# FUNCTIONAL EXTRUDED PRODUCTS FROM RICE FLOUR, PULSE FLOUR AND TOMATO POWDER BLENDS

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Dedicated to my Family and my Supervisor

#### ABSTRACT

Extrusion is a high shear, high temperature processing technique that involves a short time treatment to transform raw materials to intermediate or finished products to enhance sensory and other quality attributes. Most extruded snacks prepared using starch based blends are usually high in carbohydrates but low in other vital nutrients. Snack food consumption is ever increasing and has resulted in demand for producing healthy snack foods by replacing the starch with protein, fiber and other nutrients. The overall objective of this research was to prepare nutritious extruded snack foods by partially replacing rice flour with green lentil and chickpea flours and tomato powder.

Using the D-optimal mixture design different feed mixtures formulations were obtained and prepared using rice flour (33-60%), green lentil flour (20-45%), chickpea flour (10-30%), tomato powder (0-7%) and added moisture. These were subjected to extrusion processing at a preset screw speed of 125 rpm and barrel temperature profile progressively increasing from 80°C at the entrance to 140°C at the exit. The semi-moist extrudates were air dried at 55°C at 0.1 m/s air flow rate to reduce their water activity to < 0.75 for stability. From the drying data, moisture diffusion coefficients and drying times were computed. The physicochemical properties of dried extrudates were evaluated. Lentil and chickpea flours added to the feed mix enhanced the protein content of the product while lentil and tomato powders contributed to improving the antioxidant activity. Added lentil flour also decreased the diffusion coefficient and hence increased the drying time. The extrusion processing led to the reduction of anti-nutrients present in pulse flours by more than 80%.

In the second phase of study, sensorial and physico-chemical properties of the extrudates were enhanced using conventional deep frying or dry roasting using microwave. Deep frying was done at 200±1 °C for 70 s while microwave roasting was carried out in a domestic MW oven (1000 W) carried at 75% power level for 50 s. The frying and roasting processes significantly influenced the product properties. Addition of chickpea flour significantly decreased the expansion ratio while increasing the b\* value of the products. Lentil flour and tomato powder increased breaking stress value. Sensory properties of the products were enhanced with both frying and MW roasting.

Expansion ratio and L\* value had strong positive correlation with overall product acceptability. Microwave roasting resulted in better retention of antioxidants than the frying process.

Overall, the research contributed to better understanding of the extrusion process involving rice flour blends enriched with lentil flour, chickpea flour and tomato powder. A high protein snack with good sensory properties was prepared with help of extrusion followed by drying and frying or microwave roasting. The results obtained demonstrated that microwave roasting in combination with extrusion processing produced more nutritious and healthier snack as compared to frying.

#### RESUME

L'extrusion est un procédé de cisaillement à haute température qui implique un traitement de courte durée dans le but transformer les matières premières en produits intermédiaires ,en produits finis, ou dans le but d'améliorer les propriétés sensorielles et autres qualités des produits finis. La plupart des collations extrudées, préparées à l'aide de mélanges à base d'amidon, sont souvent riches en glucides mais faibles en nutriments essentiels.

L'accroissement de la consommation de ces collations aboutie a une augmentation de la demande pour des produits plus sains, dans lesquels l'amidon serait remplacé par des protéines, des fibres et d'autres nutriments. Le but de cette recherche était de préparer des collations extrudées nutritives en remplaçant partiellement la farine de riz par des farines de lentilles vertes et de de pois chiche et par de la poudre de tomate.

En utilisant le system de conception de mélange D-optimal, différents mélanges d'aliments ont été préparés avec notamment de la farine de riz (33-60%), de la farine de lentilles vertes (20-45%), de la farine de pois chiche (10-30%), de la poudre de tomate (0-7%) et de l'humidité ajoutée. Ces mélanges ont été soumis à un traitement par extrusion à une vitesse de rotation de la vis de 125 tr/min et à un profil de température du fût augmentant progressivement de 80 ° C à l'entrée à 140 ° C à la sortie. Les extrudés semi-humides ont été séchés à l'air à 55 ° C avec un débit d'air de 0,1 m / s pour réduire leur activité d'eau à 0,75 afin de les stabiliser. À partir des données de séchage, les coefficients de diffusion de l'humidité et les temps de séchage ont été calculés. Les propriétés physico-chimiques des extrudés séchés ont été évaluées. Les farines de lentilles et de pois chiches ajoutées au mélange ont augmenté la teneur en protéines du produit tandis que les poudres de lentilles et de tomates ont amélioré l'activité antioxydante de celui-ci. L'ajout de la farine de lentilles a également diminué le coefficient de diffusion et, par conséquent, a augmenté le temps de séchage. Le traitement par extrusion a conduit à la diminution des antinutriments présents dans les farines de plus de 80%.

Dans une seconde phase de la recherche, les propriétés sensorielles et physico-chimiques des extrudats ont été améliorées en utilisant la friture traditionnelle ou la torréfaction sèche au micro-ondes. La friture a été effectuée à une temperature de  $200 \pm 1$  ° C pendant 70 s, tandis que la cuisson au micro-ondes a été effectuée dans un four a micro-ondes domestique (puissance

nominale W 1000) portée à un niveau de puissance de 75% pendant 50 s. Les processus de friture et de torréfaction ont influencé de façon significative les propriétés du produit. L'ajout de farine de pois chiche a considérablement diminué le taux d'expansion tout en augmentant la valeur b \* des produits. La farine de lentilles et la poudre de tomate ont augmenté la valeur de résistance à la rupture. Les propriétés sensorielles des produits ont été améliorées avec la friture et la torréfaction au micro-ondes. Le rapport d'expansion et la valeur L \* présentaient une corrélation positive forte avec l'acceptabilité globale du produit. La cuisson au four à micro-ondes a conduit à une meilleure rétention des antioxydants que le processus de friture.

Dans son ensemble, la recherche a contribué à une meilleure compréhension du procédé d'extrusion impliquant des mélanges de farine de riz enrichis avec de la farine de lentilles, de la farine de pois chiche et de la poudre de tomate. Une collation riche en protéines et ayant de bonnes propriétés sensorielles a été préparée grâce a une extrusion suivie d'un séchage et d'une friture ou bien d'une cuisson au four à micro-ondes. Les résultats obtenus durant cette étude ont démontré que le traitement par extrusion utilisé avec la cuisson au four à micro-onde produisait un produit plus nutritif et plus sain qu'un traitement alliant le procédé d'extrusion a la friture.

#### **CONTRIBUTIONS OF AUTHORS**

The thesis research has been presented at several seminars and symposiums. This thesis has been written in manuscript style to suitably edit chapters highlighting research for publication. Authors involved in the thesis work and their contributions to the various articles is as follows:

**Prabhjot Singh** is the MSc. candidate who planned and conducted all the experiments, on the advice and guidance of his supervisor, gathered and analyzed the results and prepared drafts for the thesis and the manuscripts for scientific presentations and publications.

**Dr. Hosahalli S. Ramaswamy** is the thesis supervisor, under whose guidance the research was carried out, and who coordinated and supervised the candidate in planning and conducting the research, as well as in correcting, reviewing and editing of the thesis and the manuscript drafts for publication.

# LIST OF PUBLICATIONS AND PRESENTATIONS

Parts of this thesis have been prepared as manuscripts for publications in referred scientific journals:

Singh, P. and Ramaswamy, H.S., 2017. Drying characteristics and physico-chemical properties of extruded products from mixtures of rice, green lentil and chickpea flours, and tomato powder. (draft prepared)

Singh, P. and Ramaswamy, H.S., 2017. Effect of frying and microwave roasting on physicochemical properties of dried extrudates prepared from rice based blends. (draft prepared)

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**Singh, P.** and Ramaswamy, H.S. 2016. Development of ready to eat snack with rice based blends using extrusion processing and evaluating effect of frying and microwave roasting on extrudates. Food Processing Research Mini Conference, Macdonald Campus, Ste Anne de Bellevue, Montreal, Canada, October 31, 2016.

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# NOMENCLATURE

AA	Antioxidant Activity
ANOVA	Analysis of variance
aw	Water activity
BD	Bulk density
BS	Breaking stress
D	<b>Diffusion Coefficient</b>
db	Dry basis
Df	Degree of freedom
ER	Expansion ratio
М	Moisture content
MR	Moisture ratio
MW	Microwave
M.S.E	Mean Standard Error
NS	Not significant
PC	Protein Content
QRT	Quality rating test
r	Pearson's correlation coefficient
<b>R</b> <sup>2</sup>	Coefficient of determination
RR	<b>Rehydration ratio</b>
RSM	<b>Response surface methodology</b>
TC	Tannin Content
SOS	Sum of square
wb	Wet basis

#### **CHAPTER 1**

#### **INTRODUCTION**

The first application of extruder in food processing dates back to 1930's when the "forming extruder", a basic design of extruder, was used to manufacture pasta products. Since then, with the evolution of many different types of extruders, extrusion technology has been used in the food industry for developing various products including precooked and modified starches, ready to eat (RTE) cereals, snack foods, texturized vegetable proteins (TVP), breading substitutes, etc. (Harper and Clark, 1979). Improvement in these basic products have continued since then to the present day to meet the needs of people. Extrusion technology involves continuous processing of food and includes one or more unit operations such as mixing, kneading and shaping a dough.

For the development of the extruded snack, cereal grains act as the major raw materials as they provide good expansion characteristics because of high starch content. The major role of cereal starches is that they provide better mouth feel, texture, porosity as well as bulk to such snacks improving their desirability (Launay and Lisch, 1983) and they have excellent water absorption properties as they gelatinize. Hence, extrusion processing of rice, wheat and corn has been widely commercialized.

Rice is also among the highest cultivated crops of the world estimated to account for almost 20% of the total world cereal production (FAO, 2016). During the process of rice milling, broken rice is a major by-product and such broken rice can be easily converted to flour. The nutritional composition of broken rice is very much similar to whole rice but it is economically very cheap. According to Zhou et al. (2002) the composition of rice is approximately 7.3% protein, 2.2% fat, 64.3% carbohydrate, 0.8% fiber and 1.4% ash. In a formulated ready to eat snack the most important property of the rice flour is due to its bland taste, attractive white color, hypo-allergenicity and ease of digestion (Kadan et al., 2003).

However, snacks formulated from cereals are very low in other nutrients such as protein and other functional components (Petrova et al., 2015). With changing time, the market consumption trend is also changing fast with a demand for better and healthier snacks. For increasing the nutritional quality of a snack, new and novel ingredients are now being included in the product formulations. The versatility of production, with the help of extrusion processing, has created a new market segment and strong growth in the snack food market (Gill, 2014).

As compared to other raw materials for extrusion processing, incorporating pulse flour was reported to have a positive effect on protein and total dietary fiber of snack along with good expansion of the product (Berrios, 2006). Pulses are rich source of proteins and are economically manageable. The 68<sup>th</sup> United Nations Assembly has declared the year 2016 as the International Year of Pulses (IYP) with the aim of increasing awareness and utilization of pulse crops. Pulses, such as lentils, are considered best sources of plant protein. Nutritionally, lentils are not only rich in proteins but are also rich in vitamins such as riboflavin and folate and minerals like iron, zinc, magnesium and phosphate. Likewise, chickpea flour is also a good source of protein and because of its characteristic yellow color and desirable flavor it is extensively used in the preparation of savory snacks (Geervani, 1989). Even though protein content of chickpea is lower than that of lentils, they are a good source of essential polyunsaturated fatty acids and have fat content higher than other cold season legumes. The utilization of lentils in snacks is comparatively recent whereas chickpea flour finds its incorporation in many traditional snacks such as *murukku*, savory snacks, chapatti, falafel as well as popular spreads such as hummus (Gaur et al., 2015). Additionally, chickpea has a characteristic yellow color which has been found to have a positive impact on the acceptability for extruded products (Badrie and Mellowes, 1991). Furthermore, deficiency of pulse flours in methionine can be balanced with the utilization of rice (Yepiz et al., 1983). Lentils and chickpeas have very low glycemic index which help in controlling the blood sugar ultimately proving to be beneficial for controlling the diabetes (Rizkalla et al., 2002), in the prevention of tumor causing cancer (Mathers, 2002), and to prevent or decrease the risk of heart related problems (Anderson and Major, 2002).

Antioxidants in diet play a very important role in enhancing human health by preventing damage to cellular components because of the reactions that involve free radicals (Young and Woodside, 2001). A novel snack type functional food prepared from lentils can also help to enhance the health of the consumer because of the availability of bioactive compounds having antioxidant properties (Petrova et al., 2015). Similarly, dried tomato powder is a very important source of lycopene which has a high antioxidant activity (Schieber et al., 2001). The health effects of lycopene on reducing prostate and pancreatic cancer as well as preventing cardiovascular

diseases has already been established (Johnson, 2000). The antioxidant activity of fruits and vegetables is mainly because of the presence of phenolic compounds (Prior et al., 1998). Bravo (1998) reported that the phenolic content of legumes is much higher than that of fruits and vegetables. Some of the phenolic components also have anti-nutritional effect. These components include trypsin inhibitors, phytic acid and tannins. Vidal-Valverde et al. (1994) have stated that cooking and germination can bring the destruction of trypsin inhibitor activity and phytic acid. The anti-nutritional components have limited the utilization of pulses. Extrusion cooking allows to incorporate different novel ingredients to develop new snacks with better nutrition and increased acceptance by the consumer (Brennan et al., 2013). The acceptance of extruded snack is mainly due to convenience, value, better appearance and rich texture (Anton and Luciano, 2007). Ratio of mixtures in the blend, raw material and processing conditions are the three degrees of freedom to control in the final product (Yu, 2011; Muteki et al., 2007). The different phenomena taking place during extrusion processing makes mathematical modelling very difficult (Grenus et al., 1993). Studying the effects of feed formulation on the final product properties is very important in the development of a new snack (Forsido and Ramaswamy, 2011). The high-temperature short time (HTST) extrusion cooking can be used to bring beneficial changes and improve bioavailability of nutrients and destruction of anti-nutritional factors (Harper, 1981). Several extrusion studies have used pulse flours such as rice and chickpea flours (Bhattacharya and Prakash, 1994), rice and green gram flours (Bhattacharya, 1997), corn and lentil flours (Lazou and Krokida, 2010), corn starch and bean flours (navy and red bean) (Anton et al., 2009), lentil and wheat bran flours (Morales et al., 2015).

Hence, the main focus of this research was the development of enhanced nutritive value ready to eat rice-based snack food by incorporation of lentil and chickpea flours, and tomato powder.

The specific objectives of the research were:

- 1. To study the effect of twin screw extrusion processing of rice based formulation blends (rice and chickpeas flours and tomato powder) on physical properties (expansion ratio, bulk density, rehydration ratio, breaking stress and color) as well as on chemical constituents (antioxidants, anti-nutrients).
- 2. To study the effect of formulation blends on post extrusion drying time and water activity.

3. To evaluate the sensorial and physico-chemical properties of extrudates subjected to frying or microwave roasting.

#### **CHAPTER 2**

#### LITERATURE REVIEW

#### 2.1 Introduction

According to Riaz (2000) "Food extrusion is a process in which a food material is forced to flow under the pressure which is generated with the help of a piston or screw, under one or different combination of conditions of mixing, heating, and shear, through a die which is a small opening at the end of the barrel used for shaping/puffing the ingredients". Extrusion processing was initially introduced in the plastic industry and with changing time and new improvements its use in agrofood industry was identified. In present times extrusion products are finding its place on market shelves as convenience and functional foods (Moscicki and van Zuilichem, 2011). Extrusion processing employs screws to convey food forward which help in blending wide range of novel food materials together leading to easier development of functional foods. The lifestyle of people is changing now. Due to fast life and huge competition, people now have limited time during their working hours. To prevent hunger, people have turned more towards quick meals to save time. Children are especially attracted to convenience foods because of its rich taste, sensorial as well as visual characteristics.

Extrusion cooking combines the heat and the process of extrusion to produce a semi-solid product. The process of extrusion starts with feeding the ingredients into the hopper. The rotating screw conveys the material forward. As the material is conveyed forward, volume of barrel reduces along the path resulting in shearing of food and development of heat due to friction between the ingredient and screw. Additionally, barrel is provided with external heating aids for increasing temperature above 100° C. As the ingredients reach the end of barrel, it is forced to pass through a small opening in the metal plate called die which leads to buildup of pressure. The point at which the pressure is high enough to force the material through die opening, the material comes out. As the material comes out, pressure changes suddenly for the product combined with evaporation of water leaving air cells behinds. The sudden pressure change as well as moisture evaporation results in expansion of food. However, the rate of expansion is different for different food structures and is governed by the properties of the molten or plasticized material, inputs during extrusion processing as well as that of the geometry of the insert of the die (Guy, 2001).

Extrusion process is a combination of different unit operations. Depending on the desirability or food properties it has the ability to deliver intermediate as well as finished product. The effect of extrusion cooking on food components or nutrients is variable and depends upon combinations of extrusion process parameters as well as product variables. High shear accompanied with high temperature not only brings changes in chemical structure of basic components such as carbohydrates, proteins, vitamin and mineral content but also bring significant changes in physical structure. Depending upon the desirability the changes can be beneficial or deleterious also. Extrusion processing can destroy anti-nutritional factors present in food and can bring changes such as gelatinization of starch, increased soluble dietary fiber and reduce lipid oxidation (Singh et al., 2007). Whereas, other chemical changes such as Maillard reaction can reduce protein content of a product also through reaction between amino acids and reducing sugars. Additionally, effect of extrusion on some vitamins can also be destructive. However, extrusion processing offers advantage of processing wide range of raw materials increasing the option of including novel and functional materials to be processed and incorporated in food.

#### 2.1.1 2016 as International Year of Pulses

Pulses are a subgroup of legumes derived from the plants of family Leguminosae. According to FAO, pulses are the dry seeds which are edible and low in fat content. Hence, pulses do not include legumes which are considered vegetables such as green peas or the legumes for oil extraction such as soybean and groundnut. The definition of pulses includes kidney beans or navy beans (Phaseolus vulgaris L.), peas (Pisum sativum L.), lentils (Lens culinaris), chickpeas (Cicer arietinum L.), faba beans (Vicia faba L.), mung beans (Vigna radiata) as well as lupines (Lupinus albus) and bambara beans (Vigna subterranean). The 68<sup>th</sup> United Nations general assembly declared year 2016 to be felt as International Year of Pulses with aim of generating awareness as well as to increase utilization of pulses through improvements in food chain (UnitedNations, 2014). The Food and Agricultural Organization (FAO) was nominated for implementation of the same. Pulses have been regarded as key nutrients for alleviating nutrient deficiency along with sustainable food production. In the resolution (A/RES/68/231) adopted by United Nations pulses were noted as a critical source of plant based proteins and a desired food for achieving food security and nutrition. The impact of legume plants for their role as fixing nitrogen in soil as well as potential of pulses to lead to a healthy diet to address obesity and other chronic diseases such as diabetes and cancer was also recognized.

#### 2.2 Snack

Snack is defined as a food that is taken along with a meal at regular meal time or item that is taken to replace a meal. According to Gatenby (1997) the difference between snacks and meals generally depends on when the food is consumed or the nutritional composition of food. In the study done on "Food based classification of eating episodes" (Lennernäs and Andersson, 1999) eating event was categorized into meals and snacks on the basis of visible properties (food types) and invisible properties (nutrients) along with sub categorizing snacks as prepared and quick prepared. However, no definition of snacks is universally accepted (Drummond et al., 1996) and definition is selected on the basis of review of the study being conducted. During processing of cereal based snacks, cereals are mixed together and provided some amount of moisture. Sometimes flavorings are also added at this time and then this mixture is fed to an extruder to undergo extrusion process. As per the needs the temperature inside extruder can be adjusted to fully cook or partially cook the product. Sometimes flavorings or seasonings are applied after the completion of process or depending upon acceptability more cooking aids can be employed such as baking, roasting, glazing, frying etc.

