + 0.001*P + 0.012*M + 0.002*T + 0.055*P^2 + 0.020*M^2 + 0.010*T^2 - 0.054*PM + 0.035*PT - 0.032*MT$
45 minute	$Y_{MR} = 0.383 - 0.009*P + 0.019*M + 0.009*T + 0.055*P^2 + 0.020*M^2 + 0.010*T^2 - 0.054*PM + 0.035*PT - 0.032*MT$
60 minute	$Y_{MR} = 0.272 - 0.012*P + 0.026*M + 0.011*T + 0.055*P^2 + 0.020*M^2 + 0.010*T^2 - 0.054*PM + 0.035*PT - 0.032*MT$
75 minute	$Y_{MR} = 0.186 - 0.006*P + 0.034*M + 0.021*T + 0.055*P^2 + 0.020*M^2 + 0.010*T^2 - 0.054*PM + 0.035*PT - 0.032*MT$
90 minute	$Y_{MR} = 0.109 - 0.018*P + 0.047*M + 0.006*T + 0.055*P^2 + 0.020*M^2 + 0.010*T^2 - 0.054*PM + 0.035*PT - 0.032*MT$

P : Protein content; M: Moisture content ; T: Barrel temperature

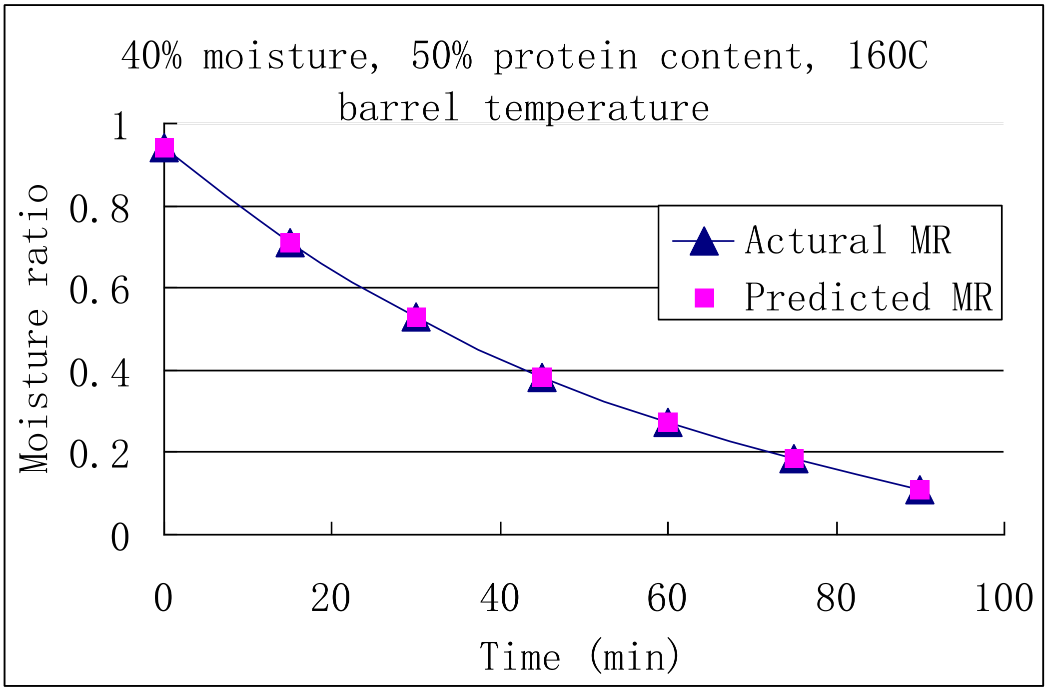


Figure 6.6 Comparison of model predicted Moisture Ratio with the Actual value of Moisture Ratio at middle point.

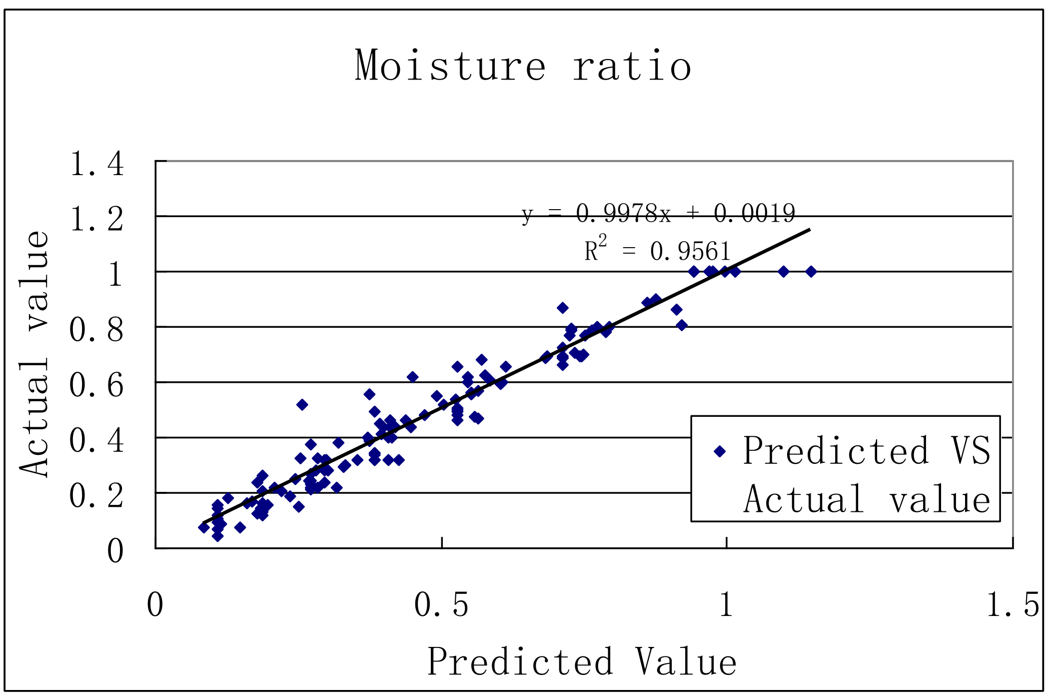


Figure 6.7 Comparison of model predicted Moisture Ratio with the Actual value

ANOVA of the p value about the moisture content and water activity (a_W) of the extruded products at different drying time are listed in Table 6.5 and Table 6.6. The result shows that the raw material moisture content and the processing temperature are significant to both extrudate moisture content and water activity during the drying process, but the protein content of the raw material is only significantly affect the moisture content after the drying time up to 75 min and significantly affect the a_w after the drying time up to 90 min. With the drying time increasing, protein content affects a_w more significant, as the drying time at 90, 105 and 120 minutes.