As reported by (Gill, 2014) snack food market amounted to around \$300 billion in 2010 has been expected to grow in the future also. Among all the snacks, cereal based snacks have occupied a large share of market which are generally dominated by extruded food products (Brennan et al., 2013). These traditionally produced foods include breakfast cereals which were produced by Kellogg's brothers in the early phase of 20<sup>th</sup> century (Fast, 1999). However, with the advent of extrusion technology new products are now available in the market. The focus of producing snacks from whole cereal grains is now shifting to incorporate other raw ingredients of better functional or nutritional composition such as pulses, vegetable or fruit powders or other novel ingredients.

Snacks are very popular all over the world because they need a little or no processing before being consumed. At times like now convenience eating has become crucial as life today is very fast and people prefer quick meals. Snacking is also becoming a popular culture in some societies. People now go to snack outlets for their free time to spend a happy time with their family. Success of snack foods can also be given to its taste as while manufacturing a snack more emphasis is given on flavoring. Some festivals are even linked to the consumption of a specific snack. Children especially are fascinated by the taste of snack foods. Piernas and Popkin (2010) reported that calorie intake among children in U.S. from snacks is more than 27 percent of their daily calorie intake. Hence, improving nutrition of snack has become an important aspect along with maintaining its sensory characteristics.

#### 2.2.1 Healthy Snacks

Foods like Dried fruits, salads and raw vegetables, juice, yoghurt, toast, dried fruits, cereal bars and savory biscuits were considered in the category of healthy foods by Brown et al. (2008) in their study of the role of parental control practices in explaining children's diet and BMI put. These snacks are lower in sodium, fat and sugars. In another study Drewnowski and Fulgoni (2008) explained that foods can be ranked on the basis of their nutrients for regulating labels, health claims and marketing. According to Lobstein and Davies (2009), the approach to nutrient profiling should be such that nutritional dimensions such as sugar, fat, salt content and energy density are summarized. Hence, the definition of a healthy snack changes with the required nutrients or nature of study being conducted. However, it can be concluded that a healthy snack should have good of nutrients to energy ratio.

#### 2.2.2 Unhealthy Snacks

Brown et al. (2008) categorized sugared squash/drinks, sausages, biscuits containing sugar, chocolate, pies burgers, potato crisps, savory snacks and ice cream in the category of unhealthy snacks. These snacks are generally very low in providing vital minerals but provide a large amount of calories as they contain sugars, saturated fats and sodium. Hsieh (2004) conducted a study on factors influencing the selection of healthy and unhealthy snacks among students at the university of Newcastle Australia and found that majority of the students were also of the view that unhealthy snacks are the foods containing high levels of sugar and fat. However, students also put the processed food in the category of unhealthy snacks.

#### 2.2.3 Effect of snacking on body function

In today's world, snacking has become very important. Snacks can help in increasing intake of some key nutrients but frequent snacking has also been associated with higher calorie intake (Larson and Story, 2013). Snacking between meals can prevent overeating. With the increase in snack consumption in adults and children, eating occasion per day have increased and with that time between eating occasions have decreased (Popkin and Duffey, 2010). Increasing Eating

occasions have very beneficial effects on health. It helps in reducing cholesterol and apolipoprotein (Jenkins et al., 1989). Snacking trends in US children show that they are getting 27% of their total calorie intake from snack foods (Piernas and Popkin, 2010), one other survey stated that around 97% of the American people are getting 24% of their total calorie intake from snacks (Popkin and Duffey, 2010). Hence, if calorie intake from snacks is so much and this calorie intake is expected to rise in near future, it becomes important that healthier option of snacks must be made available in the market.

#### Advantages of Snacks are listed below:

#### a) Helps to keep blood glucose steady

Snacks prepared from ingredients having lower glycemic index such as whole cereals or legumes have complex carbohydrates as well as dietary fiber which helps to keep the blood sugar in control. Complex carbohydrates such as resistant starch or dietary fiber can reduce rate at which energy from food is released, reduces spikes in the blood glucose. Also, energy released slowly over a longer period can help keep the blood glucose level steady.

#### b) Satisfy Hunger

Snacks helps to keep us feel full and reduce overeating during meals. Consumption of snacks prevents people from getting too hungry. Also, snacks provide much needed energy in between meals, essential for proper functioning of body. For this purpose, carbohydrate, rich food can be combined with fruits and vegetables or with protein rich foods.

#### c) Provides key nutrients

Snacks help in providing key nutrients. Snacks having considerable amount of proteins and help body get essential amino acids apart from providing essential vitamins and minerals. Snacks having higher micronutrient content and other bioactive compounds are also being developed to provide certain functional properties to food.

#### 2.3 Extruders in food industry

The application of screw for conveying material was first started by Archimedes. Now, extruders utilize one or more screws in a barrel. In 1870's extruders were developed to produce sausage (Kokini et al., 1992). At first, single screw extruders were used in food applications in 1930's to produce pasta (Drewnowski and Fulgoni, 2008). Then, after few more years ready to eat

breakfast cereals were developed with the help of extruders by General Mills, Incorporation. 1930's saw the use of first twin screw extruders also, both co rotating as well as counter rotating.

In late 1940's the desire to improve palatability, acceptability and digestibility of animal foods led to the development of extrusion cooking technology and late 1950's saw improvement and development of new single screw extruders which provided more shearing, working and shaping functions. One of the major application of extrusion cooking was in 1960's, when it was employed to plasticize soy protein concentrates and isolates to produce the textured vegetable protein, which became one of the most widely practiced application of this technology (Harper, 1981; Riaz, 2000).

In the mid 1970's cooking and forming of food products was achieved with the help of twinscrew extruders, to overcome the restrictions of single screw extruder as twin screw extruder was able to provide a more or less forced flow, and gave better results on the laboratory extruder types in use for product development (Moscicki and van Zuilichem, 2011). Nowadays, twin screw extruders are the most widely used type of extruders. Presently, use of single screw extruders in most cases has been limited to pasta production only. However, the production capacity of single screw extruders has been increased from a few hundred kilos to tons since the initial uses of single screw extruders.

#### 2.3.1 Principle of extrusion Technology

In today's world food extruder is termed as a machine with Archimedean screw characteristics that can continuously process a product. Extruders may be designed for different purposes namely mixing, homogenization, cooking, grinding, vacuumization, cooling, shaping, cutting and filling operations and not all extruders are cooking extruders (Riaz, 2000). Kearns (1998) defined extrusion cooking as, "A continuous with the help of which moistened, expansible, starchy and proteinaceous materials are plasticized and cooked by a combination of pressure, moisture, temperature and mechanical shear". So, extrusion cooking is actually a tool of using mechanical and thermal energy to food and feed ingredients, forcing basic components such as starch or proteins, to undergo chemical and physical changes to produce a product of desired shape. The main importance of extrusion technology is because of its ability to manufacture products very efficiently. During development of extruder, conveying and shaping fluid forms of raw materials was considered as the main role. Further, extrusion also offers a chance of extruding the

materials which were not regarded as better for economics of the industry but had nutritional importance (e.g., tomato pomace). Hence, depending on the type of food selection of appropriate extruder configuration to achieve desired results is crucial for the success of extrusion. The four most widely used types of extruders are: single-screw 'wet' extruders, single screw 'dry' extruders, single screw interrupted-flight extruders and twin screw extruders. After selecting the appropriate extruder, assembly and maintenance of the extruder is also very important (Riaz, 2001). Firstly, raw material is fed into the extruder barrel where the screw conveys the food along it. Down the barrel as the flights become smaller there is restriction in the volume and it causes resistance to the movement of the food and fills the barrel and spaces between screw flights compressing the food. As the raw material moves further along the barrel, the screw kneads the material into a semisolid plasticized mass. Temperature is raised because of the friction and the additional heat. The plasticized mass then moves onto the section of the barrel having smallest flights, where pressure and shearing is increased to the most. After that plasticized or molten food is pushed through the die at the discharge end of the barrel. As the pressure outside the barrel is low, pressure difference experienced by food expands it to its final shape and cools as the moisture is flashed off as steam. The rotary knife cutter controls the length of the desired product. Different types of shapes and size of material can be produced according to the configuration and dependent variables employed. So, knowledge of behavior of tempered dough and relation of transfer of mass, heat and momentum on to the physical and chemical properties of food material is also important. As mentioned by (Moscicki and van Zuilichem, 2011) that even if the mass flow in single screw extruder is controllable, it is always better take advantage of extruder offering better mixing and steadier mass flow.

#### 2.3.2 Classification of extruder

a) According to operation extruders are classified as: cold extrusion and extrusion cooking

In cold extrusion, the process of extrusion is done without any addition of thermal energy externally. Whereas for extrusion cooking thermal energy is the integral part of extrusion processing. The thermal energy is provided externally as well as generated internally with the help of friction.

b) Based on the type of construction extruders are classified as: Single screw extruder and Twin screw extruder

*Single screw extruders* consist of a screw rotating in a cylindrical barrel. Length to diameter ratio ranges between 2:1 to 25:1. The screw is driven by electric motor and speed of screw is most important factor influencing performance of the extruder. Generally, screw speed is 150-600 rpm and compression is achieved with the help of back pressure by die and with the help of different configurations of screw and barrel. Single screw extruders have three main sections: (1) Feed (2) Transition (3) Metering (Karwe, 2003). The disadvantages of a single screw extruder are that the mixing of the material is poor and its efficiency is limited with multi-component mixtures of raw materials (Moscicki and van Zuilichem, 2011).

Process	Temperature	Max. Pressure	Moisture	Max Fat	Starch
	(°C)	(bar)	(%)	(%)	Gelatinization (%)
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					
1) Single screw extrusion	80-140	80-140	15-35	22	80-100
2) Twin screw extrusion	60-160	60-160	10-45	47	80-100

Table 2.1 Range of parameters used in different extruders (Riaz, 2000)

*Twin screw extruders* are more complex in terms of design and have two barrels of same length which are placed inside the barrel of the extruder. Twin screw extruders are generally classified based on rotation of direction of the screw rotation as counter rotating or co-rotating twin screw extruders.

In counter rotating extruder, screw rotates in opposite direction. They are good conveyors. However, such extruders are not widely used in food industry. Co-rotating screw extruders are the ones in which screw rotate in the same direction. They are the most widely used extruders. Their main advantages are good control over residence time, pumping efficiency, self-cleaning mechanism and uniformity of the process (Schuler, 1986). These extruders are further divided based on position of the screw in relation to one another.

### Intermeshing and non-intermeshing

When flights of one screw engages or penetrates the channels of the other screw, it is called intermeshing screw configuration. So, it offers features like positive pumping action, efficient mixing and self-cleaning.

In *non-intermeshing screw* extruder, the screws do not engage in each others threads, so that each screw rotates without any interaction with the other screw. Like single screw extruders they also depend on friction for extrusion (Riaz, 2000).



Figure 2.1 View of extruder assembly (adapted from: Fang et al. (2003))

# 2.3.4 Components of food extruder

Feedstock- The material that is to be used for extrusion.

**Pre-conditioner-** An assembly of the extruder which helps in adjusting moisture content and temperature of ingredients and it can be used to partially cook the ingredients also.

Screw- The component of the extruder which conveys the feedstock through the extruder.

-flight: The helical surface of the screw.

-pitch: The angle of that helical surface (flight) with the central axis.

-root: The shaft or the solid part of the screw, or the base on which flights are embedded.

Barrel- A pipe like assembly which have a screw.

Vent- Vent is an opening just before the die plate from where steam and pressure can escape.

Die plate- opening at the end of the barrel that shapes the final product.

Knife cutter- It is that part of the extruder which helps in adjusting the length of the final product by adjusting the speed of the cutter.

#### 2.3.5 Factors that are affect extrusion cooking

#### a) Rheological properties of the feedstock

The final product properties especially texture and color are affected by the properties of the feedstock such as: type of feed material, moisture content, physical state of the material, chemical composition such as starch, sugar, protein, fat content as well as pH.

#### b) Operating characteristics

- 1. Temperature,
- 2. Pressure,
- 3. Diameter of die aperture,
- 4. Shear rate

#### 2.3.6 Advantages of extrusion cooking

- a) Cost: Extruders has lower cost as compared to its high productivity.
- b) Versatile: Extruders can operate with different compositions of feedstock
- c) Productivity: extruders can operate continuously with high amount of throughput
- d) Product safety: High temperature used during extrusion cooking kills pathogens and inactivate enzymes also
- e) Environment friendly: It is safe for environments as effluent production is not present.
- f) Shaping: Multiple type of shapes can be given to the food products to make it attractive.
- g) Food quality: As high temperature is provided only for a short amount of time the effect on nutrients is minimum and proteins and starches become more digestible (Riaz and Rokey, 2011).

#### 2.4 Raw Materials for extrusion cooking

There are lot of materials that are presently used in the extrusion process. All the foods that are extruded are basically similar in ingredients. It is mainly the transformation of raw materials or the ingredients during extrusion process that differentiate it from other processes. Therefore, it is very important that the ingredients or the raw materials are chosen wisely to get a good product.

#### 2.4.1 Ingredient classification based on food components

The Components that have major effect on the properties of final product are starch, water, protein, fiber, additives, oil and particle size.

#### a. Starch

It is the major component of usually all the final products. For nutritional and sensory quality of product control of starch and carbohydrate is important. Cereals contain 50-85% starch and legumes contain 25-50%, tubers have one of the highest starch contents with 60-90% starch content. Gelatinization and dispersion of starch granules occur during extrusion which results in a continuous phase formation in extruder. In extrusion cooking gelatinization can even occur at 12-22% levels. During extrusion components of starch amylose and amylopectin give expansion to the food product (Lusas and Rooney, 2001).

#### b. Water

Water is one of the major constituents and play a major role in the process of extrusion. It helps in gelatinization of starch and proper dispersion of ingredients into the mix. During cooking it is transformed into vapors and leaves the product at die leaving air cells in the product and helping in the product expansion. For most of the extrusion processes moisture is added before the product is fed to extruder, mostly moisture addition is done in the preconditioner.

#### c. Protein

With extrusion protein undergoes denaturation and its digestibility is improved as with denaturation the access sites where enzymes can act easily are exposed. Thus, extrusion also helps in enzyme inactivation also. Because of high temperatures, Maillard reaction can also occur and as most of the foods are low in proteins so inclusion of more proteins in the food is a growing trend in industry.

#### d. Lipids

The most important functions of lipids are that they act as lubricant and hence affect the quality of the product. Temperature of 40°C make the lipids melt and shearing breaks it such that the dispersed droplets have average size of less than 1  $\mu$ m and mix the parcels into the whole mix. Lipid content of 2-3% or above can have undesirable effects on the final product as the energy input for extrusion gets decreased as friction between mix and extruder is decreased (Frame and Harper, 1994).

#### e. Fiber

Fiber is considered one of the major components of healthy modern diet. Bran is fibrous material and it can be in the dispersed phase in an extruded products that can be found in the starchy continuous phase (Guy, 2001). However, fiber can have negative impact on the extrusion process as it can affect the formation of starch linkages.

# f. Additives

Additives are the components that play a significant role in the characteristics of final product. Additives are added to the food product for achieving a specific purpose like coloring a product, improved flavor or increasing expansion of the product.

# Table 2.2 Types of products prepared with extrusion processing

Туре	Example
Directly Expanded	Puffed Breakfast cereals
Unexpanded	Noodles
Half products (3 <sup>rd</sup> generation snacks)	Pellets
Co-extruded	Caramel
Modified	Starches
Texturized	Texturized vegetable protein
Candy	toffee

# source: Kokini et al. (1992)

# **2.5 Formulation Ingredients**

Cereals are main components of extruded puffed snacks. For the present study, the ingredients used are the following:

- I. Rice Flour
- II. Green Lentil flour
- III. Chickpea Flour
- IV. Tomato Powder

### I. Rice Flour

Rice (*Oryza sativa*) is one of the highest cultivated crops of the world. It is highly consumed crop with around half of the population of the world consuming rice (Shimamoto, 1995). Rice production alone accounted for almost 20% of the total world cereal production (FAOstat, 2014). Rice milling is an important step before production of consumable rice grains. During milling of rice broken rice is generated as a by-product of rice milling process. Broken rice kernels are separated from whole rice grain because of its undesirability by consumers. Such a broken rice is usually available at lesser than half the price of whole rice but have almost the same nutritional content of whole rice grains. Rice flour is considered a good ingredient for product development
as it do not interfere with taste of product to be produced because of its bland taste, white color, hypoallergenic properties (Kadan et al., 2003). In Asian countries rice is also regarded as a source of dietary protein. However, like the other cereal crops rice is also deficient in certain amino acids such as lysine and tryptophan (Sotelo et al., 1994; Chavan and Duggal, 1978). Rice flour has higher content of amylopectin as compared to amylose. Gelatinization, retro-gradation and pasting properties are mostly dependent on the structure and composition of amylopectin. Gelatinization of the range 93-99% is possible with extrusion cooking (Guha et al., 1998). Rice flour contains about 5% of protein and is gluten free, so it is suitable for the person having allergy to gluten.

It is one of the main components of ancient diet. Its soft taste, mild color and rich aroma adds to its popularity. Heating makes it elastic and chewy and after cooling it becomes glossy and gives a chewy texture. Addition of rice flour makes a product crispier and better in texture. A lot of studies involving rice flour have been done. Potato and rice have a similar proximate composition on dry basis (Juliano, 1992). Homnick and Huxsoll (1973) stated that rice is envisioned as an ingredient that can mimic potato fry like food in food industry and devised a method involving extrusion followed by frying for producing an alternative to pro fried French fries (potato fries). Zukerman and Zukerman (1988) provided a breakthrough technology for production of rice based products through usage of microwave technology. This rice based product had the provision of being stored after preparation (prepared using a forming process) and then cooked or expanded with the help of microwave technology. However, (Kadan et al., 2001) demonstrated that by using extrusion instead of a simple forming process can provide better texture as well other sensory characteristics to rice fries along with a provision of addition of supplementing fries with proteins or other nutrients. Snacks prepared from rice flour has even traditional as well as religious significance. Some of these snacks that can be found in western markets include flaked rice, murukku, papads, sev (fried rice noodles) and murmure (puffed rice). Some of these snacks are prepared from rice alone and others include incorporation of legume flours also (Lusas and Rooney, 2001).

# **II. Green Lentil Flour**

The 68<sup>th</sup> United Nations general assembly declared the year 2016 to be the International Year of Pulses. The Food and Agriculture Organization was given the task its implementation with the aim of creating opportunities to better utilize the pulse based protein and protein nutrition with

improvement in global production and processing and creating awareness among consumers. Canada is the largest producer and exporter of lentils. In 2011, Canada accounted for 34.8 percent of the global lentil production (StatCan, 2011). Among lentils, green lentils are the most commonly produced variety of lentils with green lentils accounting for almost 75% of the total lentil production of Canada (PulseCanada, 2007).