Table 6.5 ANOVA for moisture content of the sample at different drying times

	0min	15min	30min	45mon	60min	75min	90min	105min	120min
Model	***	***	***	***	***	***	***	***	***
P	NS	NS	NS	NS	NS	*	*	*	*
M	***	***	***	***	***	***	***	***	***
T	***	***	***	***	***	***	***	***	***
Lack of Fit	NS	NS	NS	NS	NS	NS	NS	NS	NS

NS: p>0.05, Not significant; *: 0.01<p<0.05; **: 0.001<p<0.01; ***: p<0.001,
P: Protein content, M: Moisture content, T: Barrel temperature

Table 6.6 ANOVA for water activity (a_w) of the sample at different drying times

a_w	0min	15min	30min	45mon	60min	75min	90min	105min	120min
Model	**	***	**	**	**	**	**	***	***
P	NS	NS	NS	NS	NS	NS	*	**	***
M	*	***	***	**	**	**	***	***	***
T	***	***	***	***	***	**	***	***	***
P^2		NS	*	**	*	*	NS	NS	NS
M^2		NS	NS	NS	NS	NS	NS	NS	NS
T^2		*	*	NS	NS	NS	NS	*	NS
PM		NS	NS	*	NS	NS	NS	NS	NS
PT		NS	NS	NS	NS	**	NS	*	**
MT		*	NS	NS	NS	NS	NS	NS	NS
Lack of Fit	NS	NS	NS	NS	NS	NS	NS	NS	NS

NS: $p>0.05$, Not significant; *: $0.01<p<0.05$; **: $0.001<p<0.01$; ***: $p<0.001$,
P: Protein content, M: Moisture content, T: Barrel temperature

6.4 Conclusions

Extrusion processing variables of protein content, feed moisture content and processing temperature influenced the extrudate drying process and the final products moisture content and water activity significantly. The raw material moisture content and the processing temperature are significant to both extruded products' moisture content and water activity during the drying process, but the protein content of the raw material affect the moisture content significent after the drying time up to 75 min and significantly affect the a_w after the drying time up to 90 min. Increased protein content in the products will decrease the drying time when the final end point is based on a_w 0.75, increasing barrel temperature and decreasing the raw material moisture content can shorten the drying time based on a_w 0.75 as the final drying end point. Increasing of protein content in the product will result in higher moisture content in the final products when a_w reach 0.75.

Extruded products' moisture content are decreased with the increasing of drying time, and the product containing fewer protein (33.2%) dried faster then the higher protein content products. Highest protein content (66.7%) products have a rapid decreasing of a_w after the drying time of 75 min, it gives a much lower a_w than the other two products (33.2% and 50% protein content) at 120 min drying time. The models for MR were found and can be used to predict the MR value of the products at different drying times

Connective statement to Chapter 7

The extrusion parametric effects on the drying behavior of the extruded products were studied. The extruded products had high water activity and were not optimal with respect to shelf stability. The stable water activity condition was considered to be around 0.75 and therefore the product needed to be dried to water activity levels below this. This was accomplished in Chapter 6. In addition to achieving stability many snack foods are prepared by frying to give desirable sensory characteristics. Most fried products on the commercial market are high carbohydrate foods with low proteins and with large amount of oil absorbed in to the product. It was hypothesized that the extruded products with high protein would be a better base for preparing high nutritive value fried snacks. The frying behavior of the product was therefore focused next. Frying gives the products desirable color, flavor, taste and texture. The frying process must be evaluated obtain desirable end products. So the focus of Chapter 7 was to evaluate the frying characteristics of extruded products with idea of developing a desired product with limited oil uptake.

CHAPTER 7 FRYING CHARACTERISTICS OF PROTEIN-ENRICHED SNACK FOOD FROM EXTRUDED CORN FLOUR – SOY PROTEIN ISOLATE FEED MIX

Abstract

Frying characteristics (moisture content, oil content, texture and color) in protein-enriched extruded products were evaluated in this study. Extruded products with 50% protein on dry basis and 40% moisture on wet basis were extruded in a twin screw extruder and fried in oil at different temperatures (145, 165 and 185°C) for different frying times (0-600s). The moisture content of the product reduced to below 5% after 210, 360 and 600 s frying at 145, 165 and 185°C respectively. Texture firmness in the form of breaking stress was observed to reach a peak during the frying process and then decrease with further frying. The color 'a' value increased with an increase in frying time and temperature. A high degree of correlation was observed between measured color a value, moisture content and oil content with color, flavor and overall acceptance of fried product obtained from sensory evaluation. The texture occurrence of the peak value showed poor correlation. Therefore, the instrumental measurements can be used as quality controlling tools for producing acceptable fried product.

7.1 Introduction

By volume, the most widely extruded food material is starch. In recent years the importance of extrusion cooking on the transformation of starch and starchy materials has increased. Extrusion processing is an effective way of modifying the physical material properties of polysaccharides and polysaccharide based foods. The thermo mechanical action during extrusion brings about gelatinization of starch. This occurs in a shear environment, resulting in a plasticized continuous mass flow of the feed through the die. These environmental effects bring physio-chemical changes and modification during processing. Extrusion cooking of starches results not only in expanded volume but also in modification of the microstructure of the products. Extrusion processing of polysaccharides for physical modification aims at the break down of microstructure and forming the product into specific shapes. The mechanical stress during extrusion process is possibly responsible for the breakdown of polysaccharides glucosidic bonds, leading to the release of oligosaccharides (Esposito *et al.* 2005). Generally, extrusion processing of starch constitutes a microstructure degenerating (Faraji *et al.* 2004) which has a significant effect on properties of the extrudate in post extrusion processes, for instance during frying.

Soy protein is widely used in food applications due to its functionality and health benefits (Liu, 1997; Riaz, 2006). While starch is the primary ingredient in expanded breakfast cereals and snacks, soy protein can enhance the nutritional value of these products. In terms of protein modification extrusion process has also a significant effect on modification of physio-chemical properties of a given protein. For instance, in extrusion cooking, wheat gluten is significantly involved in the microstructural and textural formation of the extruded products (Faubion and Hoseney, 1982). Research results indicate that intermolecular disulfide bonding is also the main cause of protein aggregation in the extrusion process (Faubion and Hoseney, 1982; Li and Lee 1996). Incorporation of soy protein can significantly impact the mechanical, physico-chemical and microstructural properties of foods.

SPI has many health benefits like cholesterol lowering effect, it has anticarcinogenic effects, and it deters obesity, diabetes, digestive tract irritation, and bone and kidney diseases (Friedman and Brandon, 2001; Messina and Barnes, 1991). Furthermore incorporation of soy protein into starch-based foods could be beneficial in the nutritional aspect. In addition to starch and protein further incorporation of oil in the extruded products during frying it makes the products rich in major nutrient components. This type of product may be very valuable in terms of tackling malnutrition problems in most of developing countries, where it is difficult to get balanced food at a time. Furthermore frying will modify the physical, chemical and sensorial properties of the extruded products. Therefore fried soy protein enriched corn flour may be preferred as a healthier and nutritionally more balanced shelf stable product as compared to other fried starch based products.

Protein is not only an essential component in the structure of many fried foods, but also participates in Maillard browning reaction with reduced sugar to form flavor and browning pigments. Soy protein isolate added in corn flour apart from improving the nutritional value of the extrudate it can also improve the flavor. Flavor development is a result of various chemical reactions and modification of food constituents at elevated temperature during frying. Reactions or modifications may involve breakdown of hydrogen bonding, formation of covalent bonding between amino acids, reaction of amino acids with sugar components and so on. Furthermore the color of fried foods can be seen as one of the range of input signals perceived by consumers, rather than just physical characteristics of the food. In addition to protein, starch gelatinization is crucial in frying, because it holds water and provide volume expansion (Chen *et al.*, 2001). On top of this water is one of the most constituents determined the texture of fried foods.

Acceptability of fried products by consumers is mainly evaluated using a sensory evaluation. Sensory test is widely used by food industry and recognized as a core resource in any quality control program. Sensory information as a part of a marketing decision has given it unprecedented attention. It identifies and quantitatively models the key drivers for a product's acceptance by consumers. Generally in protein-starch based extrudate frying has wide range of influence on product quality and their complex interaction in determining the texture, color and oil uptake of fried products. However the effect of post extrusion frying process on physicochemical and sensorial properties of soy protein isolate enriched corn flour has not been studied very well. Therefore, the objective of this study is to investigate the influence of frying parameters on physical properties of high protein content (50%) extruded corn flour and SPI blends and to evaluate and optimize the frying conditions (temperature and time) for an acceptable quality product.

7.2 Materials and methods

7.2.1 Preparation of feed for extrusion

Corn flour (composition: 1.7% lipids, 76.7% starch and 10% protein) (Brar Natural Flour Mills, Winnipeg, MB) was purchased locally. Soy protein isolate containing over 90% protein was obtained from American Health and Nutrition (Ann Arbor, MI). The moisture contents of all the flours were measured before mixing. The ingredients were first mixed (866g soy protein isolate and 1134g corn flour) in a Hobart mixer (Hobart Food Equipment Group Canada, North York, ON) for 20 min to give an approximate protein content of 50% (dry basis) and then a preset level of water (972g) was added to yield a final feed moisture content of 40% (wet basis). Compositions were selected based on previous results indicated in Chapter 5.

7.2.2 Extrusion process

A co-rotating twin screw extruder (DS32-II, Jinan Saixin Food Machinery, Shandong, P. R. China) was used to produce the extrusion products. Based on previous experimental results, the temperature of the third barrel section (metering section) was adjusted to the required 160^oC with screw speed of 100 rpm and die diameter of 5 mm, the middle part was set to 135°C, and the entrance part was set to 110°C. The length to diameter ratio of the extruder barrel was 20:1 with a die length of 27 mm. The extruder was fed automatically through a conical hopper, keeping the flights of the screw fully filled and avoiding accumulation of the material in the hopper. The extrudate was cut into 8 cm length pieces. Samples were then subjected to various frying treatments for different interval of times.

7.2.3 Frying process

The cylindrical extruded products obtained from extruder were cut to 8 cm lengths and subjected to different frying temperature (±1^oC) and time conditions (Table 7.1). A digital frying pot (T-Fal FR4017 Deep Fryer, NJ) was filled with 2 liter canola oil as a frying medium. The fryer was equipped with a metal mesh basket in which the samples were placed. The oil temperature was stabilized at the required temperature and samples were fried in small batches. Samples were drawn in regular time interval and excess oil was drained and blotted using paper towel and samples were put in plastic bag till equilibration to determine the required parameters.

Table 7.1 Frying parameters of the extruded products

Parameter	Experiment range
Frying temperature	145^{o}C, 165^{o}C, 185^{o}C
Frying time	Up to 660 s

7.2.4 Moisture and oil content determination

The moisture content was measured gravimetricaly by the AOAC-984.25 (AOAC 1995) method (see Chapter 6.2.5). The oil content was determined by using the Soxhlet extraction method with diethyl ether (AOCS 1993).

7.2.5 Texture

Texture was measured as the breaking stress (BS) using a 3 point bend test using the TA.XT Plus Texture Analyzer (Texture Technologies Corp., Scarsdale, NY/Stable Micro Systems, Godalming, Surrey, UK.) with a 50 N load cell. The extruded product was placed on two rounded stands (bridge) 30 mm apart. A rounded plunger was made to push the sample at the middle of the bridges at 5mm/min until breakage occurred. BS was determined as the breaking force per unit cross section area (N/mm^2).

7.2.6 Color

Color of test samples were measured using a color meter (Minolta Spectrophotometer, CM-508d, Canadian Office, Toronto, ON) (Chapter 3, 4, 5). Eight measurements on each sample were taken and the average value was used.

7.2.7 Sensory evaluation

Quantitative descriptive analysis (QDA) approach has gained acceptance for sensory evaluation of various food (Stone and Sidel, 1998). The principle of QDA is based on the ability to train panelists to measure specific attributes of a product in a reproducible manner to yield a comprehensive quantitative product description amenable to statistical analyses (Chapman *et al.*, 2001). In a QDA approach, panelists recruited from the general public work together in a focus group to identify key product attributes and appropriate intensity scales specific to a product.