Lentils are considered one of the best sources of vegetarian protein. The protein content of green lentils range from 28.4% to 30.1% and starch content ranges from 43.4-44.0% (Wang and Daun, 2006). Amylose content of lentils ranges from 29.8-32%, with onset gelatinization and peak gelatinization temperatures for starch being 61°C and 69°C, respectively (Joshi et al., 2013). Amylose concentration of greater than 30% along with good amount of dietary fiber can reduce rate of release of energy into the body (Chibbar et al., 2010; Yokota et al., 1994). Hence, diets that have considerable amount of pulses such as lentils have low glycemic index and can improve stability in blood sugar level. Lentils are also one of the best sources of micronutrients such as calcium, iron, magnesium, phosphorus and zinc as well as B-complex vitamins especially folate (Wang and Daun, 2006).

Antioxidants in diet play a very important role in enhancing human health by preventing damage to cellular components because of the reactions that involve free radicals (Young and Woodside, 2001). Tocopherols, ascorbic acid and phenolic compounds are the components that occur naturally in fruits, vegetables and grains and increase their antioxidant value. Phenolic compounds have been demonstrated to exhibit antioxidant properties (Kähkönen et al., 1999) and in case of lentils it is its high phenolic content that increases its antioxidant value (Fernandez-Orozco et al., 2003). Mode of action of phenolic compounds is in several ways they can act as scavengers, can bind with free radicals or can chelate with metal ions reducing oxidation reactions (Lopes et al., 1999; Bravo, 1998). However, some of the phenolic compounds such as tannins and phytic acid can also act as antinutrients which can limit consumption of lentils (Salunkhe and Kadam, 1989). Tannins can reduce bioavailability of amino acids (Davis, 1981) whereas phytic acid can reduce absorption of minerals by body (Deshpande et al., 1982). Extrusion processing of foods is a good method of reducing these antinutrients (Harper and Clark, 1979). Effect of extrusion in significant destruction of antinutrients have been demonstrated by many researchers (Anton et al., 2009; Shimelis and Rakshit 2007; Alonso et al., 2000)

Extrusion processing by incorporating lentil flours can not only add value to this pulse, but is also beneficial for Canadian growers. Nutritionally it is considered one of the best sources of plant proteins. Several studies have incorporated lentil flour develop snacks. Some of the studies include preparation of snacks using Corn-lentil flour (Lazou and Krokida, 2010); corn-lentil and corn oil (Dogan et al., 2013) lentil flour based extrusion (Hicsasmaz et al., 2011) as well as incorporation of lentils as a part of blend of legume flour with rice (Balasubramanian et al., 2012) or sorghum and wheat (Balasubramanian et al., 2012).

# **III. Chickpea Flour**

It has been reported as the third most important legume in the world (Singh et al., 1991). Chickpea is popular in India and Mediterranean regions of Asia and Africa. India is one of the leading producers of this pulse accounting for 75% of the total world production. Canada is also a significant producer of this legume and stands 9<sup>th</sup> for its share in the total world production (StatCan, 2011).

Chickpea flour has been used in traditional Indian snacks as well as Mediterranean cuisine. In Indian cuisine, it has been in popular use to produce fried snacks in combination with cereals especially rice such as sev, boondi, chakli/murukku, sweets and main meals. Similarly, in Mediterranean cuisine it is used in making fried patty called falafel and famous spread called hummus (Gaur et al., 2015). As reported by Geervani (1989) the main reason of its inclusion to snacks is because it imparts a distinctive flavor and taste to the snacks apart from nutrition. Roasted chickpea, which is a famous snack is popular because of its natural flavor only.

Nutritionally, Chickpea flour is also very good source of protein and carbohydrates covering 80 percent of the total flour. Even though protein content of chickpea (20-22%) is lower than other legumes it has high protein efficiency ratio of 2.6 better than mung beans, black gram and even soybean (Bahl, 1990; Kadam et al., 1989). Chickpea is the only cold season legume which has high fat and is a rich source of essential polyunsaturated fatty acids and imparts hypercholesteremic effects to functional snack products (Jukanti et al., 2012; Zulet and Martinez, 1995). Amylopectin is major component of starch and gelatinization of chickpea flour can be obtained at 63.5-68 °C. It also provides high amount of crude fiber ranging from 7.1 to 13.5%. Chickpea is rich in micronutrients with good iron bioavailability, better than other legumes. It is also a good source of potassium and calcium, and is beneficial in controlling hypertension (Chavan

et al., 1987; Cowan et al. 1967). Over the last two decades, Chickpea flour has been extruded alone or with different cereals to obtain snacks. Some of these major studies include extrusion of defatted chickpea flour (Batisuti et al., 1991); maize-chickpea extrudates as weaning food (Milán-Carrillo et al., 2007) as well as rice-chickpea flour (Guha et al., 1997). In another study, Bhattacharya and Prakash (1994) demonstrated that extrusion of rice and chickpea flour blends after produced acceptable snacks and can provide an alternative to cereal based snacks in market.

#### **IV. Tomato Powder**

Tomato is one the major vegetables consumed all over the world, has become integral part of diet. Tomatoes are the second most produced vegetables in North America. However, high amount of water in tomatoes has made them perishable and its storage is also difficult. Hence, 80% of tomato consumption comes through processed food such as ketchup, paste or puree (Gould, 2013). Numerous health benefits are reported with the consumption of tomatoes or a diet rich in tomato products (Agarwal and Rao, 1998). Lycopene, a carotenoid present in tomatoes in high amounts is considered responsible for its health enhancing effect. Lycopene has a high oxygen scavenging activity and hence act as an antioxidant, thereby decreasing risk of certain types of diseases like prostate cancer, asthma (Wood et al., 2008) along with providing protective effect against oxidative damage (Johnson, 2000). Lycopene dietary intake of 6-15 mg daily is recommended for health improvement (Kun et al., 2006). Tomato powder apart from having antioxidant effect induce intercellular communication and has modulating effect on hormones, immune system and metabolic pathways (Rao and Agarwal, 1999). Masatcioglu et al., (2013) reported that 12% incorporation of tomato powder in corn extrudates can increase antioxidant activity by 12 times. Several other studies have been conducted incorporating tomato powder mainly to improve antioxidant properties and functional properties of a food product, however it is necessary to take care of the substitution level of tomato pomace as it can also impart color and characteristic flavor to the product (Navarro-González et al., 2011).

# 2.6 Response surface methodology (RSM)

Response surface methodology a tool to develop and describe relations between a process variable with product quality response statistically (Giovanni, 1983). The methodology use a first-degree polynomial model to approximately give response variables (Box and Wilson, 1992). The model has the advantage of being employed with ease and can do approximate estimation of the

variables even when the knowledge of process is little. Hence, the method finds frequent use to explore influence of input variable on the response variables of a product or a process. RSM is based on the idea of using designed experiments to optimize responses. For this purpose Box and Wilson (1992) acknowledged that this model is only an approximation and suggested to use a second-degree polynomial model.

D-optimal mixture design is a type of experimental design that is used in RSM. D-optimal mixture design offers advantage over completely replicated factorial design especially in a circumstance of limited budget and time (Ruseckaite et al., 2014). Salunke (2016) explained this by taking example of three factors as: A with two levels, B with three levels and C with six levels. Hence, one complete replication of this experiment may require 2x3x6 which is equal to 36 runs and can take a long time.

# 2.7 Drying Process of food

Dehydration is the process of removal of water from a specific substance, mainly employed in foods for increasing its shelf life by arresting growth of microbes (Ratti, 2008). Drying is used for the purpose of food preservation since a long time and currently also, it is employed on a large scale. The medium of drying used frequently in industry include air, hot oil, solvents or solutions (Karel and Lund, 2003).

Among all the methods air drying is the most frequently used method. During air drying moisture from product is taken up by air until equilibrium conditions leaving the air behind saturated. Equilibrium is condition at which vapor pressure of food as well as that of surrounding air becomes equal (Ramaswamy and Marcotte, 2006). Also, even though removal of moisture continues till achievement of equilibrium conditions but drying rate will not remain same because of the changing vapor pressure of both product and the medium. As reported by (Yu, 2011) calculation of drying rate or drying curve can help give information about time to dry a product under specific conditions.

# 2.7.1 Water activity $(a_w)$

Water activity is a dimensionless quantity and is calculated as the ratio of water pressure above sample to that of pure water at the same temperature. Water activity of pure water is one. Water activity of a product is an indirect indicator of its perishability. Moisture content as well as food system are the major factors that affect water activity of a product.

Measurement of water activity gives estimation of stability of microorganisms (spoilage causing and pathogens) and internal physiological process (enzymatic and non-enzymatic). Water activity affects growth of microorganisms and a water activity below 0.75 is considered safe (Ramaswamy and Marcotte, 2006).

# **PREFACE TO CHAPTER 3**

Extrusion technology being a versatile technology has been in use since a long time for producing snack foods. Extruded snacks are highly popular because of the convenience as well as sensory properties they offer. However, unhealthy snacks with poor nutrient composition are increasingly becoming centre of concern because of the potential health risks they pose to human population. Hence, it is necessary to develop snack foods that are more nutritious and/or have health enhancing functional components.

The focus of Chapter 3 is to understand effect extrusion processing variables namely the proportions of rice flour, green lentil flour, chickpea flour and tomato powder on the physical properties of the extruded snack food produced using twin screw extrusion processing. The proportions of these four ingredients in formulations were chosen using a D-optimal constrained mixture design. Effect of processing on antioxidants and antinutrients present in food is also evaluated. The ultimate objective is to produce a snack rich in protein and with higher amounts of antioxidants.

All the experimental work and data analysis were conducted by the candidate under supervision of Dr. H. S. Ramaswamy. A manuscript draft from this chapter is prepared for publication:

**Singh, P**. and Ramaswamy, H.S., 2017. Drying characteristics and physico-chemical properties of extruded products from mixtures of rice, green lentil and chickpea flours, and tomato powder. (draft)

# DRYING CHARACTERISTICS AND PHYSICO-CHEMICAL PROPERTIES OF EXTRUDED PRODUCTS FROM MIXTURES OF RICE, GREEN LENTIL AND CHICKPEA FLOURS, AND TOMATO POWDER

# Abstract

In this study, a nutritious snack was prepared with different blends of rice flour (R), green lentil flour (L), chickpea flour (C) and tomato powder (TP) using a D-optimal mixture design with constrained levels of 33-60% for R, 20-45% for L, 10-30% for C and 0-7% for TP. Extrusion conditions were set at a predetermined temperature, screw speed and die diameter. Twin screw extrusion was followed by drying to reduce extrudate water activity. Experimental conditions were obtained using the D-optimal mixture design and contours plots and ANOVA were used to evaluate the significance of independent and interaction effects of formulation variables on product's physical properties (expansion ratio, breaking stress, bulk density, rehydration ratio and color) and moisture content of the product. ANOVA results showed that the formulation variables had significant effects (p<0.05) on all physical properties. Incorporation of lentil and chickpea flours produced snack foods with 12-19% protein. Further, addition of tomato powder and lentil flour improved the antioxidant properties of the extruded products. Furthermore, the extrusion process resulted in destruction of more than 80% of anti-nutritional factors. Nutritious products could therefore be obtained from extrusion cooking of L, C and TP supplemented R.

# **3.1 Introduction**

Twin screw extrusion cooking is defined as a processing technique that employs high temperatures, high pressures, short residence times that brings multiple physiochemical changes in the raw materials (Mercier and Cantarelli, 1986). The changes produced during extrusion has led to production of different types of directly expanded snacks foods such as ready to eat breakfast cereals, puffed snacks, chips, and others.

Starch acts as the main ingredient for structure formation in expanded snacks. Rice is a good source of starch and is considered good for product development because of its bland taste and attractive white color necessary for developing products of lighter color (Kadan et al., 2003). Furthermore, the milling process for rice produces broken rice, a major by-product. Broken rice has the same nutritional and functional properties of whole rice (Alam and Kumar, 2014).

Therefore, not only extrusion provides value addition to broken rice but also decreases cost of production.

Because rice is predominantly carbohydrate rich, and because higher growth projections for extrusion food markets have increased the demand for nutritional diversity, pulses can be used to make up for the lack of nutritional diversity. For this reason, the United Nations has made recommendations for increasing utilization of pulses to alleviate nutrient deficiencies especially among developing countries. Green lentils and chickpeas, like all other pulses, are rich in protein, fiber, and micronutrients including vitamin B especially folate and minerals such as iron, zinc, calcium, magnesium, potassium, and phosphorus (Mitchell et al., 2009; Jukanti et al., 2012; Faris et al., 2013). Pulses, especially chickpeas, have been part of Indian and Mediterranean diet since a long time. India is the largest producer of chickpeas, and chickpea flour, because of the characteristic flavor it imparts, has been used for production of traditional snacks such as murukku, savory snacks, chapatti, falafel as well as popular spreads such as hummus (Geervani, 1989; Gaur et al., 2015).

Even though chickpeas have lower protein content as compared to other major pulses, it has significantly higher fat content especially the nutritionally important polyunsaturated fatty acids and essential fatty acids that cause an hypercholesteremic effect on the body (Jukanti et al., 2012; Zulet and Martinez, 1995). Green lentils on the other hand have higher protein content as compared to chickpeas, and Canada is the largest producer of lentils in the world (StatCan, 2011). Both chickpeas and lentils are rich sources of dietary fiber that help to reduce the glycemic index of snacks, helping control blood glucose levels. However, presence of antinutrients in pulses such as tannins and phytic acid can affect the bioavailability of certain nutrients present in diet (Champ, 2002). Extrusion cooking is considered one of the best method to get rid of these antinutrients without modifying the protein content of snack (Alonso et al., 2000).

The processing effect on antinutrients (carried out mainly by extrusion) depends on techniques and conditions including time, temperature, type of ingredients and moisture content of the formulation being processed (Nestares et al., 1999). Hence, for production of a new snack, determination of these antinutrients in final product is necessary to make health claims. Additionally, successful incorporation of tomato powder in a snack can enhance its health applications apart from providing basic nutrition. Tomato powder is rich in bioactive compounds

especially lycopene with high antioxidant activity and has been related to decreasing risk of cancers (Johnson, 2000).

Further, the moisture content of the extrudates immediately after the extrusion process can be high which affects the shelf life of product. Hence drying followed by mild extrusion process may be a step required for shelf stability of extruded products. Extrudates can have water activity of 0.9 or above depending on the initial moisture content, ingredients and extrusion process variables. However, for achieving shelf stability of the product water activity of  $\leq 0.75$  is required. Higher protein content leads to interaction with carbohydrates which influences the functional properties and drying behavior of the product (Onwulata et al., 1998). Increase in protein and dietary fiber at the expense of starch can alter the physical characteristics as well as drying behavior of the product making it necessary to evaluate drying conditions to characterize the drying behavior. Some studies have reported that higher fiber inclusion into the snack can also lead to decrease in puffiness, non-crispiness and undesirable texture (Lue and Huff, 1991). However, these challenges can be tackled using proper adjustments in feed material ingredients, and hence optimization of ingredients incorporated in the snack to get maximum desirability is required.

Consequently, the objectives of this research were to produce snacks with extrusion processing followed by suitable drying to reduce moisture content of the snacks for better shelf life. Drying kinetics of the extruded product were to be evaluated to identify effect of variables on drying process. Subsequently it was also essential to evaluate physical and nutritional characteristics of the product after extrusion processing.

#### 3.2 Material and Methods

# 3.2.1 Materials

Rice flour (R) and green lentil flour (L) were purchased from a local Bulk Barn store located in Montreal, Canada (with rice flour composition of protein: 5%, fat: 1.25%, carbohydrate: 80% and dietary fiber: 2.5% and green lentil flour (L) with manufacturer composition of protein: 36 %, fat: 1.25%, carbohydrate: 60 % and dietary fiber: 2.5% as declared by the manufacturer). Chickpea flour (C) was purchased from Well Canada, Ontario, with the manufacturer specified composition of protein: 20%, fat: 6.66%, carbohydrate: 60% and dietary fiber: 16.65%. Tomato powder (TP) was purchased from Z Natural Foods (West Palm Beach, Florida, USA) having a label declared composition of protein: 13 %, fat: 0%, carbohydrate: 75% and dietary fiber: 16%.

#### **3.2.2 Experimental Design**

The study was carried out using different combinations of R, C, L and TP to prepare preextrusion feed mixtures. A statistical experimental design was generated using Design Expert software (Version 9.0.3, State-Ease Inc., Minneapolis, MN) using the D-Optimal Mixture Design. The design generated 20 experimental test run conditions with the 4 product variables with the predetermined set of constraint values. To calculate and minimize the effect of different runs, experimental runs were performed in random fashion and with replicated points for error computation. The constrained values of the of the independent variables were: Rice flour 33-60%, green lentil flour 20-45%, chickpea flour 10-30% and tomato powder 0-7%.

#### 3.2.3 Experimental material preparation

The quantities of the different variables given by D-optimal mixture design were recorded (based on a mass balance approach) using an excel sheet. The flours were weighed according to the predetermined experimental runs and were mixed using the Hobart mixer (Hobart Food Equipment Group Canada, North York, Ontario, Canada). Water was added while mixing to get the desired level of moisture content. The prepared blends were then sealed in a polyethylene bags and then kept at 4° C for 12 h before extrusion processing.

#### 3.2.4 Extrusion processing

After keeping the experimental material for 12 h, extrusion processing was carried out in a twin-screw extruder (DS32-II, Jinan Saixin Food Machinery, Shandong, P. R. China). The extruder had a barrel length to diameter ratio of 20:1 with the screw diameter of 30 mm. The length of the die was 27 mm with the hole having diameter of 5 mm. The extruder consisted of four individually controlled temperature zones for cooking the product and adjustable screw speed for better control on the variables of finished product. Before processing the extruder, barrel was allowed to heat up and processing was only started after it reached the desired temperature of 140°C at die exit. Extruder feeding was done manually through the conical hopper and the feed rate was adjusted such that the flights of the screw were always filled and accumulation was avoided.

Run	Rice % (g)	Lentil % (g)	Chickpea % (g)	Tomato Powder % (g)	Moisture Content (% wb)
1	33 (132)	33.5 (134)	30 (120)	3.5 (14)	27.34
2	52.83 (211.32)	28.33 (113.32)	17.08 (68.32)	1.75 (7)	27.19
3	41.50 (166)	28.5 (114)	30 (120)	0 (0)	27.19
4	60 (240)	20 (80)	16.5 (66)	3.5 (14)	27.24
5	39.33 (57.32)	36.33 (145.32)	22.58 (90.32)	1.75 (7)	27.24
6	33 (132)	45 (180)	22 (88)	0 (0)	27.19
7	33 (132)	45 (180)	15 (60)	7 (28)	27.44
8	33 (132)	45 (180)	15 (60)	7 (28)	27.44
9	50.75 (203)	35.75 (143)	10 (40)	3.5 (14)	27.25
10	60 (240)	23 (92)	10 (40)	7 (28)	27.35
11	43 (172)	20 (80)	30 (120)	7 (28)	27.44
12	60 (240)	30 (120)	10 (40)	0 (0)	27.10
13	51.5 (206)	20 (80)	21.5 (86)	7 (28)	27.40
14	60 (240)	30 (120)	10 (40)	0 (0)	27.10
15	60 (240)	23 (92)	10 (40)	7 (28)	27.35
16	33 (132)	45 (180)	22 (88)	0 (0)	27.19
17	33 (132)	33.5 (134)	30 (120)	3.5 (14)	27.34
18	44.5 (178)	30.5 (122)	18 (72)	7 (28)	27.41
19	41.5 (166)	45 (180)	10 (40)	3.5 (14)	27.27
20	50 (200)	20 (80)	30 (120)	0(0)	27.16

Table 3.1 Experimental runs generated with D-optimal mixture design with (actual) values of product variables for a batch of 400 g feed mix (excluding salt and water proportion)

*Note: Level of salt and water added was same for each run; 6 g of salt and 92 g of water was added to each batch of 400 g* 

With the attainment of stable conditions in the extruder, the product was then collected at the barrel end. It was then cooled by keeping them in an air tight container for overnight and after that, the extrudates were air dried in an air convection drier with the air velocity controlled at 0.1 m/s and temperature of 55° C to achieve a final moisture content of 18% (dry basis). After drying, the samples were again stored in air tight containers and stored in a dark place for further physical as well as chemical analysis.