In this work, 12 trained panellists (seven male and five female with age between 20 and 35 years) were selected to participate in the study. The panel was trained during 6 sessions for QDA of quality fried soy protein-starch based extrudate. The response variables were obtained by means of QDA on a 10 cm non-structured linear scale (Meilgaard *et al.*, 1991), and Quality Rating Test (QRT) (Muñoz *et al.*, 1992) on a 7-point numerical scale (1 = very bad, 2=bad, 3=below fair-above bad, 4=fair, 5=below good-above fair, 6=good, 7 = very good). The following QDA descriptors and respective related words were selected by panel consensus during the training period: color intensity (light gold and dark gold), hardness (soft and hard), crispiness (less crispy and crispy), oiliness (less oily and high oily). The measured characteristics of QRT were: color, favour, texture and overall quality. Samples were randomly evaluated in 9 sessions (2 samples per session). For sensory analysis, the frying temperature of 185°C was selected and samples were withdrawn at 30, 90, 150, 210, 270 s. Samples at each frying time were prepared in duplicate and total 10 samples are prepared. Samples were randomly evaluated in 10 sessions (5 samples per session). Each panellist was provided with plain tea to cleanse the palate between tasting.

7.3 Result and discussion

7.3.1 Moisture loss in fried sample

The moisture loss in soy protein isolate-corn flour mix extruded products fried at different temperatures for different time intervals was evaluated. During frying process there is a simultaneous heat and mass transfer process which allows the evaporation of moisture from the extrudated products. Different extrusion conditions have an effect on density, porosity and expansion of extruded products (Thymi *et al.*, 2005). These properties play a significant role in mass and heat transfer processes during frying of extruded products. Particularly porosity of extruded products enhance the mass and heat transfer process for fast frying and moisture reduction which is explained in terms of exponential decay of moisture reduction.

Furthermore temperature and residence time in the oil enhance the rate of moisture removal. Figure 7.1 shows an exponential decay in moisture content of the fried product with time at different temperature levels.

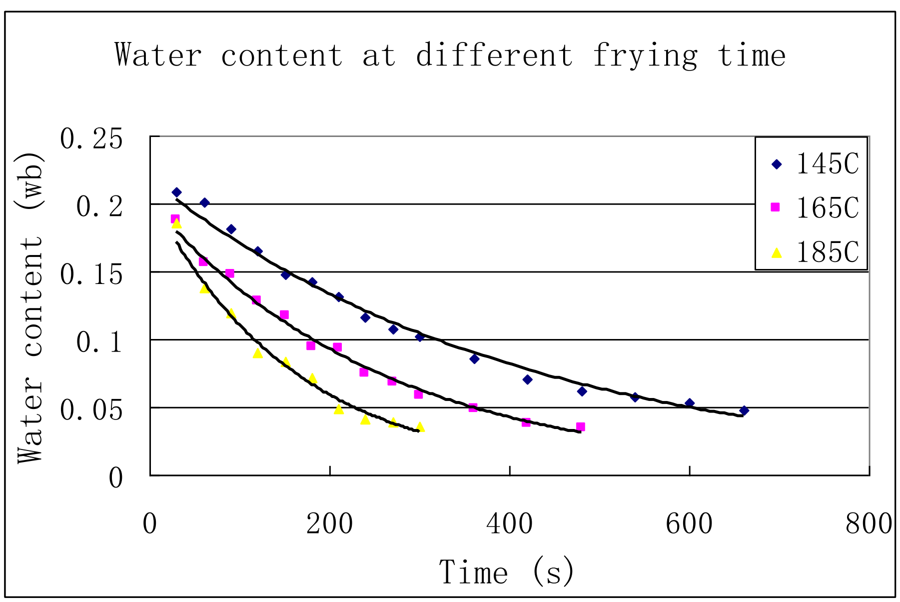


Figure 7.1 Moisture loss of fried soya protein isolate corn flour mix extruded products after treated at 145 °C, 165 °C and 185°C as a function of time

Table 7.2 Moisture content exponential equations with regression coefficients for fried soya protein isolate corn flour mix extruded products

Temperature	Equation	Regression coefficients
145°C	$y = 0.2183e^{-0.0025x}$	$R^2 = 0.9879$
165°C	$y = 0.2019e^{-0.0039x}$	$R^2 = 0.9904$
185°C	$y = 0.207e^{-0.0063x}$	$R^2 = 0.9773$

The moisture content of the samples decreased during frying with frying time, as reported in other works (Garayo and Moreira, 2002; Baik and Mittal, 2002; Moreira *et al.*, 1995). Higher frying temperature resulted in faster moisture reduction. The frying time needed to achieve moisture content below 5% (wb) were 210, 400 and 600s for 185, 165 and 145°C, respectively. In this case frying may be seen as a high temperature drying process in an oil heating medium (Costa and Oliveira 1999) and this allows production of shelf stable protein enriched extruded products.

Compared with low temperature frying process, rapid moisture loss was found at high temperature frying process. Exponential decay in moisture content at different temperature with frying time can be explained by given exponential equations (Table 7.2) with higher values of regression coefficients ($R^2 \geq 0.98$). The result showed that for every 20°C increase in frying temperature the frying time was decreased by 200 s on average. Therefore processing time can thus be shortened considerably by increasing the temperature,

However, with increasing temperature, there might be a limitation on sensory property of the product. Therefore temperature increase during frying time should also consider the development of desired sensory quality property apart reduction in moisture content.

7.3.2 Oil absorption by extruded products

Fried foods are considered as concentrated sources of energy and fat. Oil absorption during deep drying process is an inevitable occurrence and the rate of absorption is related to the type of product, frying oil temperature and contact time of the product with the heating medium. Likewise other fried products the amount of oil absorbed by the extrudate was increased with frying temperature and contact time (Figure 7.2). The oil gain for samples at 185°C was higher as compared to lower temperatures at the same frying time. This indicated that likewise moisture lose, oil gain also associated with an increase in temperature. This might be associated with the rate of lose of moisture from the sample and simultaneous gain of oil in to the product. Faster and more moisture lose at higher temperature, meanwhile, faster and more oil gain accompanied. That is why the oil gain after 300 s frying at 185°C reached close to 22% but it took around 400s at 165°C to gain the same percentage of oil (Figure 7.2). This shows high degree of association between loss of moisture and gain of oil, and hence the gain of oil can be controlled by optimizing the rate of moisture loss from the product in order to get a product with lower oil content. Moreira *et al.*, (1999) also indicated that oil absorption is not only dependent on amount of moisture lost but also how the moisture is lost and products with large quantities of small moisture loss sites will absorb less oil.