# 3.2.5 Tray dryer

For drying the sample, a pilot scale tray dryer was used (Figure 3.1). The tray dryer in the lab employed a source of heating and a blower to force the heated air inside the dryer cabinet for the desired amount of time. The temperature and speed of air was controllable. To remove the air

from cabinet a separate unit to exhaust the air out of the dryer cabinet was also employed, so as to reduce the accumulation of saturated air which can have negative effect on drying rate of the food sample.

To dry the food sample, dryer was operated at  $55^{\circ}\pm1^{\circ}$  C to achieve the required moisture content of 18% (dry basis) in the sample. The air velocity was controlled at 0.1 m/s. Weight reduction in the sample due to removal of water from food sample was monitored constantly with the help of a weighing balance so that drying behavior of the product can be understood. The weighing balance was placed in the upper compartment and was attached to the drying tray with the help of a vertical rod.



Figure 3.1: Tray Dryer installed at Pilot Plant

# 3.2.6 Moisture content

For the determination of moisture content in the food sample the gravimetrical method AOAC-984.25 (Helrich 1990) was used. A measured amount of sample was taken and then transferred to a glass pan for drying the product. A convection oven (Fisher Scientific Isotemp Oven, Asheville, North Carolina) was used for complete removal of moisture. After placing the glass pan with the extrudate, the oven was operated at  $105 \pm 1^{\circ}$  C until no further drying took place which was marked with the constant weight of the product. Hence, moisture content (% wet basis) was calculated as:

M (% Wb) = 
$$\left[\frac{Wo-Wd}{Wo-Wp}\right] \times 100$$
 .....(3.1)

where:

Wo = Weight of the extrudate sample with the pan before drying (g),

Wd = Weight of the extrudate sample along with the pan after drying (g),

Wp = Weight of the empty pan (g).

Dry basis moisture (M% db) content from the wet basis moisture content (M% wb) can be calculated as:

$$M(\% wb) = \left[\frac{M(\% Wb)}{100 - M(\% Wb)}\right] \times 100$$
 (3.2)

# 3.2.7 Moisture ratio

Moisture ratio (MR) is an indicator of the available moisture that is left in the food. It helps in measuring and comparing the behavior of drying by different products after plotting a graph of moisture ratio against time. Moisture ratio for the extrudate samples was determined as:

$$MR = \left[\frac{Mt - Me}{Mi - Me}\right] \tag{3.3}$$

where:

Mt = moisture in the sample extrudates at time t,

Me = equilibrium moisture content in the sample extrudates,

Mi = moisture in the sample extrudates at initial time.

The equilibrium moisture content is the moisture content of the sample extrudates which cannot be reduced further at the given temperature and pressure conditions.

# **3.2.7.1 Diffusion coefficient (D)**

The diffusion coefficient was calculated using solution provided by Crank (1975) for an infinite slab with the help of equation:

Moisture ratio=
$$\frac{8}{\pi^2} \sum_{n=0}^{\infty} \frac{1}{(2n+1)^2} \exp[\frac{(2n+1)^2 \pi^2 D t}{4 L_0^2}]$$
 .....(3.4)

(Pabis and Henderson, 1961) stated that a linear solution is the simplest approach for this series equation and gave the assumption that it is only the first term of the series which has a significant effect and all other terms can hence be dropped. Following this assumption to make the equation simple (putting n=0) and then taking natural log of both sides gives equation:

$$\ln (MR) = \ln \frac{8}{\pi^2} - \frac{D \pi^2 t}{4 L_0^2} \qquad \dots \dots \dots (3.5)$$

Hence, using equation 3.5 and then plotting graph of ln (MR) vs time (t) will give a straight line whose slope is equal to:

slope=
$$\frac{D \pi^2}{4 L_0^2}$$
 .....(3.6)

Hence, using equation 3.6 value of D can be calculated (diffusion coefficient).

# 3.2.8 Water activity

Water activity is a very important property to measure as it has a very big effect on the shelf stability of a food product. Water activity is dependent on the available and free moisture content and varies with the blending of different raw materials and with different ratios. As water activity can be different at different temperatures, it has to be measured at a specific temperature and in this study, they were measured at a constant temperature of 25° C. Water activity of the extrudate samples was measured with a water activity analyzer in the lab (Rotronic HygroLab 3, Rotronic Instrument Corp., NY). For measuring the water activity, the samples were placed in the chamber of the analyzer until the analyzer gave a beep sound which marks the establishment of the equilibrium and the value of water activity was recorded.

#### **3.2.9** Color

The body responsible for enacting international recommendations in the field of photometry and colorimetry is CIE (Commission Internationale de l'Eclairage transcribed to English as the International Commission on Illumination). The color system standardized by CIE included specifications of the light source, the observer and the methodology used for deriving the values that have the ability to describe color.

The CIE Color Systems utilizes three coordinates to locate a color in a color space. These color spaces include:

- CIE XYZ
- CIE L\*a\*b\*
- CIE L\*C\*h°

For the current experiment, color of the extrudate sample was determined with the help of a lab scale Minolta CM-500d colorimeter (Optical Sensor, Hunter Associates Laboratory Inc., Reston VA, USA) which uses an aperture of diameter 1.2 cm. To obtain the color of sample three parameters of color were determined with the colorimeter namely: L\*, a\* and b\*. Here, L\* is the measure of the lightness of the product on a scale of 0 to 100, where 0 is for dark or black and 100 for pure white product. The a\* value of the product gives the redness or greenness of the product sample with negative values for red and positive values for a product with higher greenness. In the same manner b\* gives value for yellowness and blueness in the product with negative values for yellow and positive for blue.

# 3.2.10 Breaking stress (BS)

Breaking stress (BS) is a measure of hardness. For measuring breaking stress, 3-point bending test was employed with the help of TA-XT2 texture analyzer (Texture Technologies Corp., Scarsdale, NY/Stable Micro Systems, Godalming, Surrey, UK) available in texture analyzing lab of McGill University. The texture analyzer was equipped with a load cell of 20 kg. Following this method, the extrudate samples were placed on two rounded stands which were apart from each other by 30 mm. A round plunger was taken and mounted on the pressuring arm of the analyzer. The round plunger was then allowed to put pressure at the middle of extrudate sample at a rate of 40 mm/min until there was a break in the extrudate. The pressure required to bring the break was recorded and hence breaking stress was calculated as the force (N) required per unit cross section area (mm<sup>2</sup>). Breaking stress of ten replicated samples were calculated and then average was taken.

# **3.2.11 Expansion ratio (ER)**

To describe puffiness in the extrudates, the expansion ratio was used. Expansion ratio is the ratio of diameter of the extrudate to diameter of the die hole (Jyothi et al., 2009). ER of 10 replicates was taken with the help of a Vernier caliper and then average value was calculated.

• ER was calculated as= 
$$\left[\frac{\text{Diameter of the extrudate sample}}{\text{Diameter of the die hole}}\right]$$
 .....(3.7)

Diameter of the die hole was 5 mm.

#### 3.2.12 Rehydration ratio (RR)

Rehydration ratio is the ratio of the change in weight of the extrudate after absorbing water to that of weight of extrudate before rehydration, which is expressed in percentage (Lewicki, 1998). For the determination of rehydration ratio 15 g of air dried extrudate samples were taken and placed in a beaker containing 500 ml of water at room temperature for 15 min. After 15 min, water from the beaker was drained out and extrudates were collected. Collected extrudates were immediately weighed.

• The rehydration ratio was calculated as= 
$$\left[\frac{W_2 - W_1}{W_1}\right]$$
 .....(3.8)

W1= Weight of the extrudates before rehydration

W2= Weight of the extrudates after rehydration

W1 was taken as 15 g and was same for all the observations.

#### 3.2.13 Bulk density (BD)

For measuring bulk density, extrudate samples were cut into strands which were 25 mm long. The number of strands used for displacement method were adjusted such that the total quantity of the strands taken was around 15 g. The strands were then put into the measuring cylinder which was then filled with coriander seeds upto 100 ml volume mark. After that extrudates samples were separated from the coriander seeds in the cylinder and only the volume of coriander seeds was measured.

Hence the bulk density was calculated as:  $\left[\frac{W}{V_{E+C}-V_C}\right]$  .....(3.9)

Where:

W = Quantity taken of extrudate strands (g),

 $V_{E+C}$  = Volume of the cylinder upto which coriander seeds along with strands were added (100 ml),

 $V_C$  = Volume of coriander seeds after separating out extrudate strands (ml).

#### **3.2.14 Antioxidant activity (AA)**

The antioxidant activity (AA) of extrudates was determined using the DPPH free radical scavenging assay method. The method to determine AA was adapted from (Martínez-Valverde et al., 2002). First, for the preparation of methanolic extracts a 0.1 g of sample was taken in 0.9 ml of methanol. The solution was prepared in a 1.5 ml Eppendorf. For extraction of antioxidants the sonication of solution was done in dark for 30 minutes and after that the samples were centrifuged at 4000 rpm for 15 minutes to separate the supernatant and the extract, the supernatant was collected, and methanol was then again added to centrifuge pellet for extraction, and hence the procedure was repeated and supernatant was collected again. This supernatant was the methanolic extract that was used for examining DPPH scavenging activity. For this, 1 mM solution of DPPH was prepared in methanol (40 mg in 100 ml methanol). It is important that DPPH solution is prepared fresh every time. After this, Trolox standard curve was plotted using different concentration of Trolox (0-500  $\mu$ m). The methanolic extract was taken in 100  $\mu$ l quantity and was added to 1.5 ml of DPPH solution. After this it was kept in dark for 30 minutes and absorbance was compared with the Trolox standard curve to express the values in  $\mu$ mol TE/ 100 g db. The wavelength used was 517 nm and solution was blanked to air.

#### 3.2.15 Determination of tannin content

Tannin content was determined using the method by Price et al. (1978). It is determined using vanillin hydrochloride assay. Vanillin is a reagent that has the ability to react with a phenol having an unsubstituted resorcinol or pholoroglucinol nucleus (Siwela et al., 2007). The principal of this method is based on the development of color during reaction which is proportional to the amount of tannins present in food.

For tannin determination, first the extract is prepared by mixing 1g of sample in 50 ml of methanol (100%). For better extraction, the sample was shaken for 24 hours in methanol. After that, it was centrifuged at 1200 rpm for 10 mins at 25° C, supernatant from the centrifuge was collected and then filtered through a filter paper. This filtered extract was taken and 1 ml of this extract was added with 5 ml of hydrochloride reagent (8% hydrochloric acid in methanol and 4% vanillin in methanol). The mix was incubated at 30° C for 20 mins. Absorbance was measured at 500 nm. The amount of tannin was measured using catechin (CE) as a standard curve (10-100  $\mu$ g/ml) and data was expressed as mg CE/100 g db.

#### **3.2.16 Determination phytic acid content**

For the determination of phytic acid content, a Megazyme phytic acid (Phytase/ Total phosphorus) assay kit (#K-PHYT, Megazyme International Ireland) was used. The assay was based on the principle of hydrolysis of phytic acid in the presence of phytase as well as alkaline phosphatase into myo-inositol (phosphate)<sub>n</sub> and inorganic phosphate (Pi) (Loewus and Murthy, 2000). Further this Pi then reacts with ammonium molybdate, which can be then reduced to molybdenum blue in acidic conditions (Fiske and Subbarow, 1925), absorbance of molybdenum blue (measured with spectrophotometer) at 655 nm is hence proportional to the amount of Pi present in the sample.

To measure phytic acid content, 2.5 g of sample was extracted using 20 ml hydrochloric acid (0.66 M) accompanied with stirring the sample for 8 hours. The extract was centrifuged at 13,000 rpm for 10 min. Then 50  $\mu$ l of sample extract was taken and 20  $\mu$ l of phytase was added to it. The sample was then vortexed and was incubated in water bath for 10 mins. Thereafter, 20  $\mu$ l of alkaline phosphatase was added, vortexed and again incubated at 40 °C for 15 min. Sample was then again given centrifugation for 13,000 rpm for 10 min. At the end, determination of phosphorus was performed calorimetrically using color reagent [1-part ammonium molybdate (5% w/v) into 5 parts ascorbic acid (10% w/v)/sulphuric acid (1 M)] at 655 nm against a phosphorus calibration curve.

#### **3.2.17 Data analysis**

To predict the dependent variable second order polynomial equation was used:

• 
$$Y = b_0 + \sum_{i=1}^{i=n} b_i x_i + \sum_{i=1}^{i=n} b_{ii} x_i^2 + \sum_{i=1}^{n-1} \sum_{j=i+1}^n b_{ij} x_i x_j$$
 ...... (3.10)

where:

Y is the response variables (AA, TC, BD, ER, RR, BS or Color),

 $b_0$ ,  $b_i$ ,  $b_{ii}$  and  $b_{ij}$  are the regression coefficients and are constants,

 $x_i$  and  $x_j$  are the independent processing variables,

n is the total number of product variables (here n = 4)

The model chosen includes all the effects produced by experimental variables. It gives linear, quadratic, cubic as well as interactive effects produced by the processing variables on the response variables. The results obtained for the parameters were analyzed with the help of Design-Expert 9.0.4 (Stat-Ease Inc. Minneapolis, MN). The goodness of model was justified with the help of Analysis of Variance (ANOVA) Table. ANOVA also helped in determining the significance of effects produced by independent variables on the response parameters.

# 3.3 Results and Discussion

#### 3.3.1 Moisture content and water activity results obtained after extrusion-drying

The experimental arrangement (test run number), data observations on the moisture content and water activity at different points of drying time for the extrudate after extrusion processing (0 minutes) are listed in the Table 3.2. A part of the moisture was lost from what was present in the raw food formulation to the one present in extrudate. This is evident from comparison of the feed formulation moisture and moisture content at 0 minutes of drying in the Table 3.2. For example, it can be seen from the moisture content of extrudates at 0 min, the moisture has already been reduced from 37.62 % (dry basis) for the 1<sup>st</sup> run to 28.77 % (db) after extrusion processing. Moisture loss occurred because extrusion cooking was done at 140° C well above the boiling point of water. Most of the moisture loss happened at the die exit where water vapors leaving the extrudate are used for imparting puffing properties to the extrudate. However, even after losing some of the moisture, the moisture content of the extrudates varied between 26-30 %(db) with water activity between 0.82-0.91. Such a high water activity has a severe effect on the product's shelf stability as this high level is favorable for microorganisms to grow. Hence, the product needed further drying to bring the levels of moisture under safe level to increase shelf stability.

The regression equation for relating initial moisture content (before drying) of extrudates to product variables was obtained as:

$$Y = 0.230104 * R + 0.345842 * L + 0.294781 * C + 0.26672 * TP \qquad (3.11)$$

Where: R is percentage rice flour content; L is percentage lentil flour content; C is percentage chickpea flour content; TP is percentage Tomato Powder content. The value of  $R^2$  is 0.8409 and mean standard error is 0.57 for the fitted model.

Run	Moisture content of	a <sub>w</sub> of extrudate	Drying time to	Moisture content of	Drying time taken	a <sub>w</sub> of extrudate	Protein content at
	extrudate	before	reach a of	extrudate at a	to reach	at 18%	18% db
	before drying	drving	0.75	0.75	18% (db)	(db)	moisture
	(% db)		(min)	(% db)	(min)		content
1	28.77	0.884	54.00	22.31	138.00	0.689	17.52±0.59
2	27.67	0.849	42.00	21.27	103.00	0.702	14.42±1.03
3	27.32	0.874	44.00	21.51	129.00	0.714	$16.08 \pm 0.85$
4	26.47	0.827	36.00	20.75	70.00	0.725	12.33±0.54
5	28.25	0.876	52.00	22.20	117.00	0.692	$17.24 \pm 0.74$
6	30.80	0.897	67.00	23.04	156.00	0.685	18.99±0.27
7	29.84	0.891	54.00	23.67	143.00	0.673	$18.78 \pm 0.45$
8	28.97	0.902	57.00	22.27	137.00	0.709	$18.90 \pm 0.65$
9	28.36	0.854	42.00	22.29	107.00	0.689	$15.55 \pm 0.71$
10	26.74	0.837	39.00	21.07	74.00	0.714	$12.53 \pm 0.31$
11	27.49	0.872	38.00	21.46	87.00	0.670	$14.29 \pm 0.67$
12	27.04	0.834	42.00	21.15	81.00	0.700	$13.87 \pm 0.19$
13	27.21	0.847	41.00	21.29	78.00	0.713	$13.19 \pm 0.37$
14	26.98	0.846	38.00	21.19	80.00	0.690	$13.88 \pm 0.29$
15	26.74	0.835	37.00	20.99	72.00	0.697	$12.53 \pm 0.58$
16	29.84	0.890	41.00	23.30	149.00	0.674	$19.14 \pm 0.24$
17	29.27	0.887	37.00	22.56	129.00	0.669	$17.45 \pm 0.41$
18	27.78	0.861	40.00	21.71	94.00	0.688	$15.53 \pm 0.21$
19	28.07	0.873	56.00	21.98	139.00	0.679	$18.09 \pm 0.27$
20	27.49	0.857	43.00	21.45	91.00	0.699	13.75±0.43

Table 3.2 Moisture content prior to drying, at  $a_w$  (0.75) and after drying of extrudates, along with initial and final water activity and drying time to achieve moisture content of 18% (db)

*Note:*  $a_w = Water activity, db = dry basis$ 

Behavior of moisture change in products with respect to time can be observed through observing the change in moisture ratio with respect to time. Moisture ratio was calculated (Table 3.3) and equations for different runs for drying of extrudates (Table 3.4) were obtained with the help of Microsoft excel software. The aim was to study the effect on the moisture loss by the four product variables (rice flour, green lentil flour, chickpea flour and tomato powder) at varying drying time points (0, 15, 30, 45, 60, 75, 90, 105 mins).