It also has been found from Figure 7.2 that at the end of the frying (moisture content less then 5%), lower frying temperature (145°C) products get a slightly higher oil content (more then 22%) then the higher frying temperature products, which even have lower moisture content in the products (see Figure 7.1). This result may be caused by the rapid evaporation of moisture and fast oil absorption stopped the oil enter the inner part of the products from the surface, some of other studies also have the similar results. Moyano and Pedreschi, (2006); Rojas *et al.*, (2006) indicated that low frying temperature such as 120ºC resulted in longer frying time and higher oil uptake for the same residual moisture content. Blumenthal and Stier (1991) mentioned this phenomenon that during frying at a very low oil temperature, such as 120ºC, crusts exhibit a low level of firmness that could let the oil penetrate easily into the product.

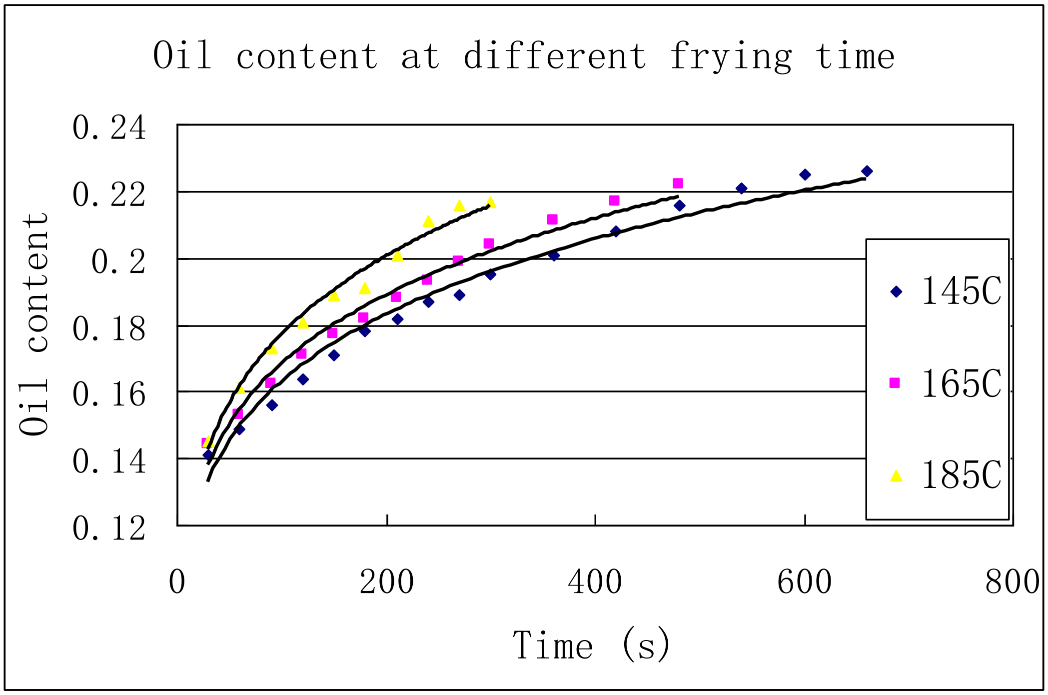


Figure 7.2 Oil content of fried soy protein isolate corn flour mix extruded products after treated at 145°C, 165°C and 185°C as a function of time

Table 7.3 Oil content exponential equations with regression coefficients of fried soy protein isolate corn flour mix extruded products

Temperature	**Equation**	**Regression coefficients**
145	$y = 0.0752x^{0.1678}$	$R^2 = 0.9783$
165	$y = 0.0785x^{0.1656}$	$R^2 = 0.9799$
185	$y = 0.0776x^{0.1792}$	$R^2 = 0.9861$

The modification of microstructure of soy protein isolate-corn flour mix extruded products during extrusion process also pays a significant role in terms of oil diffusion in the product (Mallikarjuna *et al.*, 1997), the larger pore space and lower bulk density could promote the absorption of more oil. As indicated in Figure 7.2, oil absorption is a non linear phenomenon and showed an exponential pattern of oil gain which could be explained by the exponential equations with high degree of regression coefficients (Table 7.2). Particularly for lower temperatures, the oil gain increased with an increase frying period. However with high temperature the rate of oil gain was very fast and reached the maximum about 22% oil gain. Exposing the extrudate for 300 s at 145°C provided a product having a moisture content of 10% with oil content approximately close to 20% (Figure 7.1 and 7.2). Further increase both in medium temperature and exposure time results an increase in oil content more than 20% which may not be advised from health point of view. The development of products that are more acceptable to consumers who are increasingly conscious and concerned about their health has led to the need to reduce oil incorporation during the frying process (Mallikarjuna *et al.*, 1997). Reduction in up take of oil during frying process can be reduced through the use of edible coating materials. Recent studies have experimented with the use of edible coatings (including soy protein isolate) and films as barriers to prevent more oil gain during the frying process (Albert and Mittal, 2002). Furthermore studied conducted on evaluation of the performance of different edible coatings (Freits *et al.*, 2009) showed that whey protein and soy protein isolated films gave the best results with respect to the efficiency of the edible coatings in decreasing fat absorption during the frying process of cassava purée. However where daily calorie intake below the recommended level like in developing nation such type of product which is rich in protein, carbohydrate and oil could be a potential candidate to tackle malnutrition and daily energy demand requirements.

7.3.3 Texture

Texture of fried extrudate was measured using a Texture Analyzer. Hardness was measured in terms of breaking stress, which is the force required to break the fried product. The breaking stress of the fried extrudate (frying temperature 145-185°C for different frying time) is presented in Figure 7.3. It was observed that the breaking stress, which reflects the hardness of the fried product, increased at a slower rate for 145°C and a rapid increase was observed up to approximately 300 s (1.05 N/mm^2) and then showed a decrease in BS with an increase of frying time. However in relatively high temperatures

165 and 185°C, the highest BS was measured at 180 s and 120s respectively, the higher one is measured at 165°C (1.02 N/mm^2) than at 185°C (0.99 N/mm^2). Likewise the first case the BS in these temperatures decreased with frying time. The result confirmed that highest BS was achieved in lower temperatures with extended frying time as compared to relatively high temperature. Starch and other ingredients generally play an important role in defining textural quality of different kinds of products (Edwards *et al.*, 1995; Hatcher *et al.*, 2002). The pasting and swelling qualities of starch combined with the binding qualities of protein from soy protein isolate may have resulted in increased network formation and contributed to the observed effect on higher BS of fried product at lower temperatures as compared to highest one.