Run	0 mins	15 mins	30	45 mins	60 mins	75 mins	90	105	Diffusion coefficient
			mins				mins	mins	$(D*10^8 \text{ m}^2/\text{s})$
1	1	0.88	0.79	0.72	0.67	0.64	0.61	0.58	4.30
2	1	0.88	0.78	0.70	0.65	0.61	0.58	0.55	5.80
3	1	0.89	0.81	0.73	0.67	0.63	0.60	0.58	4.71
4	1	0.85	0.75	0.66	0.59	0.54	0.51	0.48	7.22
5	1	0.88	0.81	0.75	0.69	0.65	0.61	0.58	4.55
6	1	0.90	0.83	0.77	0.72	0.68	0.64	0.61	3.78
7	1	0.91	0.84	0.78	0.72	0.69	0.68	0.64	3.32
8	1	0.90	0.83	0.76	0.70	0.66	0.61	0.57	4.40
9	1	0.89	0.78	0.70	0.64	0.60	0.57	0.54	5.64
10	1	0.87	0.78	0.69	0.61	0.56	0.52	0.49	6.56
11	1	0.88	0.77	0.69	0.62	0.57	0.54	0.51	5.33
12	1	0.87	0.78	0.71	0.64	0.59	0.54	0.50	6.07
13	1	0.88	0.78	0.70	0.62	0.58	0.55	0.52	5.80
14	1	0.87	0.77	0.69	0.63	0.59	0.55	0.52	6.45
15	1	0.86	0.74	0.65	0.59	0.56	0.53	0.51	6.22
16	1	0.90	0.82	0.75	0.70	0.67	0.64	0.61	4.18
17	1	0.89	0.80	0.74	0.69	0.65	0.62	0.59	4.20
18	1	0.87	0.76	0.70	0.66	0.62	0.58	0.54	4.86
19	1	0.91	0.82	0.76	0.72	0.68	0.65	0.62	4.06
20	1	0.86	0.75	0.67	0.61	0.57	0.55	0.53	5.88

# Table 3.3 Values of moisture ratio for different drying times and diffusion coefficient for

each run

Table 3.4 Equations obtained for different runs by plotting moisture ratio vs time

Run	Equation obtained for moisture ratio (y) vs time	R <sup>2</sup>	Mean standard
1	$v = 3E-05x^2 - 0.0074x + 0.9904$	0.9959	0.1
2	$y = 4E - 05x^2 - 0.008x + 0.9938$	0.9976	0.18
3	$y = 0.0073x^2 - 0.1253x + 1.1159$	0.9996	0.12
4	$y = 4E - 05x^2 - 0.0092x + 0.9908$	0.9985	0.17
5	$y = 3E - 05x^2 - 0.0065x + 0.9879$	0.9964	0.1
6	$y = 2E - 05x^2 - 0.0059x + 0.9921$	0.9981	0.12
7	$y = 3E - 05x^2 - 0.006x + 0.9975$	0.9961	0.14
8	$y = 2E - 05x^2 - 0.006x + 0.9937$	0.9986	0.2
9	$y = 4E - 05x^2 - 0.0082x + 0.9992$	0.9985	0.22
10	$y = 3E - 05x^2 - 0.0084x + 0.9967$	0.9994	0.2
11	$y = 4E - 05x^2 - 0.0085x + 0.9983$	0.9995	0.12
12	$y = 3E - 05x^2 - 0.0075x + 0.9887$	0.9977	0.2
13	$y = 4E - 05x^2 - 0.0084x + 0.9988$	0.9992	0.3
14	$y = 4E - 05x^2 - 0.0082x + 0.9917$	0.9979	0.12
15	$y = 5E - 05x^2 - 0.0097x + 0.9942$	0.9973	0.1
16	$y = 3E - 05x^2 - 0.0066x + 0.9954$	0.9979	0.17
17	$y = 3E - 05x^2 - 0.0069x + 0.9917$	0.9971	0.11
18	$y = 3E - 05x^2 - 0.0077x + 0.9838$	0.9891	0.18
19	$y = 3E - 05x^2 - 0.0063x + 0.9958$	0.9971	0.2
20	$y = 5E - 05x^2 - 0.0092x + 0.9925$	0.9982	0.12

Experimental formulation Run 4 and Run 7 had the lowest and highest moisture ratio's respectively after 115 mins, which means that moisture content of Run 4 was the lowest and Run 7 had the highest moisture content among all the formulations.

For understanding the behavior of loss of moisture in a food product, it is very necessary to see the behavior of diffusion inside the food system. During drying, removal of moisture occurs from the surface of food. However, the internal moisture is transferred to the food boundary through diffusion occurring inside the food. Bruin and Luyben (1980) reported that diffusion coefficient (D) is an important parameter to measure for understanding rate of drying, as in low moisture foods controlling factor for drying is usually internal mass transfer rate.

Understanding the relationship between type of food components, structure and mobility of water molecules can help in the development of an efficient method of drying. Diffusion coefficient was found by plotting moisture ratio vs time using equations 3.5 and 3.6. Slope of the curve was determined and hence value of diffusion coefficient (D) was found for each extrudate. The effect of all the individual components of the flour mix was found to be significant (p<0.05). The regression equation was obtained showing effect of the product components (actual value) on diffusion coefficient:

$$Y_{D} = 0.102622 * R + -0.00380587 * L + 0.0290101 * C + 0.0150883 * TP$$
 ....... (3.12)

The value of  $R^2$  for the fitted model was 0.9262 and standard error was 0.14. As observed from the Table 3.5 the diffusion coefficient showed strong positive correlation with rice flour, therefore value of diffusion coefficient increased with increasing rice rich in starch. However, increasing lentil flour had strong negative correlation with diffusion coefficient decreasing rate of diffusion in the extrudate and ultimately leading to increase in the drying time for extrudate.

Table 3.5 Analysis of correlation coefficient between model variables and diffusion coefficient

Value of correlation coefficient					
Variable	Diffusion Coefficient				
Rice	0.94				
Lentil	-0.80				
Chickpea	-0.34				
Tomato Powder	0.01				

Lentil flour has the highest protein content among other variables. The effect of protein on drying of the extrudate can be understood from the phenomena explained during drying process by Van Arsdel and Copley (1963). According to the phenomena, drying process is accompanied by the diffusion process inside the food product that helps in the migration of moisture from inside the extrudate to the boundary from where removal of moisture takes place. The rate of diffusion from inside a food matrix is affected by several factors including the difference in concentrations and ease of movement within the food matrix, as in food extrudate the diffusion occurs within the solid structure and/or within the pores, capillaries and small spaces which are saturated with vapors apart from the other factors such as superficial area, orientation of constituents, type and concentration of solutes (Barbosa-Cánovas and Vega-Mercado, 1996). The important factors of drying include pre-drying structure of food and composition of food. Components of food such as proteins and other macromolecules cause resistance to diffusion by obstructing the passage or because of higher bonding strength. Hence, increasing protein content yielded lower value for diffusion constant. Also, the manner of diffusion can be different depending on the food structure as well as drying conditions. As the product dries the moisture gradient between external and internal part of the food decreases leading to reduction in diffusion rate. Reduction in rate of diffusion affects the drying rate resulting in fall of drying rate, marking the start of falling rate period. Therefore, with the loss of moisture drying is slowed down.

# 3.3.2 Impact of product variables on the drying time to achieve a<sub>w</sub> of 0.75

Food is a complex system consisting of different macromolecules including proteins, carbohydrates as well as lipids. The removal of moisture from food depends on the nature of interactions between water and other components (such as hydrogen bonding or hydrophilic or hydrophobic interactions). Water activity  $(a_w)$  is the term used to relate these interactions of water with food components (Labuza, 1977). Changing food components or ingredients of a food will hence lead to change in water activity also. This is evident from the Table 3.2 that even with nearly same feed moisture, loss of water was different for different runs after extrusion. The time taken to achieve the target water activity of 0.75 was highest for Run 8 with the time taken as 57 minutes having the formulation of 33% R, 45% L, 15% C, 7% TP. The resulting moisture content for water activity to be 0.75 for the same run was 22.27% (db). Whereas the time taken to achieve targeted

water activity was shortest for Run 4 which took 36 minutes with the feed formulation of 60% R, 20% L, 16.5% C, 3.5% TP. The moisture content at 0.75 water activity was 20.75% (db).

Figure 3.2 is the response surface plot of the drying time to achieve water activity of 0.75 for the extrudate. The time taken to achieve water activity was affected significantly by all the constituents of flour formulation blends i.e. rice flour, green lentil flour, chickpea flour and tomato powder. From this Figure, it can be observed that increasing rice flour helped in decreasing the drying time to achieve water activity of 0.75, whereas increment of green lentil flour and tomato powder increased the drying time of product. The drying time was affected more by increasing the lentil flour. This is because of highest protein and fiber content of the lentil flours. Free moisture is the most important drying factor. Fiber has high affinity towards water. Hence, higher composition of such molecules can result in longer drying times.



# Figure 3.2 The response surface plot indicating time required for drying to achieve a<sub>w</sub> of 0.75 at the chickpea flour proportion of 30% in the formulation

ANOVA for drying time to achieve water activity of 0.75 is presented in Table 3.3. Drying time was influenced by all the individual proportions of variables significantly ( $p \le 0.05$ ). The model was suitable as the lack of fit for model was not significant.

Source	Drying time at a <sub>w</sub> 0.75					
	Df	SOS				
Model	3	5127.09				
Residual	16	46.56				
<b>R</b> <sup>2</sup>	0.9538*					
Standard Error	2.4					

# Table 3.6 ANOVA results for time to achieve a<sub>w</sub> of 0.75

p < 0.05 = significant

# **3.3.3 Physical characteristics**

The experimental arrangements (test run number) and data of the results obtained by examining various physical properties such as breaking strength (BS), bulk density (BD), expansion ratio (ER), rehydration ratio (RR) and color (L\*, a\*, b\* values) of the extrudates are shown in the Table 3.7 below. The approximate proximate composition of proteins in the dried extrudates from each test run has also been indicated in the Table 3.2. Run 16 with the flour composition of 33% R, 45% L, 22% C and 0% TP gave the highest protein content of 19.14%. It is evident from the Table 3.2 that increasing protein content was observed with higher incorporation of green lentil flour. Equations of second order polynomial equations were established at probability level  $0.01 \le p < 0.05$  and coefficients obtained are presented. The equations hence established were able to show up an empirical relationship between physical properties (BS, ED, ER, RR and color) and actual values obtained for independent variables of rice flour (R), green lentil flour (L), chickpea flour (C) and tomato powder (TP). To analyze the impact on physical properties of BS, BD, ER, RR and the color parameters of dried extruded products by the independent variables of rice flour (R), green lentil flour (C) and analyze the color parameters of dried extruded products by the independent variables of rice flour (R), green lentil flour (C) and analyze the impact on physical properties of S, BD, ER, RR and the color parameters of dried extruded products by the independent variables of rice flour (R), green lentil flour (C) and tomato powder (TP) an analysis of variance (ANOVA) was hence performed.

The values obtained from ANOVA shows that Rice flour (R), Lentil flour (L), Chickpea Flour (C) and Tomato Powder (TP) had significant effects  $(0.01 \le p < 0.05)$  on ER, BD, a\* and b\* values. All the four parameters showed quadratic effects  $(0.01 \le p < 0.05)$  to BS, BD, RR and b\* values, while for values of ER and a\* parameters showed linear effect. However, the parameters also showed special cubic effect towards L\* value. Interactive effects of rice flour and lentil flour were found significant for  $(0.01 \le p < 0.05)$  L\*, BS, and RR values, while bulk density was affected by the interactive effects of rice flour and tomato powder as well as that of chickpea and tomato

powder. Also, significant interactive effect  $(0.01 \le p < 0.05)$  of rice flour and chickpea was found on RR.

Run	expansion ratio	L*	a*	b*	Breaking Stress	Bulk Density	rehydration ratio (%)
- 1	1 71 + 0.02	24 (5) 2 22	4.9+0.50	10 (7) 1 02	$(1N/mm^{-})$	(g/mi)	01.0+7.51
	1./1±0.03	24.65±2.22	4.8±0.56	10.6/±1.92	$0.5/\pm0.02$	$0.59 \pm 0.04$	81.9±/.51
2	$1.89\pm0.02$	28.54±5.51	$4.2 \pm 0.89$	$9.12 \pm 1.02$	$0.33 \pm 0.03$	$0.47\pm0.03$	159.01±9.61
3	$1.79 \pm 0.02$	22.45±2.97	4.55±0.29	9.73±1.34	0.31±0.04	$0.57 \pm 0.03$	135.56±10.82
4	$1.91 \pm 0.02$	29.13±4.89	$3.85 \pm 0.34$	8.81±2.01	$0.31 \pm 0.05$	$0.47 \pm 0.04$	165.74±7.13
5	$1.79 \pm 0.02$	25.23-4.44	$3.9{\pm}1.02$	9.87±2.31	$0.52{\pm}0.02$	$0.56{\pm}0.01$	151.56±12.11
6	$1.71\pm0.01$	21.42±4.05	4.55±1.23	9.67±2.28	$0.69{\pm}0.06$	$0.59 \pm 0.04$	89.78±10.62
7	$1.67 \pm 0.02$	25.65±3.54	$4.95 \pm 0.45$	$10.17 \pm 2.20$	$0.61 \pm 0.02$	$0.61 \pm 0.05$	71.55±13.11
8	$1.76 \pm 0.03$	22.67±2.29	4.51±0.43	9.85±1.97	$0.65 \pm 0.03$	$0.65 \pm 0.04$	59.89±8.18
9	$1.85 \pm 0.02$	30.53±4.44	$3.99{\pm}0.81$	9.56±1.50	$0.35 \pm 0.04$	$0.5 \pm 0.04$	167.56±11.71
10	$1.87 \pm 0.01$	27.35±3.32	$4.02 \pm 1.16$	9.02±1.29	$0.31 \pm 0.03$	$0.54{\pm}0.02$	$149.78{\pm}10.89$
11	$1.72 \pm 0.02$	24.73±2.34	4.65±1.02	9.38±1.24	$0.45 \pm 0.05$	0.51±0.04	$117.86 \pm 8.43$
12	$1.85 \pm 0.02$	29.23±4.02	$4.01 \pm 2.00$	8.97±1.93	$0.42{\pm}0.06$	$0.51 \pm 0.05$	135.87±7.09
13	$1.82 \pm 0.01$	28.67±3.45	$4.2 \pm 0.95$	9.27±2.01	$0.32{\pm}0.05$	$0.45 \pm 0.03$	$145.89{\pm}11.36$
14	$1.92 \pm 0.03$	27.82±3.56	$3.93{\pm}0.88$	9.37±2.31	$0.31 \pm 0.02$	$0.47{\pm}0.05$	119.45±6.61
15	$1.82 \pm 0.02$	29.92±5.43	4.13±1.23	9.15±1.11	$0.45 \pm 0.04$	$0.49{\pm}0.03$	$135.78 \pm 8.55$
16	$1.78 \pm 0.01$	19.32±2.95	4.71±1.11	10.21±2.19	$0.65 \pm 0.02$	0.61±0.05	62.89±13.47
17	$1.72 \pm 0.03$	22.41±3.10	5.02±1.34	10.41±2.31	$0.63 \pm 0.04$	0.57±0.02	87.9±9.98
18	1.73±0.02	20.11±2.71	4.56±1.01	9.81±1.94	$0.42{\pm}0.05$	0.56±0.04	165.45±13.09
19	1.79±0.01	20.23±3.07	4.87±2.11	9.27±1.45	$0.49{\pm}0.02$	0.52±0.07	117.65±12.89
20	$1.82 \pm 0.02$	27.82±3.19	$4.19 \pm 1.28$	9.18±1.60	$0.28{\pm}0.01$	0.51±0.03	$163.78 {\pm} 8.01$

Table 3.7 Experimental runs and their respected observed values (mean values plusstandard deviation) of the measured physical properties

Several researchers have used rice as a base for producing extruded products along with other ingredients to enrich the snack with nutrients (Pansawat et al., 2008; Ding et al., 2005; Onwulata et al., 2001; Chauhan and Bains, 1988) with or without using pulse flours. Some studies have also been conducted for increasing the antioxidant content of snack using cereal and tomato products (Altan et al., 2009). Likewise, many researchers working on extrusion snacks have employed expansion ratio, rehydration ratio, water solubility index, water absorption index, bulk density, true density, and porosity for measuring the physical characteristics. Basically, in all these studies the influence of processing and product variables was studied. Researchers such as (Yu, 2011) studied the corn flour blends with soy flour and soy protein isolate (SPI) using twin screw extruder and the factors influencing expansion ratio (ER), bulk density (BD), breaking stress (BS), water solubility index (WSI), rehydration ratio (RR) and color (L\*, a\* & b\*) of the extrudate. Similarly, Forsido and Ramaswamy (2011) developed a protein rich extruded products from tef,

corn and soy protein isolate blends where physical properties (color, ER, RR, WSI, BD and hardness) and its relation with extrusion process variables were measured.

Responses	Source	Sum of squares	Df	Mean squares	F- value	P-value
	Regression	230.46	13	17.73	7.26	.0115*
	Lack of fit	1.20	1	1.20	0.45	0.5337
L*	Pure error	13.45	5	2.69		
	Residual	14.65	6	2.44		
	Total	245.11	19			
	Regression	1.86	3	0.62	11.38	.0003*
	Lack of fit	0.73	11	.066	2.32	0.1820
a*	Pure error	0.14	5	0.029		
	Residual	0.87	16	0.055		
	Total	2.74	19			
	Regression	35.55	9	3.95	11.93	0.003*
	Lack of fit	1.68	5	0.34	1.04	0.4853
b*	Pure error	1.63	5	0.33		
	Residual	3.3	10	0.33		
	Total	38.86	19			
	Regression	0.081	3	0.027	24.28	.0001*
	Lack of fit	7.48E-003	11	6.80E-004	0.33	.9407
Expansion ratio	Pure error	0.010	5	2.050E-003		
	Residual	.018	16	1.109E-003		
	Total	.098	19			
	Regression	23901.66	9	2655.74	17.46	0.0001*
	Lack of fit	840.31	5	168.06	1024	0.4112
Rehydration ratio	Pure error	680.32	5	136.06		
	Residual	1520.63	10	152.06		
	Total	25422.29	19			
	Regression	0.34	9	0.037	12.77	0.0002*
	Lack of fit	0.010	5	2.020E-003	0.52	0.7520
Breaking Stress	Pure error	0.019	5	3.85E-003		
	Residual	0.029	10	2.935E-003		
	Total	0.37	19			
	Regression	0.054	9	5.997E-003	11.11	0.0004*
	Lack of fit	2.149E-003	5	4.298E-004	0.66	0.6695
Bulk Density	Pure error	3.250E-003	5	6.500E-004		
	Residual	5.399E-003	10	5.399E-004		
	Total	0.059	19			

Table 3.8 ANOVA results for the evaluated physical properties of extrudates

\* $P \leq 0.01$ ; ns = not significant

# Table 3.9 ANOVA for individual variables as well as for interactions effects (significance level p≤0.05)

Source	L*	a*	b*	BD	RR	ER	BS
Model	0.0115	0.0003	0.0003	0.0004	< 0.0001	< 0.0001	0.0002
Linear	0.0016	0.0003	< 0.0001	< 0.0001	< 0.0001	< 0.0001	< 0.0001
Mixture							
AB	0.0037		0.0639	0.6988	0.0004		0.0277
AC	0.0066		0.0206	0.0182	0.0005		0.0912
AD	0.0120		0.0036	0.0326	0.0281		0.7365
BC	0.0050		0.0628	0.0527	0.0219		0.0354
BD	0.0101		0.0043	0.0696	0.0453		0.6898
CD	0.0080		0.0093	0.0183	0.1028		0.8903
ABC	0.0041						
ABD	0.0081						
ACD	0.0220						
BCD	0.0211						

Note: A- Rice flour; B- Green lentil flour; C- Chickpea flour; TP- Tomato powder

Table 3.10 Fit of experimental of	ata evaluated through ANO	VA in a D-optimal mixture
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design

Source	L*	a*	b*	ER	RR	BS	BD
R <sup>2</sup>	0.9402	0.6808	0.9148	0.8199	0.9402	0.9200	0.9091
Adj R-	0.8107	0.6209	0.8381	0.7861	0.8864	0.8479	0.8272
Squared							
Pred R-	-20.029	0.5482	0.6431	0.7067	0.7619	0.6637	0.5804
Squared							
Adeq	7.864	8.818	10.831	13.44	12.440	11.146	10.859
Precision							

# I. Color

Color is one of the important factors affecting the consumer acceptance. A color is differentiated and identified based on the three basic elements of it which are hue, value (lightness) and chroma. The color of the dried extrudates was measured with Minolta Tristimulus Colorimeter (Minolta Corp., Ramsey, NJ, USA) and software SpectraMagic (Minolta Corp., Ramsey, NJ, USA) was used to obtain parameters in terms of CIELAB L\* (degree of lightness), a\* (red/green) and b\* (yellow/blue) values. The effect of product variables on the color value is shown in the Table 3.7. The values of L\* ranged between 19.32 and 30.53, a\* ranged between 3.85 to 5.02, while b\* ranged between 8.81 and 10.67.