During the frying process of protein enriched starch extruded products because of moisture evaporation, and temperature effect on the polymers, certain structural modifications can be induced. The peak value in BS level might make the product crunchier which is the desired characteristic of the product. Increase in strength for the first few minutes might be due to the moisture remaining in the product which could play a role in the development of strong starch and protein association and inter linkage. The resulting protein-starch network is important to the overall texture of the product. However with increase duration of frying leads to an increase in the moisture loss and break the temporary associations and linkages developed in the first few minutes. This result in produce crunchy and crispy protein enriched fried product. In different work, tortilla chips became harder within 30 s of frying, and then crunchy until the end of frying as moisture decreased (Kawas and Moreira, 2001) and its crispiness increase as frying time increase (Moreira *et al.* 1995).With extended frying period on simultaneous reduction and oil gain the textural properties of the product changed. Increase in oil content after sometime contributes decrease in strength of the product. Generally it can be hypothesized that the interaction and association of protein-starch complex increased with frying time up to sometime which leads to an increase in BS. However after the maximum BS time further frying might result in denaturation of protein and modification of starch ultimately breaks the developed protein-starch linkage which would give a crunchy crispy product with reduced strength. Therefore in terms of desirable textural properties of the product the frying process either stopped before the maximum BS time or should continue for sometime after the maximum BS. This recommendation should be supported by associated sensorial test to determine the best textural property of the product.

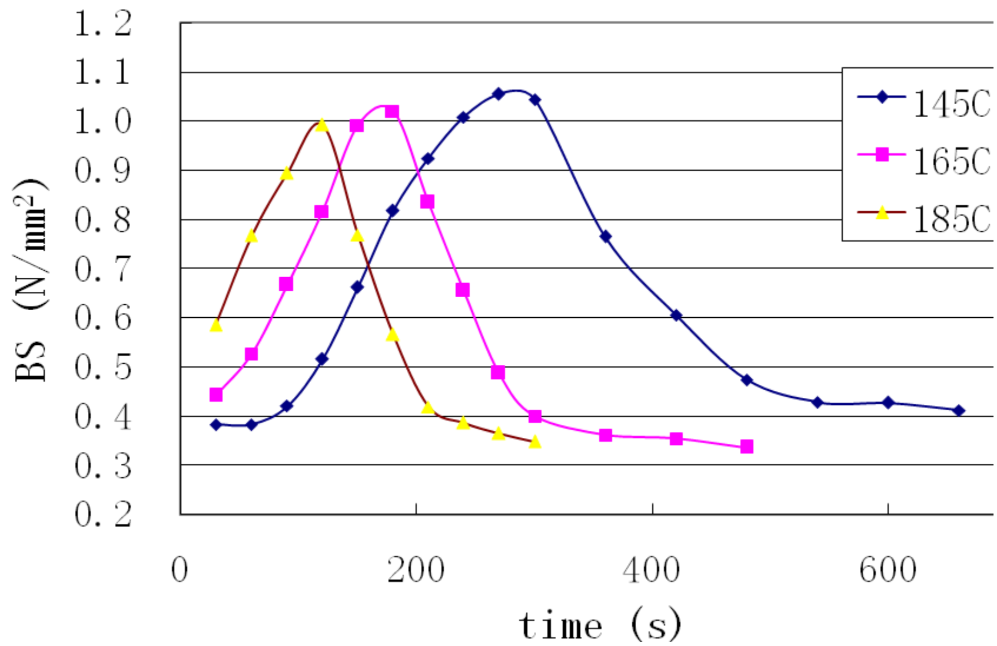


Figure 7.3 Breaking stress of extruded products as affected by frying temperature and time

7.3.4 Color

The appearance (color) of a food is one of the most important factors defining consumer acceptance. Color is the result of complex interactions of incident light with the food matrix affect transparency, translucency, gloss, lightness (Caivano 1991) and chromatic perceptions (Briones *et al.* 2006).

The effect of frying time and temperature on color of soy protein isolate-corn flour mix were investigated. The quality of fried products mainly determined by the type of color developed after frying. Undesirable color and off flavor development due to excessive frying is the undesirable properties of fried products both in terms of economic and health point of view. Non enzymatic reaction known as Maillard reaction is the result of reaction of reducing sugar and amino acid at higher temperature. This reaction is mainly responsible for the development of color of foods during frying process. Optimum time-temperature combination during frying contributes for development of good color. In addition to time of frying and level of temperature applied, color development during frying can be influenced by concentration of amino acid and reducing sugars present in the product. The more these components the more color development, but an excessive frying time and temperature results in development of undesirable color and toxic compounds.

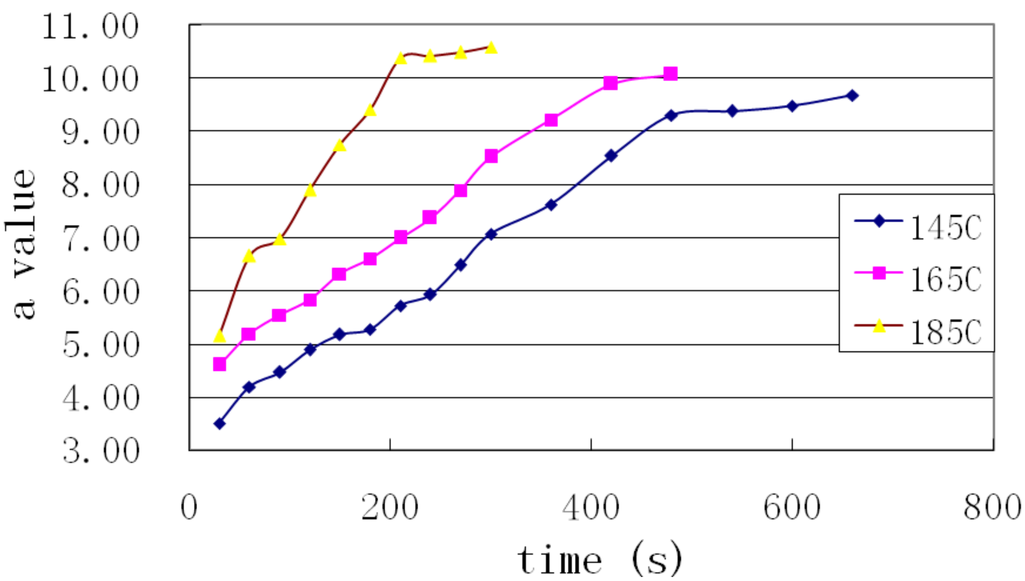


Figure 7.4 Hunter color a values of extruded product subjected for different frying time and temperatures