Food color and appearance of a snack is of utmost importance as it shapes the expectations and actual taste and flavor sensory characteristics of a food product. As stated by (DuBose et al., 1980) increasing color intensity of the solutions can give rise to more strong flavor, hence affecting overall flavor intensity. In a quantitative sense, color has the ability to make a product sweeter even if the sugar has been reduced or removed just by inducing the senses of a person as it has the ability to affect decisions (Clydesdale, 1993). Clydesdale (1984) reported that color is a very dominant factor in the perception of the sensory characteristics of a food product such as saltiness, sweetness and flavor.

# (a) L\* Value

Figure 3.5 shows the contour graph obtained by fitting special cubic equation to L\* value. For evaluating the equation, Analysis of Variance (ANOVA) was used. The lack of fit for the fitted model was insignificant (Table 3.8) which demonstrated that model was adequate. L\* is a measure of negative darkness and lightness and the product processed at high temperatures ought to be darker mainly due to Maillard reaction and non-enzymatic browning.

C was the only linear term showing significant effect on the L\* value of the extrudate. Also, the results from ANOVA demonstrated that L\* value was significantly affected by interactive effects such as that of rice-lentil, rice-chickpea, chickpea-tomato powder and rice-tomato powder (Table 3.9).

The regression equation which established the relationship between L\* value  $(Y_{L*})$  and independent variables in terms of actual values is as follows; special cubic model was selected for L\* value according to its statistical significance (Design Expert 9.0.4).

The  $R^2$  is 0.9402 and mean standard error is 3.45.

From the equation 3.13, it can be conferred that rice-lentil, rice-chickpea and rice-tomato powder interactions gave positive regression coefficient, whereas linear and cubic effects of product variables had negative regression coefficient which demonstrates opposite of the former. The increase in rice flour contributed to higher L\* value. The higher incorporation of lentil flour and tomato powder produced darker products. The value of L\* varied depending upon interaction effect of ingredients. Lowest value was obtained when tomato powder proportion was highest in the formulation. During extrusion, rice flour rich in starch gives free sugars due to starch hydrolysis. Fragments of dietary fiber, sucrose or products obtained due to hydrolysis of starch are available for Maillard reaction (Singh et al., 2007). Similarly, tomato powder is a high source of reducing sugar. (Davoodi et al., 2007) reported more than 50% of tomato powder on dry basis is sugars, most of which are reducing sugars. On the other hand, green lentil flour is rich in amino acid lysine as compared to cereal flours. As reported by



Figure 3.3 Mixture formulation effect on the value of L\* when Chickpea flour was at 20% in the proportion

O'Brien et al. (1989) lysine is the most reactive amino acid as it has two amino groups. As Maillard reaction is a reaction between reducing sugars and amino acids, increasing proportion of lysine can lead to more of Maillard browning as lysine being highly reactive complexes with reducing sugars vigorously. Maillard reaction results in a product with lower L\* value, which is why increasing lentil flour is decreasing the L\* value. However apart from that screw speed can also

play a major role as in this case the screw speed was 125 rpm, which increases residence time of the raw mixture and melt, making it more vulnerable to temperature induced color change or product expansion can also make the product darker. Additionally, tomato powder has a dense red color that can also effect overall brightness of product. Bhattacharya et al. (1997) in their extruded product of green gram and rice found the similar results. Similarly, Masatcioglu et al. (2013) reported decrease in L\* value with increase in tomato powder upto 8%. Hence, increasing amount of more reactive amino acids and increasing the exposure time will lead to darker products. Additionally, natural color of the ingredients used also play an important role on overall product color.

The model was significant with p = 0.0115 (<0.05) (Table 3.9). With R- squared value of 0.94 it can be conferred that 94% of the variability of the response could be explained by the model. The adjusted R-square of the model came out to be 0.81 and the adequate precision value came out to be 7.86 indicating that the model should be used to navigate the design space.

# (b) a\* value

The contour graph of a\* value is presented in Figure 3.4. a\* value provides the redness and greenness in product with redness as positive values and greenness as negative values.

In this study the value of a\* ranged from 3.85 to 5.02 (Table 3.7), which shows that product has more of redness in it even though it is dull not bright. The model suggested by Design-expert 9.0.4 was linear model and all the linear terms affected the value of a\* significantly. The value of  $R^2$  (Table 3.10) that explains the fit of the model came out to be 0.68. The lack of the fit for the model was insignificant, which therefore demonstrates that the model is adequate. The model gave adequate precision of 8.18 indicating that this model is good for usage in navigating the design space.

The regression equation obtained from Design Expert 9.0.4, which provides relationship between a\* value (Ya\*) and independent variables in terms of actual values is given below:

$$Ya^* = 0.0275643 * R + 0.0550799 * L + 0.0574035 * C + 0.0789005 * TP \qquad \dots (3.14)$$

The R<sup>2</sup> is 0.68 and mean standard error is 0.80. Linear model was chosen for evaluating the results.

The a\* value as can be seen from the contour graph is increasing with the increasing proportion of chickpea and green lentil flour. All the product variables showed positive linear regression coefficient. Enhanced a\* values were observed with increasing lentil flour and increasing tomato powder. The pigment lycopene present in tomato powder imparts redness to tomato powder and increasing tomato powder increased redness of the product. Even though the amount of tomato powder added was low as compared to other product variables but intensity of red color provided by tomato powder was high. Furthermore, Inclusion of rice as a cereal leads to increment of starch content which can hydrolyze and hence induce caramelization at high temperatures, as the product was processed at high temperatures the browning produced by caramelization can be the other reason of getting positive a\* value.



Figure 3.4 Mixture formulation effect on the value of a\* when Chickpea flour was at 20% in the proportion

# (c) b\* value

For the extruded products b\* value is also an important property to measure as it helps in determination of the desired yellowish color in the extrudate. The positive value of b\* shows yellow color in the extrudate while negative value shows blue color in the product. Figure 3.5 shown below provides the contour graph for b\*. The values for b\* ranged from 7.45 to 12.77 (Table 3.5).

The regression equation for b\* value (Yb\*) is given below, Linear model was suggested for a\* value according to Design Expert 9.0.4.

 $\begin{aligned} Yb^* &= -(0.0856297 * R) - (0.0926015 * L) - (0.257452 * C) - (8.25186 * TP) + (0.00560405 * R \\ &* L) + (0.0095028 * R * C) + (0.0955078 * R * TP) + (0.00869914 * L * C) + (0.0920028 * L * \\ &TP) + (0.0827063 * C * TP) & \dots \dots (3.15) \end{aligned}$ 



Figure 3.5 Mixture formulation effect on the value of b\* when tomato powder was at 3.5% in the proportion

The  $R^2$  for fitted model is 0.9148 and mean standard error is 1.72 is for the fitted model.

All the linear terms significantly affected (p<0.05) the b\* value (Table 3.9). Furthermore, ANOVA results showed that the interaction effects of R-C, R-TP, C-TP also had significant effect (p<0.05) on the b\* value. The regression coefficients of linear terms were all negative whereas the all the interactions showed positive quadratic regression coefficient.

From the contour graph (Figure 3.5), it was observed that with the increasing chickpea flour the value of b\* was also enhanced and the peak values for b\* were obtained when the percentage of chickpea flour in the product was highest. According to the results obtained high b\* value can be attributed to the natural yellow color which helped the product retain this color in the runs when

chickpea flour was in higher proportion. Also, the color of the extrudate might have brighten due to the formation of air cells in the product after expansion rather than dull color which is the other reason of enhancement in the b\* value due to chickpea flour. Shirani and Ganesharanee (2009) observed similar results with increasing chickpea flour in the product.

# **II. Expansion ratio (ER)**

Expansion ratio is property providing insights of crunchiness, puffiness, crispiness and water absorption. During extrusion water acts as a plasticizer helping the starch present in the raw material to undergo glass transition during extrusion. This water then exits through the die hole leaving air cells behind, giving a puffing effect to the extrudate. This puffing effect makes the structure of the product porous due to presence of air cells which therefore makes the product's texture more soft (Yu et al., 2012).

Statistical analysis of the data explained the fitness of linear model (Table 3.9). The lack of fit for the fitted model was insignificant demonstrating that the model employed is adequate. The relationship between Expansion Ratio ( $Y_{ER}$ ) and independent variables in terms of actual values is given in the following regression equation, linear model was suggested for Expansion Ratio according to Design Expert 9.0.4.

$$Y_{ER} = 0.0213008 * R + 0.016096 * L + 0.0148005 * C + 0.00850027 * TP$$
 ...... (3.12)

All the linear terms of the product variable showed significant effect (p<0.05) on ER values. The R<sup>2</sup> is 0.8199 and mean standard error is 0.023. The values of the expansion ratio ranged from 1.67 to 1.92 (Table 3.7), which indicates that there was not much difference between the ER of all the formulation which shows that with the increasing addition of pulse flours also the extrudates can be produced of having good puffing properties. The employed model was significant with p<0.0001. Adequate precision value of the model came out to be 13.44 which is very good value as the desirable value is greater than 4. Adequate precision value shows that the model can be used to navigate the design space.

From the results, it was concluded that all the variables showed a positive linear regression coefficient (Table 3.9). Furthermore, the results explain that with the increment of rice flour the expansion ratio had an enhancement while the increment in lentil and chickpea flours reduced the expansion of the product. Also, it is observed that the lowest expansion ratio was calculated at

highest levels of chickpea flour. Chickpea flour has highest fat content among all the cold legumes and fat can have greasy effect inside the barrel, reducing shear and friction and hence affecting starch gelatinization and expansion properties. Also, chickpea flour has lower starch content as compared to lentil decreasing the total starch content in the formulation. The observations were in agreement with results obtained by Frohlich et al. (2015) where chickpea flour inclusion reduced expansion by almost 30% as compared to extrudates produced by green lentil flour.



Figure 3.6 Mixture formulation effect on the expansion ratio when tomato powder was at 3.5% in the proportion

Overall reduction in expansion ratio is also because both the pulse flours have high proportion of fiber and protein in it. The fiber and protein has tendency to absorb water and can compete for water with starch (Camire and King, 1991). Now, water acts as plasticizer for starch melt and gelatinization of starch. Unavailability of water for complete gelatinization to starch can affect the structure formation and hence the expansion of the product also. Apart from that the starch is the basic structure of the extrudate in this case as it is a rice based product, the increasing incorporation of protein or fiber in the matrix hinders the structure formation which can then again reduce the products expansion. Also, a mixture of different types of starch itself can reduce the expansion ratio of the product. Chinnaswamy and Hanna (1988) in their study on amylose content of corn flour on extrudates found that different native starches of corn had different expansion at
different temperature points. The varying proportion of starch from different flours effects the amylose and amylopectin proportion in different runs hence producing different expansion ratios.

#### **III. Rehydration ratio (RR)**

The contour graph obtained by fitting model to RR value is shown in the Figure 3.7. Extruded products are many a times used as breakfast cereals consumed along with water or milk. Hence, rehydration ratio becomes an important property to measure. For RR, the quadratic model was found significant with p= 0.0011 (Table 3.8). Hence, the model was employed for analysis of variance. The value of rehydration ratio was found to be ranging from 59.89 to 167.56 (Table 3.7). The value of RR was affected significantly (p<0.05) by rice content of the product as well as by the interactive effects of R-L, R-C, R-TP, L-C and L-TP (Table 3.9).

The fitted regression equation in actual values for RR is as following:

$$\begin{split} Y_{RR} &= -4.96699 * R - 9.08301 * L & -14.1649 * C -107.618 * TP + 0.299884 * R * L + 0.378557 * \\ R * C + 1.38585 * R * TP + 0.241519 * L * C + 1.22547 * L * TP + 0.990264 * C * TP ...(3.13) \end{split}$$

The model was adequate to explain variability in the responses as R-squared value of the fitted model came out to be as 0.9402. The mean standard error 10.05 was. The Adequate Precision value of 12.440 is desirable value and indicates that that this model should be used to navigate the design space.

From the Figure 3.7, it was observed that higher rice content in combination with decreasing lentil flour yielded a product with higher rehydration ratio. The lowest rehydration ratio was observed with highest tomato powder in the formulation mix. Results were in agreement with the findings of Altan et al. (2008), where tomato powder decreased rehydration ratio of snack. Rehydration ratio of the product is effected by the starch content as well as that of protein and fiber. It is easier to rehydrate a product with more air spaces or air cells as water can easily impregnate and diffuse through these cells. Increasing protein and dietary fiber reduces expansion but apart from that the increase in dietary fiber reduces formation of air cells. The dietary fiber has a high affinity for water. At the die exit it can prevent water to leave the product, reducing formation of air cells which ultimately have negative effect on the rehydration ratio of the product. The correlation factor between expansion ratio and rehydration ratio was 0.62 which indicates the increasing rehydration ratio was due to increasing porosity of the snack.



Figure 3.7 Mixture formulation effect on rehydration ratio value when chickpea flour was at 30% in the proportion

## **IV. Bulk density**

Bulk density is also a very important property of a food product as it directly affects product's packaging volume as well as consumer acceptability. Hence, it affects the strategy for marketing of a product. Bulk density is a measure of the weight of the product divided by the total volume it occupies. Total volume for extruded products includes particle volume, inter-particle space volume as well as internal air pore volume. Hence, bulk density reflects the porosity of the snack. It is very much dependent upon the expansion ratio, but geometry of the product also plays a very big role in bulk density of product. The recommended method to measure the total volume of an extruded product is displacement method (Yu, 2011).

Figure 3.8 shows effect of mixture formulation on the value of BD for dried extrudes at 20% chickpea flour. The values obtained for BD under different mixture designs were presented in Table 3.7. The value of BD for the experimental product ranged from 0.45-0.65 g/ml. It was found that quadratic model is significant with p=0.0004 (Table 3.9). The BD values were affected significantly (p<0.01) by all the formulation types as well as by the interaction effect of R-TP and C-TP.

The relationship between Bulk Density  $(Y_{BD})$  and independent variables in terms of actual values is given in the following regression equation (3.14);

$$\begin{split} Y_{BD} &= (0.00642349 * R) + (0.00495479 * L) + (0.0123348 * C) + (0.234522 * TP) - (4.32739e-005 * R * L) - (0.000196938 * R * C) - (0.00252358 * R * TP) + (0.000100474 * L * C) - (0.00205235 * L * TP) - (0.00292793 * C * TP) & \dots (3.14) \end{split}$$

Design Expert suggested quadratic model for bulk density. The mean standard error value for the fitted model was 0.04. The R-squared value was 0.9091, which explains that model was adequate to explain variability in the responses. The lack of fit for the fitted model was not significant which demonstrates the adequacy of the model and the adequate precision value of 10.85 demonstrates that the model should be used to navigate the space.

The regression equation (3.14) indicates that while each of the linear variables showed a positive quadratic regression coefficient, the interactive effects showed negative quadratic regression coefficient except interactive effect of L-C. The analysis of the data revealed that bulk density decreased with increasing rice flour and tomato powder in the product, while increment in both lentil and tomato powder increased the bulk density of the product. The expansion ratio showed negative correlation with bulk density with correlation = -(0.72). Although, shape or geometry of the product as well as difference in mass of different constituents of the mix, effects bulk density a lot, but still puffiness or size of the product is a very big factor that effects the bulk density. As is evident from the results also. The bulk density decreased with the increasing expansion ratio. Hence, bulk density depended significantly on the size of the product. Increasing fiber or protein content in the formulation decreased the expansion ratio of the product because of reduction of air cell size, number of air cells or because of incomplete gelatinization of starch. The extent of gelatinization of starch effects the bulk density of the product (Yu, 2011). Apart from that the presence of additives such as salt can also decrease the expansion, adding to the bulk density of the product by reducing gelatinization by providing competition to starch for water. Dietary fiber not only reduces gelatinization but also results in the thickening of the cells walls decreasing the air cell in the microstructure of the product (Jin et al., 1995). The findings of the present study are in agreement with the other studies as well where increase in BD was observed for decreasing ER (Wójtowicz et al., 2013; Jin et al., 1995; Hsieh et al., 1991). However, there are some studies where increasing fiber or protein did not have much effect on the bulk density of the

product. Hence, the bulk density of the product depends mainly on the product formula (type and amount of protein, starch, fiber, etc.), as well as the processing conditions (Schoenfuss et al., 2013).



Figure 3.8 Mixture formulation effect on BD when chickpea flour was at 20% in the proportion

## V. Breaking stress (BS)

The contour graph given in the Figure (3.9) shows the influence of ingredient proportion on the value of BS. The proportion of rice four, lentil flour in the formulation and the interaction effect of rice and lentil flour significantly affected (p<0.05) the breaking stress. The regression equation for breaking Stress (Y<sub>BS</sub>) and independent variables in terms of actual values is given:  $Y_{BS} = (0.0154046 * R) + (0.0273724 * L) + (0.0170435 * C) + (0.0560637 * TP) - (0.000652098 * R * L) - (0.000609043 * R * C) - (0.000821134 * R * TP) - (3.65036e-005 * L * C) - (0.00096757 * L * TP) + (0.000342951 * C * TP)$ 

Design expert suggested that quadratic model to explain the effect of variables on breaking stress. After fitting the quadratic model the value of  $R^2$  came out to be as 0.9200 which indicates that the model explained variability of the response very well. The mean standard error for the fitted model was 0.05 (Table 3.10). The non-significant lack of fit value demonstrated the adequacy of the model. The adequate precision value for the model was 11.146. For the analysis

of variance (ANOVA) it was found that quadratic model is significant with p=0.0002 (Table 3.9). The value of BS ranged from 0.28 N/mm<sup>2</sup> to 0.69 N/mm<sup>2</sup> (Table 3.7). From Figure 3.9, it was observed that lower values of breaking stress were obtained with high rice flour content and low lentil flour content whereas increasing tomato powder increased the breaking stress of the product. Increasing breaking stress means the product is becoming more hard and more force is required to bring the first break in the material. The analysis of quadratic regression equation disclosed that each process variable was positively related to BS, whereas the interactive effect of the variables was negatively related to BS except the interaction of C-TP.



Figure 3.9 Mixture formulation effect on BS when chickpea flour was at 20% in the proportion

Dietary fiber (DF) inclusion in the formulation leads to increases in product density as well as hardness. It thickens the cell wall (Rinaldi et al., 2000). As DF affects the expansion of the product and makes product denser along with thickening of cell wall it increases breaking strength by increasing the force required to break the product. During the extrusion cooking expansion depends on entrapment of water vapor by starch matrix. However, high moisture can change molecular structure of amylopectin reducing elastic viscosity and hence effecting its expansion in negative manner (Ding et al., 2005). Higher break strength can also be because of the inclusion of flour containing higher protein content as that also effects viscoelastic behavior of starch reducing air bubble and making it denser (Taverna et al., 2012). An important factor to note here is crystal formation of starch which can increase the rigidity of the product adding to its break strength. After extrusion and air drying, proper time is given to extrudate for cooling down. Biliaderis (1991) mentioned that chain aggregation and recrystallization of starch molecules takes place during storage and crystallization of amylopectin is rapid during cooling and rate decreases with storage time. Mestres et al. (1988) in his study gelation and crystallization of maize starch reported that co-crystallization of both amylose and amylopectin takes place after extrusion cooking occurs and adds to the rigidness of the product.

## 3.3.4 Antioxidant activity

Antioxidant activity of the dried extrudates was calculated using DPPH scavenging method. DPPH is a simple and effective method of measuring antioxidant activity apart from being cost effective also. Trolox was used as a standard to calculate the value of AA. The beneficial effects of antioxidants on human health has been deeply studied. They help to protect body against damage causing reactive oxygen species and nitrogen and chorine and ameliorate human health (Shahidi, 2000).

Raw material	AA (μmol TE/ 100 g)
Rice Flour	14.89±4.26
Lentil Flour	186.62±3.88
Chickpea Flour	82.01±5.83
Tomato Powder	810.14±4.56

Table 3.11 Antioxidant activity values of raw materials used in the formulations

The values of the Antioxidant activities are given in the Table 3.10 below. The antioxidants were successfully incorporated into the product increasing the antioxidant activity of extrudate. The highest antioxidant activity recorded in the products was given by run 7 with antioxidant activity as 114.48 µmol TE/100 g. Although a decrease in the antioxidant activity of dried extrudates was observed after extrusion-drying of extrudates, the incorporation of tomato powder and lentils helped increase in the overall antioxidant activity of product. The decrease in the antioxidant activity ranged from 20-40% of the original values of antioxidant activity of raw flour formulations.

Run	AA (µmol	%
	TE/ 100 g)	change
1	84.07±4.31	30.17
2	58.52±3.18	34.19
3	58.64±4.39	30.17
4	63.26±4.59 28.23	
5	74.16±3.06	30.27
6	68.06±5.09	36.35
7	$114.48 \pm 3.91$	27.50
8	109.91±2.97	30.40
9	79.88±3.09	27.93
10	86.66±5.77	25.79
11	94.57±5.21	24.37
12	46.10±3.57	36.95
13	91.88±4.98	23.01
14	46.56±4.82	36.32
15	91.07±6.67	22.01
16	67.53±3.86	36.85
17	83.58±5.01	30.58
18	$101.18 \pm 5.11$	25.06
19	93.09±5.17	26.53
20	44.39±3.77	36.01

## Table 3.12 Antioxidant activity values of different experimental runs after extrusion drying

Table 3.13 ANOVA results for AA of different formulation runs

Source	AA of formulations		
	df	SOS	
Model	3	8130.98	
Residual	16	97.78	
R <sup>2</sup>	0.9881		
Standard Error	4.42		

From the Table 3.12, it can be concluded that the amount of change in antioxidant activity varies with changing flour formulation. Furthermore, contour graph (Figure 3.10) shows that higher tomato powder incorporation produced snacks with more antioxidant activity. Table 3.11 shows that tomato powder exhibits the highest antioxidant activity whereas rice flour exhibits the lowest antioxidant activity. Lentils are also a good contributor to the antioxidant activity of the dried extrudates. Total phenolic compounds found are considered good constituents responsible for antioxidant activity, especially in case of lentils the major antioxidant properties come from phenolic compounds rather than tocopherols or ascorbates present in it (Prior et al., 1998; Fernandez-Orozco et al., 2003). Han and Baik (2008) reported that lentils have a high phenolic content. Tannins constitute a considerable amount of share in the phenolic composition of lentils and exhibit antioxidant properties (Amarowicz et al., 2010).



Figure 3.10 Mixture formulation effect on the AA (µmol TE/ 100g db) of extrudates when chickpea flour was at 30% in the proportion

 $Y_{AA} = 0.110292 * R + 1.17924 * L + 0.553679 * C + 7.07178 * TP$  ...... (3.16)

For tomato powder lycopene is considered the main constituent imparting antioxidant properties. Lycopene has a high antioxidant activity and have highest physical quenching rate for a singlet oxygen (Di Mascio et al., 1989). Shi and Maguer (2000) reported that lycopene is stable at high temperatures and even heating tomato to 130°C for 1 min can only bring 7% destruction in lycopene content. The time temperature combination used to produce extrudates was very low and drying of the extrudate was carried out at 55°C, which could have decreased the losses of lycopene content and hence, reducing extent of destruction of antioxidant of extrudate. The effect of extrusion on antioxidant activity is variable and it depends upon processing conditions as well as the constituents of formulation. In the published literature, loss of antioxidants after extrusion ranges from 7-25% (Mora-Rochin et al., 2010) upto the loss of 60-68% (Altan et al., 2009). Also, loss of antioxidants with increasing barrel temperature has found to be uneven because of formation of Maillard compounds at higher temperature which can also show antioxidant properties (Gumul and Korus, 2006). The increasing screw speed as well as decreasing moisture can also increase loss of antioxidants. In this research, the loss of antioxidants was uneven ranging from 20% to 35% as compared to raw mixture formulation. Also, it was observed that loss is higher

in formulation without tomato powder containing lycopene, which has higher resistance to heat degradation as compared to phenolic compounds responsible for antioxidant activity in lentils. Hence, tomato powder not only increased the total antioxidant activity of the product but also reduced losses during extrusion drying.

## **3.3.5** Antinutrients

Lentils and other legumes have considerable amounts of antinutritional factors present which can have negative effect on the availability of nutrients to our body hence, reducing the significance of producing a nutrient rich snack (Sgarbieri and Whitaker, 1982). Tannin content present in food can reduce availability of protein to body by complexing with amino acids (Davis, 1981). Similarly, phytic acid can reduce bioavailability of certain minerals especially calcium (Vidal-Valverde et al., 1994). Hence, it is necessary to determine the amount of antinutrients present in the extrudate prepared using pulse flours.

Table 3.14 Tannin content and Phytic acid content of raw materials used in the

formulations

Raw material	Tannin content (mg CE/100g db)	Phytic acid content (g/ 100 g db)
Rice Flour	$0.1 \pm 0.03$	$0.22\pm0.05$
Lentil Flour	$1.97\pm0.05$	$1.38\pm0.03$
Chickpea Flour	$0.95 \pm 0.06$	$0.85\pm0.06$
Tomato Powder	$1.05\pm0.03$	$0.67 \pm 0.05$

## Table 3.15 ANOVA results for Tannin and Phytic acid content of different formulation

Source	Tannin content of formulations		Phytic acid content	t of formulations
	df SOS		df SOS	
Model	3 0.010		3 3.271E-003	
Residual	16 9.106E-003		16 1.384E-003	
R <sup>2</sup>	0.5287		0.7026	
Standard Error	0.04		0.02	

#### runs

Table 3.16 Tannin content and Phytic acid content values along with ±standard deviation

Run	Tannin content (mg CE/100g db)	Phytic acid content (g/ 100 g db)
1	$0.15{\pm}0.04$	$0.07{\pm}0.02$
2	$0.14{\pm}0.03$	$0.06{\pm}0.03$
3	$0.18{\pm}0.08$	$0.05{\pm}0.01$
4	0.1±0.01	$0.05{\pm}0.02$
5	$0.14{\pm}0.05$	$0.06{\pm}0.01$
6	$0.19{\pm}0.06$	$0.09{\pm}0.04$
7	$0.18{\pm}0.04$	$0.08{\pm}0.03$
8	$0.18{\pm}0.05$	$0.07{\pm}0.02$
9	$0.14{\pm}0.05$	$0.05{\pm}0.02$
10	0.11±0.06	$0.05{\pm}0.03$
11	$0.12{\pm}0.04$	$0.04{\pm}0.01$
12	0.13±0.03	$0.06{\pm}0.01$
13	$0.07{\pm}0.05$	$0.06{\pm}0.02$
14	$0.09{\pm}0.02$	$0.04{\pm}0.02$
15	0.11±0.05	$0.05{\pm}0.01$
16	0.13±0.04	$0.09{\pm}0.04$
17	0.13±0.02	$0.07{\pm}0.03$
18	$0.12{\pm}0.01$	$0.07{\pm}0.02$
19	0.12±0.03	$0.08{\pm}0.02$
20	0.11±0.04	$0.04{\pm}0.01$

for different experimental runs after extrusion drying

## 3.3.5.1 Tannin content (TC):

In Figure 3.11, the effect of feed formulations on values for tannin content is graphically presented. It can be observed from the Table 3.14 that tannin content was reduced by 80%-90% as compared to the raw flour formulation mixtures. Lentil flour had the highest tannin content among the raw materials used (Table 3.15). However, the values of tannin content in all the experimental runs was found to be below 0.19 mg CE/ 100 g db, which is a considerably very low level of tannin to effect bioavailability of proteins. Hence, extrusion cooking followed by air drying of the products successfully reduced tannins present in extrudates. With high heat treatment accompanied by shear forces the tannins can undergo intense polymerization (Van der Poel et al., 1991). Also, high temperature processing can lead to decrease in chemical reactivity or destruction of the tannin molecule (Barroga et al., 1985). The findings of the study were in agreement with the studies of Rathod and Annapure (2016) and Alonso et al. (1998) for the extrusion processing of pulse flours done at 140° C.



Figure 3.11 Mixture formulation effect on the Tannin content of extrudates when TP was at 3.5% in the proportion

Regression equation (3.16) indicating relationship between Tannin content and independent variables in terms of actual values is given below:

 $Y_{TC} = 0.000297742 * R + 0.00274108 * L + 0.00154264 * C + 0.000504034 * TP \qquad \dots (3.17)$ 

## 3.3.5.2 Phytic acid:

Phytic acid content of each formulation run is given in Table 3.14, while Table 3.15 gives phytic acid content of raw flour formulation mixtures. From Table 3.14, it can be observed that significant reduction in the composition of phytic acid in dried extrudates were obtained, increasing the bioavailability of certain minerals. Comparing results from Table 3.15, it was found that phytic acid content was highest in lentil flour. After extrusion drying destruction of phytic acid by almost 90%-94% was achieved. Alonso et al. (1998) reported that high temperature can lead to hydrolyzation of the inositol hexaphosphate to penta and tetraphosphates. Also, Rathod and Annapure (2016) reported the same results and observed that destruction of antinutrients is higher at higher moisture content.

Regression equation (3.18) indicating relationship between phytic acid content and independent variables in terms of actual values is given below:

 $Y_{PC} = 6.22532e-005 * R + 0.00151399 * L + 0.000405315 * C + 0.000774916 * TP \dots (3.18)$ 



Figure 3.12 Mixture formulation effect on the phytic acid content of extrudates when TP was at 3.5% in the proportion

## **3.5 Conclusions**

The extrusion product variables viz. rice flour (R), green lentil flour (L), chickpea flour (C) and tomato powder (TP) were found to have significant effect (p<0.05) on the drying behavior, product's moisture content after extrusion, water activity as well as physicochemical properties of the extrudate. Drying to achieve water activity ( $a_w$ ) of 0.75 was affected significantly by the individual proportions of all the above-mentioned product variables. The drying time was affected most by lentil flour whose increase in the formulation decreased value of diffusion coefficient and increased drying time.

The product variables and their interactive effects significantly influenced (p<0.05) physical properties (BS, ER, BD, RR and color) as well as on antioxidant activity and antinutrient content (tannin and phytic acid) of the dried extrudates. Effect of increasing C was seen on the ER as well as b\* value of the dried extrudates as higher b\* value was obtained, producing snack of acceptable color, however ER decreased with increasing amount of C in the formulation. The increasing incorporation of L and TP in the formulations produced higher BD and BS. R increased the value of ER, RR and L\* in the product, while decreasing the value of BS. In addition, TP had high effect on increasing the value of a\* in the product. More importantly higher lentil flour increased the

protein content of the snack. The antioxidant activity of snack was enhanced significantly with incorporating tomato powder and lentil flour with value ranging from 44.39  $\mu$ mol TE/ 100 g db to 114.48  $\mu$ mol TE/ 100 g db. The processing of raw formulations with extrusion followed by drying reduced the tannin and phytic acid content in the extrudate by about 80-90% and 90-95% respectively as compared to the amount of antinutrients present in raw formulations. In all nutritious snack with a good shelf life was produced which can have health enhancing effect. The study was helpful in identifying effect of each product variable on physical properties of such a snack. It is necessary to enhance these physical properties to increase desirability of product with post extrusion processing.

## **PREFACE TO CHAPTER 4**

In the previous Chapter 3, a high protein snack with good amount of antioxidants was developed using rice-green lentil-chickpea flour and tomato powder. The effect of proportions of formulation was identified on the physical characteristics of the product. Extrudates collected after extrusion had high water activity because of moisture content. The products were subjected to drying and influence of product variables on drying kinetics was also identified. Even though after drying the products had low moisture content for enhanced shelf life, but they did not posses desirable sensory properties for its direct consumption. Therefore, in Chapter 4 the focus was to enhance sensory properties of extrudates.

The sensory properties of extrudates were enhanced by using frying or microwave roasting techniques. The effect of processing techniques on physical properties and antioxidants present in extrudates is evaluated. The products obtained from both processing techniques are compared with the help of sensory evaluation (Quality rating test).

All the experimental work and data analysis of the responses obtained was conducted by the candidate under supervision of Dr. H. S. Ramaswamy. A part of this research was presented at Food processing research mini conference at Montreal. One manuscript from the chapter is being prepared for publication.

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## EFFECT OF FRYING VS MICROWAVE ROASTING ON PHYSICO-CHEMICAL PROPERTIES OF DRIED EXTRUDATES PREPARED FROM RICE BASED BLENDS

#### Abstract

In this study, effect of frying or microwave roasting on physical properties and antioxidant activity of dried extrudates was evaluated. The acceptability of snack was evaluated through sensory properties. Extrudates dried to 18% (db) moisture content were subjected to deep frying at 200°C temperature for 70 s in canola oil or microwave roasting for 50 s in a 1000 W domestic MW oven at 75% power level. The post drying processing of frying or microwave roasting decreased moisture content of extrudates below 5% (db) in all the extrudates. Model generated response surface plots and ANOVA were employed to determine the significance of individual and interaction effects of product variables on physical properties of extrudate such as color (L\*, a\*, b\*), expansion ratio, breaking stress and bulk density as well as on antioxidant activity. The results showed that increasing chickpea flour and lentil flour decreased expansion ratio, and increased the breaking stress of the product. However, inclusion of chickpea flour helped to produce snacks of golden yellow color increasing its acceptability. Both the processing techniques reduced antioxidant activity, however MW roasting retained more than 80% of the extrudate antioxidants. The expansion ratio and L\* value had a strong positive correlation with overall acceptability of products. MW roasting was found to be the preferred processing technique for producing products with better nutritional and sensorial properties. The results demonstrated that extrusion drying followed by MW roasting can help produce an acceptable snack with high protein content enriched through pulses and antioxidants enriched through tomato powder which has the ability to ultimately increase consumption of underutilized plant nutrients.

#### 4.1 Introduction

Snacking is the practice of consumption of food in episodes between the three major meals of the day. However, nowadays this practice of consumption has become so frequent and significant that sometimes it is even replacing major meals of the day (de Graaf, 2006). Most of the snacks available in the market are high energy snacks (usually 400-500 Kcal/100 g) and contain lower amount of essential nutrients required by our body for growth (Table , 1993). Hence, production of nutrient rich snacks with better sensory acceptability is needed. Extrusion cooking is a high shear, high temperature and high pressure, short time cooking process and has been in use since a

long time contributing to a large share of snack market (Riaz, 2000). However, success of these snacks produced with the help of extrusion depends on multiple variables that needs appropriate adjustments to give the snack desired physical conditions and chemical properties within the extruder barrel (Gill, 2014). After determination of independent variables and dependent variables along with its appropriate levels for the product type, they should be kept closer to the optimum levels so as to make sure that variables of extrudate are also kept near to levels which are desired (Guy, 2001). It is not necessary that the products that we get after extrusion drying have the desired sensory characteristics, moreover such dried extrudates have advantage of easy and longer shelf life and can be produced with required quantities whenever desired (Van Laarhoven and Staal, 1991). Hence, post extrusion processing (frying or microwave roasting) is employed to make desirable physical, chemical, and sensorial changes to increase its acceptability.

Ingredients viz. green lentils and chickpeas are cool season legumes and are grown in Canada on large scale. Especially for lentils Canada has now become the largest exporter, with 75% of the total lentil production accounted by green lentils only (Watts, 2011; PulseCanada, 2007). Both green lentils and chickpeas have many health promoting benefits like prevention of cardiovascular disease (Bazzano et al., 2001; Anderson et al., 1999); cancer prevention (Mathers, 2002); lowering of glycemic index and preventing diabetes (Pittaway et al., 2008). Chickpea flour is a yellow colored flour used in traditional Indian snacks because of its characteristic flavor (Geervani, 1989). Chickpea flour and lentil flour are not only a rich source of proteins but also provide significant amounts of vitamins and minerals. Mudryj et al. (2012) reported that even ½ a cup of pulses can help increase nutrient intake of proteins, thiamin, vitamin B<sub>6</sub>, folate, Iron, magnesium, phosphorus, and zinc by substantial amounts. Chickpea as compared to other pulses is a rich source of essential fatty acids and hence incorporation of both pulse flours will help in production of a nutrient dense snack. Proteins are not only required for increasing the nutrition of snack but they also play an important role in flavor formation through reaction with reducing sugars and giving Maillard browning compounds.

Reactions or modifications during high temperature involve hydrogen bonding, complexes between amino acids and reducing sugars as well as covalent bonding among amino acids itself, which lead to production of browning as well as flavor development. Apart from formation of protein complexes and protein denaturation, starch gelatinization is an important process of snack production process. Starch, which forms the main structure of snack is responsible for holding water and providing puffiness through expansion in volume (Rossell, 2001). The Starch matrix and protein-starch structure produces large variations in quality of product with crisp texture development, color and by governing oil uptake during frying of product. For determining the acceptability of snack produced, sensory test is widely used in food industry and is considered core of quality control program. Not only it provides guidance for product development but also help in improvement, optimization and maintenance of that product (Resurreccion, 1997).

It has been proposed that a rice based product prepared using extrusion followed by drying and microwave roasting can be a very popular choice and has the ability to mimic even a potato fry (Zukerman and Zukerman, 1988; Homnick and Huxsoll, 1973). During extrusion drying processing followed by frying or microwave roasting enables a lot of variables to interact together and hence outcomes might vary with different product as well as process variables. The effect of post extrusion processing viz. Frying versus microwave roasting in physical and sensorial properties as well as on antioxidants present in a rice based pulse flour and tomato powder enriched snack has not been studied yet. Hence, the focus of this study was to determine the influence of frying vs microwave roasting parameters on physical and sensory properties and to study effect of both processing techniques on retention of antioxidants in a snack prepared from rice based blends of green lentil flour, chickpea flour and tomato powder and evaluate and optimize the processing conditions (temperature-time combination for frying and power-time combination) to produce a snack with acceptable quality.