Figure 7.4 shows that development of red color during deep fat frying of extrudate was markedly affected by frying time and temperature. In this work the color value of fried product is expressed in terms of redness (a) value. Results from color evaluation showed that there was a variation in the redness values among the fried samples as shown by a linear increase in a value with temperature and time to a certain level and remained constant with an increase in frying time (Figure 7.4). A rapid increase in a value was observed for 185°C and reached to maximum around 200 s as compared to 300 and 500 s at 165 and 145°C respectively. After these time ranges the values remain consistent with an increase in frying time and hence the frying processes could be stopped.

a b

Figure 7.5 Extruded products before (a) and after frying at 185°C for 180 s (b)

Golden yellow or light brown color of fried products is one of the most desirable physical characteristic which is selected by consumers. Figure 7.5 showed that the differences in color between fried and not fried samples (Figure 7.5-a). The decrease of lightness and yellowness and the increase of redness during the frying process could be attributed to yellow gold color pigment formation (Figure 7.5-b). However this color was obtained when the sample had the highest BS with oil and moisture content close to 20% and 7% respectively. Generally complex set of reactions between amines, usually from proteins, and carbonyl compounds, generally sugars contributes for the development off such type of color.

7.3.5 Sensory quality

Instrumental and corresponding sensorial results of fried samples are indicted in Table 7.4. The basic strength of descriptive statistics analysis is its ability to allow relationships between descriptive sensory and instrumental measurements to be determined. Comparing the sensory evaluation results, with an increase in frying time, the experimental results like a value, BS and oil content had a similar trend with the color, crispness and oiliness QDA evaluation results and increasing values for color and flavor quality in QRT (Table 7.4). These trends shows that extended frying time would allow more oil absorption and Maillard reaction for better color and flavor development which are more like by assessors. However the BS of instrumental measurement was observed at 90 seconds frying time the highest hardness (QDA) and textural quality (QRT) were observed at 150 seconds of frying (Table 7.4). The overall acceptance of the quality was increasing with frying time 210 seconds. Therefore based upon matching results from instrumental and sensory tests fried extruded products at 150 seconds could have the best preferred one by consumers.

Table 7.5 shows the degree of correlation between instrumental measured values with sensorial evaluation results. Instrumental color value showed that high degree of positive correlation with color, flavor and overall acceptance quality (QRT). Hence the instrumentally measured color value of the fried product can be used as a quality monitoring criterion during frying process of soy protein enriched-corn flour mix fried extruded products. Furthermore oil content of fried samples also had a strong positive correlation with overall quality of the samples. This confirms that like other fried products oil content plays a significant role in modification of texture, and flavor of fried samples in favor of their acceptability by consumers. BS showed a strong positive correlation with hardness (QDA), and a weak negative correlation with crispness and strong negative correlation with texture and overall quality. Moisture content had also a negative correlation with crispiness and overall quality of fried samples. Negative correlation between variables showed the existence of an inverse relationship between variables. For instance a more moisture content indicates the product is less crispy and has less acceptability. Therefore color value and oil content of the fried samples were the two instrumental measurements which showed a strong positive correlation with sensorial taste values (QDA and QRT) and can be used as a quality controlling variables in production of such type of product.

Table 7.4 Response values of the Sensory and Instrumental tests for fried sample at different frying time (185°C).

Sample at frying time (s)	Instrumental responses				Sensory responses							
					Quantitative Descriptive Analysis				Quality Rating Test (QRT)			
	Water Content (%)	Oil Content (%)	a value	BS (N/m m^2)	Color	Hardness	Crispness	Oiliness	Color quality	Flavor quality	Texture quality	Overall quality
30	18.6±2.03	14.5±3.56	5.15±0.86	0.59±0.028	1.47±0.45	4.81±0.57	2.45±0.21	4.12±0.73	5.28±0.73	3.01±1.35	3.21±0.65	3.68±0.43
90	12±1.32	17.3±3.28	6.97±0.73	0.89±0.13	3.02±0.62	6.67±0.41	2.76±0.48	4.76±0.95	5.48±0.75	4.37±1.13	4.32±0.48	4.52±0.56
150	8.4±0.53	18.9±2.74	8.75±1.04	0.77±0.22	4.66±0.89	7.67±0.82	4.11±0.72	5.25±0.85	5.89±0.87	5.87±1.28	5.89±0.70	5.67±0.53
210	4.9±0.45	20.1±2.17	10.38±1.32	0.42±0.057	5.86±0.84	5.67±0.98	6.48±0.83	7.47±0.97	6.34±0.52	6.26±1.67	5.67±0.80	5.84±0.72
270	3.9±0.21	21.6±1.89	10.49±1.27	0.36±0.028	8.63±1.23	4.37±1.35	7.38±0.61	7.67±1.25	6.43±0.68	6.52±1.09	5.28±0.55	5.36±0.80

Table 7.5 Correlation between instrumental and sensory responses

Instrumental response	Variable	Sensory response	Correlation
Color	a	color	0.932
	a	color quality	0.984
	a	Flavor quality	0.985
	a	overall quality	0.923
Texture	BS	Hardness	0.778
	BS	Crispness	-0.805
	BS	Texture quality	-0.212
	BS	overall quality	-0.308
Oil content	Oil content	Oiliness	0.919
	Oil content	overall quality	0.872
Moisture content	Moisture content	Crispness	-0.907
	Moisture content	overall quality	-0.920

7.4 Conclusions

Temperature and time of frying are the most important factors studied in this work. During frying work the moisture content of the product reduced below 5% after 210, 360 and 600 s of frying at temperatures 145, 165 and 185°C, respectively. The frying resulted in reduced water activity level and increase shelf stability of the product. However with a decrease in moisture content the oil content increased and reached more than 20% at all frying temperatures which might be undesirable from economic and health point of view. However, it has a significant effect on texture, color, flavor and overall acceptability of fried samples.

Maximum BS was observed close to 120 s of frying at 185°C which might be associated with protein-starch inter linkage during earlier time of frying. The pasting and swelling properties of starch and binding properties of protein with the presence of moisture might also be result for an increase in strength. With an extended period of frying the moisture content decrease and the developed strength might be reduced and contribute for the development of desired texture. Therefore crunchiness and crispiness of the fried product developed after the BS reached the maximum level with extended drying time.

Color is also one of physical properties which determined the acceptability of the product. With an increase of frying time and temperature a value increased. This is mainly because of reaction between amino acid groups of protein and sugar groups of starch with the influence of high temperature.

Sensory evaluation of fried extruded product is the ultimate test to determine the acceptability of the product. There were high degrees of correlation between instrumentally measured quality parameters of fried extruded products with those from sensory tests.

CHAPTER 8

GENERAL CONCLUSIONS, CONTRIBUTION TO THE KNOWLEDGE AND RECOMMENDATIONS

GENERAL CONCLUSIONS

1. The effect of twin screw extrusion process variables on the physical properties of SPI and corn flour blends were studied. Three important extrusion parameters (screw speed, feed moisture and barrel temperature) affected the physical properties of extruded products. Feed moisture content was the most important factor for the physical properties of the extrudate in the experimental range.
2. The influence of screw speed, die diameter and raw material moisture content to residence time distribution (RTD) of the SPI and corn flour mixture extrusion were studied. Mean residence time decreased with increase in the screw speed, moisture content and die diameter. RTD was wider when the screw speed or the moisture content was lower or the die diameter was smaller. All the parameters were significantly ($p<0.05$) affected the Total Extrudate Collection Time (ECT), mean residence time t_m and variance and only the material moisture content was significantly affected the fastest particle residence time. The cumulative accumulation (F) and transient concentration (E) models were shown to well fit the experimental data.
3. In the subsequent extrusion study with protein rich formulations of SPI – corn flour, the protein content, feed moisture and processing temperatures significantly influenced the physical properties (BS, BD, ER, WSI, RR and color) of the extruded products. BS, BD increased with increasing of protein content, but at higher protein content in blend decreased ER, WSI, RR and L value. Higher feed moisture content of blend played a very important role to increase BS, BD and L value and decrease WSI and RR. ER increased with increasing feed moisture content at a certain level and then decreased with further increases in process variables. Higher extrusion processing temperature showed a dominant effect to increase BS, ER, RR and L value and a decrease BD and WSI. The optimum processing conditions identified are expected to be very useful for developing protein rich extruded products.

4. Extrusion processing variables significantly influenced the subsequent drying process. Increased protein content in the products decreased the drying time when the final end point was based on achieving a water activity of 0.75. Increasing barrel temperature and decreasing the raw material moisture content reduced the drying time. Increasing of protein content in the product resulted in higher moisture content in the final product at a_w of 0.75. Moisture content decreased with the increasing drying time, and the product with lower protein content dried faster. The product with highest protein content (66.7%) showed a rapid decrease in a_w and after a drying time of 75 min, it gave a much lower a_w than the others (33.2% and 50% protein content) even after at 120 min drying time. The moisture sorption isotherms generally merged at high content levels but started to separate when the product moisture decreased below 20%. Higher protein content products gave lower a_w at the same moisture content and in the same a_w level. Models were developed for MR was found to well describe MR values at different drying times.
5. Fried snacks were prepared from the extruded products containing 50% protein. The moisture content in the fried product reduced below 5% after 210, 360 and 600 s of frying time at 145, 165 and 185°C, respectively. However, the product's final oil content reached a little bit more than 20%. Maximum BS was observed when the product moisture content was around 10%. With an increase of frying time and temperature, the color a-value increased. Sensory evaluation results well correlated objective of color measurements and oil content. Frying conditions which gave acceptable quality products were identified.

CONTRIBUTIONS TO KNOWLEDGE

1. Previous literature results reported that incorporating SPI to corn flour could significantly increase the nutritive value and quality characteristics of the extruded end product. However, the study directly related process parameters and physical properties of the extrudate with its high SPI content has not been studied in detail. This study systematically studied the effect of different extrusion processing variables

to the physical properties of high protein content products (protein content up to 66.7% in the products).

2. A potential high protein source of extruded product with high soy protein content can meet the soy protein intake requirement of 25g per day as suggested by FDA.

3. The detailed residence time distribution (RTD) of SPI and corn flour mixture was evaluated for the first time in a twin screw extruder using a full factorial design of experiments. The cumulative distribution F curve model fitted well the experimental RTD for SPI-corn flour mixture during the extrusion process and E-curve data could be easily generated from the F models.

4. There is limited scientific information about the drying characteristics of extruded products. The present work described the influence of different extrusion variables on the drying behavior and drying time models for achieving a desirable a_w in the final products were developed.

5. Extrusion and frying are combined to create a range of fried snack foods which offered new characters to the protein-rich extruded products The frying process was evaluated for their influence the physical and sensory characters of the products, and the sensory test were used to identify products of acceptable quality.

RECOMMENDATIONS FOR FUTURE RESEARCH

This research has demonstrated several important findings. Meanwhile, it also showed some ideas of interest for future research and development, which could be summarized as follows:

1. Testing different other carbohydrate based products to replace corn flour and keep the high content of soy protein in the extrudates. Use of sensory evaluation to find out the

better solution for the taste. Some work in this area was initiated as a short project employing Tef an ingredient commonly used in Ethiopia and desirable protein rich product were obtained.

2. Use of methods to test the SPI degradation during the extrusion process. Especially interesting and important will be to track the degradation of iso-flavones in the soy based products.

3. Frying was successfully combined with extrusion in the current study. A more detailed study including how to reduce the oil uptake during the frying would be desirable.

4. The formation of resistant starches during the extrusion process has not been studied and this is also important from the nutrition aspects of the extrudates.

5. Different drying methods and drying conditions can be evaluated as alternatives and optimized processes based on time, energy efficiency etc could be identified.

6. Other post extrusion treatment to the extrudate should be considered, including baking, steaming, frozen, etc.

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