## 4.2 Materials and methodology

#### 4.2.1 Materials

Rice flour (R) and green lentil flour (L) were purchased from a local store Bulk Barn located in Montreal, Canada (with rice flour composition mentioned by the manufacturer as protein: 5 %, fat: 1.25%, carbohydrate: 80% and dietary fiber: 2.5% and green lentil flour with manufacturer composition as protein: 36 %, fat: 1.25%, carbohydrate: 60 % and dietary fiber: 2.5%). Chickpea flour (C) was purchased from Well Canada, Ontario with the manufacture composition of the floor as protein: 20%, fat: 6.66%, carbohydrate: 60% and dietary fiber: 16.65%. Tomato Powder (TP) was purchased from Z natural foods (Florida, USA) having labelled composition as protein: 13%, fat: 0%, carbohydrate: 75% and dietary fiber: 16%. For frying the extrudates, canola oil available commercially was used.

#### **4.2.2 Preparation of dried extrudates**

As specified in the previous chapter, the study of the 20 formulations (experimental conditions) given by D-optimal mixture design using the software Design Expert Version 9.0.3 State-Ease, Inc., Minneapolis, MN) with four variables whose values were set using predetermined levels. The independent variables used in this study were rice flour (R), lentil flour (L), chickpea flour (C) and tomato powder (TP). For minimizing the effect of experimental errors replicate points were included and experiments were performed in a random manner. Hobart mixer (Hobart Food Equipment Group Canada, North York, ON) operating at medium speed was used to mix the flours. Water and salt was added during the process of flour mixing. After mixing, the blends were allowed to sit for the whole night for the moisture to mix uniformly all over the blend.

Twin screw extruder (DS32-II, Jinan Saixin Food Machinery, Shandong, P. R. China) was used to carry out extrusion process. The extruder barrel consisted of three independent zones of controlled temperature. The diameter of the screw was 30 mm and the length to diameter ratio of screw 20:1. The diameter of the hole in the die was 5 mm with 27 mm as the length of the die. The extruder had the provision of controlling the temperature as well as screw speed according to desired levels. Raw material feeding was done manually to reduce any accumulation of feed in screw flights. As the conditions of flow through stabilized, extrudates were collected at the die exit and were cooled under mild air flow conditions at room temperature overnight and then further drying of the process was carried out at 55° C with the flow of air maintained at 0.1 m/s to get a final moisture content of 18% (dry basis). After drying the samples were put in air tight containers and were stored in a dark place at room temperature for further analysis.

## 4.2.3 Processing given post extrusion-drying for enhancing sensory properties

## (i) Frying:

There have been only limited studies measuring the effect of post extrusion process of frying on the pulse flour enriched with rice based dried extrudates. To carry out the frying process of the extrudates, they were cut in cylindrical shapes of 5 cm length and then subjected for frying under different time-temperature combinations (Table 4.1). For the process of frying a digital pot fryer (T-Fal FR4017 Deep Fryer, NJ) was used for controlling the temperature at desired value. The pot was filled with 2 L of canola oil for frying. The fryer was provided with a wire mesh made of metal to hold the products. Before the start of frying process canola oil was allowed to stabilize at the desired level of temperature and frying was done in small batches of the product. The products were withdrawn after every 10 seconds starting from 30 seconds to 90 seconds. After removal of products excess oil was drained out and paper towel was used for blotting and samples were allowed to reach to an equilibrium and then transferred to a plastic bag so as to carry out further analysis.

Table 4.1 Trial conditions for optimizing frying parameters

Trial No.	Frying Temperature (°C)	Frying Time* (sec)
1	160	30-90
2	180	30-90
3	200	30-90
4	220	30-90

\* = Effect of frying was studied for every 10 s time interval from 30 s to 90 s

## (ii) Microwave roasting:

Frying has been considered to increase the calorie content of the food because of the inevitable oil taken up by the food during frying (Fillion and Henry, 1998). Hence, it is important to study the effect of alternative technique of microwave roasting on dried extrudates. The main aim is to produce desired results by optimizing the combinations of power level of microwave and time required for processing of the extrudates. Hence, for carrying out the process the extrudates were first cut into 5 lengths and then given conditions (Table 4.2) of power- time were applied. For achieving the processing, a 1000 W microwave oven was used and samples were drawn at regular intervals of 10 s starting from 20 s to 90 s.

	Table 4.2 Trial	conditions for	<sup>,</sup> optimizing	microwave	roasting	parameters
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Trial No.	Power level (%)	Time* (sec)
1	100	20-90
2	75	20-90
3	50	20-90
4	25	20-90

\* = Effect of microwave roasting was studied for every 15 s time interval from 30 s to 120 s

## 4.2.4 Measurement of physical characteristics

## 4.2.4.1 Color

Same as in section 3.2.9

#### 4.2.4.2 Expansion ratio

Same as in section 3.2.11

#### 4.2.4.3 Rehydration ratio

Same as in section **3.2.12** 

## 4.2.4.4 Breaking Stress

Same as in section 3.2.10

## 4.2.4.5 Antioxidant activity

Same as in section 3.2.13

## 4.2.4.6 Sensory Evaluation

10 untrained panelists were chosen for evaluation (five males and five females with age between 20 and 35 years) of selected characteristics with the help of quality rating test on a 7-point scale (1=very bad, 2=bad, 3= below fair-above bad, 4=fair, 5= below good- above fair, 6=good, 7 = very good). The measured characteristics of tests were: color, crispiness, oiliness and overall quality.

For sensory evaluation of the product, the frying condition of temperature of 200 °C for 70 seconds was selected. All the samples were withdrawn after 70 seconds of frying process whereas for microwave roasting of extrudates, the samples were roasted in a microwave for 50 seconds at the power level of 75%. Samples were prepared in duplicates. Evaluation of samples was done in random sessions with evaluation of 10 samples in each session (5 samples of deep fried extrudate and other 5 samples were of microwave roasted samples). Panel of sensory evaluators was provided with tea so as to cleanse the palate between tasting of the extrudates. The extrudates were processed for 48 h before they were provided to panelists for their evaluation.

## 4.2.4.6.1 Sensory evaluation scorecard

For evaluating the sensory properties, a sensory evaluation scorecard was given to the panelists to record their data:

# Sensory evaluation scorecard

Name:		Date:
Product: Rice	based extruded snack	
Ingredients: F	Rice Flour, Green lentil flour, Ch	ickpea flour, Tomato powder, Salt, Water and
Canola oil.		
Instructions:		
*	Note the name of the sample an mark(s) in appropriate box us characteristics of snack in your of	d then taste the sample. After that record your ing given scale which describes the sensory opinion.
*	You can also write down your c attributes.	comments in the space provided along with the
*	For cleansing the palate after each drink tea to avoid the carryover as much as you like, and sample	ch sample, tea is provided and it is suggested to effect of any previous snack. You can drink tea s can also be tasted again.

Color	Crispiness	Oiliness	Overall Acceptability
(Dark or dark brown or	(Hard or crispy)	(High oil or less oil)	(Consumable or non-
light yellow)			consumable)

Very good	good	Below good- above fair	fair	Below fair - above bad	bad	Very bad
7	6	5	4	3	2	1
Score			Samp	ole code		
	GF1	DS2	FT3	DC1	EH2	JD3
Color						
Crispiness						
Oiliness						
Overall Acceptability						

## **4.2.4.7** Correlation coefficient (r)

Any scientific journal cannot be reviewed without analyzing and statistically interpreting the research data. The use of statistical numbers and terms so as to minimize understandings of the concept of statistics has long been in use. Use of statistical methods that involve correlation analysis in which a coefficient of correlation which represents the linear degree relationship between two variables has been in use (Taylor, 1990). Pearson's correlation coefficient can be defined as the measure of the strength between linear relationship and paired data. Let the two variables be A and B, with each of the variables having n values A1, A2, ..., An and B1, B2, ..., Bn respectively. Let the mean of A be  $\overline{A}$  and the mean of B be  $\overline{B}$ . Then the Pearson's correlation coefficient r is:

$$r = \frac{\sum (A_i - \overline{A}) (B_i - \overline{B})}{\sqrt{\sum (A_i - \overline{A})^2 \sum (B_i - \overline{B})^2}} \qquad (4.1)$$

it is denoted by "r" in a sample and it's constrained as follows  $-1 \le r \le 1$ .

## 4.3 Results and Discussion

## 4.3.1 Fried extrudates

Deep fat frying has been one of the oldest methods of preparation of food products. In this method food is immersed deep in the hot oil. It is helpful in imparting a unique texture and flavor to food and hence enhancing a snacks sensorial acceptability (Yu et al., 2013).

As the temperature of oil is too high, heat is transferred via oil and the moisture contained in the sample evaporates and leads to drying at the same time. So, there is simultaneous heat and mass transfer going on in the frying. Along with that chemical reactions like Maillard reaction and caramelization imparts color and better texture to the food (Moreira et al., 1999). The frying of a product is usually done at temperatures of 160 to 180 °C and hence because of such a high temperature the removal of moisture from food is also at a very high rate (Baumann and Escher, 1995). However, in a product system having high protein and starch content the significance of frying is also increased because of the high chances of Maillard reaction, especially when protein is coming from pulses with high amounts of lysine present.

Run	Expansion	L*	a*	b*	Breaking stress	Rehydration ratio
	ratio				(N/mm <sup>2</sup> )	(%)
1	1.98±0.09	9.94±2.57	9.02±0.53	-1.38±1.14	0.42±0.05	102.36±2.67
2	2.07±0.05	15.3±3.98	7.73±0.57	2.73±1.09	0.26±0.06	149.58±3.89
3	2.07±0.06	11.36±3.09	8.83±0.62	1.97±1.43	0.32±0.04	114.31±5.27
4	2.21±0.05	17.89±4.98	7.27±0.57	2.56±1.00	0.23±0.05	149.37±3.95
5	2.07±0.05	10.94±2.95	8.69±0.79	0.78±0.43	0.37±0.06	110.94±3.04
6	2.01±0.05	9.47±2.09	8.63±0.48	-1.46±0.95	0.38±0.07	97.78±4.94
7	1.97±0.06	10.27±2.78	8.27±0.28	-2.35±0.34	0.42±0.03	95.39±4.02
8	2±0.04	9.72±1.97	8.74±0.56	-0.73±0.45	0.47±0.05	101.31±4.48
9	2.09±0.05	13.36±4.09	8.21±0.87	1.8±1.09	0.29±0.06	150.25±3.98
10	2.1±0.03	14.51±4.87	7.63±0.23	2.88±1.08	0.28±0.05	135.42±4.92
11	2.04±0.10	$11.91 \pm 2.98$	8.96±0.54	0.85±1.09	0.36±0.04	117.81±4.65
12	2.08±0.11	13.47±3.09	7.89±0.64	2.78±0.34	0.27±0.05	135.83±4.98
13	2.11±0.09	16.83±4.69	8.26±0.53	2.02±1.01	0.23±0.06	145.32±4.90
14	2.1±0.06	14.72±5.67	8.34±0.87	4.34±2.09	0.25±0.04	141.9±6.94
15	2.14±0.06	16.04±5.20	7.92±0.75	5.16±2.09	0.23±0.03	145.6±5.78
16	$1.98{\pm}0.04$	10.35±2.93	8.23±0.54	-2.18±0.98	0.41±0.04	93.21±6.38
17	2.01±0.06	9.01±2.76	8.94±0.75	-1.72±0.88	0.38±0.03	97.32±6.86
18	2.03±0.05	14.09±3.89	8.39±0.24	1.09±0.76	0.34±0.05	132.23±5.09
19	2.07±0.06	13.52±3.09	8.35±0.65	-1.66±1.09	0.37±0.05	113.27±8.011
20	2.13±0.06	14.73±5.87	8.17±0.45	2.32±1.21	0.28±0.06	142.48±4.73

Table 4.3 Responses obtained for physical characteristics of fried extrudates

In the present study, the frying process was employed to increase the acceptability of the product by imparting better sensorial attributes. Significant changes were observed after frying the extrudate. The data on the values of physical properties [breaking stress (BS), expansion ratio (ER), rehydration ratio(RR) and color (L\*, a\*, b\* values)] against the experimental arrangements has been provided in the Table 4.3. Physical properties were then analyzed with the help of ANOVA. For discussion, the properties of the product were divided into two subcategories: texture and color.

Responses	Source	Sum of squares	df	Mean Squares	F- value	P- value
	Regression	125.78	9	13.98	17.15	< 0.0001
	Lack of fit	5.22	5	1.04	1.79	0.2696
L*	Pure error	2.92	5	0.58		
	Residual	8.15	10	0.81		
	Total	133.92	19			
	Regression	3.79	9	0.42	8.56	0.0012
	Lack of fit	0.15	5	0.031	0.46	
a*	Pure error	0.34	5	0.067		
	Residual	0.49	10	0.049		
	Total	4.28	19			
	Regression	82.42	3	27.47	33.47	< 0.0001
	Lack of fit	7.69	11	0.70	0.64	0.7496
b*	Pure error	5.45	5	1.09		
	Residual	13.13	16	0.82		
	Total	95.55	19			
	Regression	0.066	9	7.297E-003	11.88	0.0003
	Lack of fit	3.794E-003	5	7.588E-004	1.61	0.3060
Expansion ratio	Pure error	2.350E-003	5	4.700E-004		
	Residual	6.144E-003	10	6.144E-004		
	Total	0.072	19			
	Regression	7983.59	9	887.07	18.18	< 0.0001
	Lack of fit	377.10	5	75.42	3.40	0.1027
Rehydration Ratio	Pure error	110.91	5	22.18		
	Residual	488.01	10	48.80		
	Total	8471.60	19			
	Regression	0.092	3	0.031	45.88	< 0.0001
	Lack of fit	6.769E-003	11	6.154E-004	0.78	0.6622
Breaking Stress	Pure error	3.950E-003	5	7.900E-004		
	Residual	0.011	16	6.699E-004		
	Total	0.10	19			

# Table 4.4 ANOVA obtained by fitting models of results obtained for physical characteristics of fried extrudates

*Note: significant at* P < 0.05*, df: degrees of freedom* 

## Table 4.5 ANOVA results for the fit of data D-optimal mixture design (Fried extrudates)

Source	L*	a*	b*	ER	RR	BS
R <sup>2</sup>	0.9392	0.8852	0.8626	0.9145	0.9424	0.8959
Adj R-Squared	0.8844	0.7818	0.8368	0.8375	0.8906	0.8763
Pred R-Squared	0.6852	0.6109	0.7824	0.6806	0.7419	0.8282
Adeq Precision	13.779	11.109	14.265	11.737	12.075	17.336

Physical properties	Equations (Actual value)	M.S.E
L*	$ \begin{split} &Y_{L*} = (0.1269 * R) + (0.16716 * L) - (0.800066 * C) - (7.36716 * TP) - (0.00184039 * R * L) + (0.0184855 * R * C) + (0.0853025 * R * TP) + (0.00514249 * L * C) + (0.0745433 * L * TP) + (0.0861325 * C * TP) \end{split} $	3.68
a*	$\begin{split} Y_{a*} &= (0.0362283 * R) + (0.0252934 * L) + (0.284703 * C) + (1.79467 * TP) + \\ (0.00247933 * R * L) - (0.00267188 * R * C) - (0.0181051* R * TP) - (0.00187085* L * C) - (0.0182039 * L * TP) - (0.0177071 * C * TP) \end{split}$	0.57
b*	$Y_{h*} = (0.114331 * R) - (0.0978701 * L) - (0.0421598 * C) - (0.0883463 * TP)$	1.03
Expansion ratio	$ \begin{split} \mathbf{Y}_{\text{ER}} &= (0.0236958 * \text{R}) + (0.0261418 * \text{L}) + (0.0158931 * \text{C}) - (0.281194 * \text{TP}) - \\ & (0.000180876 * \text{R} * \text{L}) + (0.000151385 * \text{R} * \text{C}) + (0.00334013 * \text{R} * \text{TP}) - \\ & (0.000150043 * \text{L} * \text{C}) + (0.00310678 * \text{L} * \text{TP}) + (0.00286771 * \text{C} * \text{TP}) \end{split} $	0.06
Rehydration Ratio	$\begin{split} &Y_{RR} = (0.225801 * R) - (1.69869 * L) - (4.74002 * C) - (39.2249 * TP) + (0.0709888 * R * L) + (0.126815 * R * C) + (0.46381 * R * TP) + (0.0692135 * L * C) + (0.461207 * L * TP) + (0.433769 * C * TP) \end{split}$	4.97
Breaking Stress	$Y_{BS} = -(0.000245896 * R) + (0.00689417 * L) + (0.00477592 * C) + (0.00823958 * TP)$	0.05

## Table 4.6 Regression equations obtained for physical properties of fried extrudates

# Table 4.7 Analysis of Variance (ANOVA) for the individual variables and interactions

Source	L*	a*	b*	RR	ER	Hardness
Model	< 0.0001	0.0012	< 0.0001	< 0.0001	0.0003	< 0.0001
Linear	< 0.0001	0.0002	< 0.0001	< 0.0001	< 0.0001	< 0.0001
Mixture						
AB	0.6721	0.0378		0.0549	0.1497	
AC	0.0067	0.0730		0.0129	0.3339	
AD	0.0564	0.0919		0.1606	0.0117	
BC	0.4485	0.2697		0.2001	0.4215	
BD	0.0866	0.0882		0.1598	0.0163	
CD	0.0587	0.1045		0.1952	0.0271	

## among them

*Note: p*≤0.05- *significant; A- Rice flour; B- Green Lentil Flour; C- Chickpea Flour; D- Tomato powder* 

## 4.3.1.1 Texture

The textural properties of the snack were measured through measurement of 3 different properties of the product namely; Expansion ratio, breaking stress and rehydration ratio. Expansion ratio is the ratio of products expansion to that of hole of the diameter in die of extruder. It provides with the indication of puffing in the product which is related to the crispiness, absorption of water

as well as crunchiness of the product. The breaking stress gives us the ratio of force required per unit area to bring first break in the product. Hence, breaking stress is directly related to mouthfeel of the product, whereas rehydration ratio is a measurement that indicates ability of the product to absorb water. Rehydration ratio is important because depending on the trend, culture or taste an extruded product can be consumed along with a liquid also. For example, rice flakes which are usually consumed with milk in North-America are popularly consumed as savory snacks in Indian subcontinent.

## (i) Expansion ratio:

The expansion ratio of the fried products was significantly affected (p<0.01) by proportion of rice flour, lentil flour as well as by chickpea flour (Table 4.7). The interaction effect of R-TP, L-TP and C-TP also had significant effect on expansion of extrudate. The expansion ratio of the extrudates lies in the range of 1.97 to 2.21 (Table 4.3).

The contour graph shown in Figure 4.1 gives the effect of individual ingredients on expansion ratio of product. As can be seen from the values obtained in Table 4.3, the expansion ratio increased after frying of the extrudate. This is because of the increased gelatinization of starch. Several studies have suggested that the expansion ratio of a product is influenced by the degree of starch gelatinization for extrusion cooking as well as for direct heating also (Maneerote et al., 2009; Biliaderis, 1992; Chinnaswamy and Bhattacharya, 1983). However, the presence of protein as well as fiber have negative effect on the starch gelatinization and hence indirectly effect the expansion of the product, that is why the increase in the pulse flour reduced the expansion ratio of the product (Onwulata et al., 2001; Camire and King, 1991).

The expansion ratio of dried extrudates was affected the most by the incorporation of chickpea flour with decrease in expansion ratio for increasing chickpea flour. However, frying improved expansion in the extrudate as compared to the dried extrudate. Frying was done at high temperature of 200 °C, it resulted in rapid evaporation of water from the surface. Water evaporates from the boundary of the product where it comes in contact of oil. As observed by Rossell (2001) the volume of a molecule of water increases rapidly while evaporating, hence it results in the increase of expansion of product. This is even more supported by the observation that expansion in the product was not throughout the whole body of product as air cell size of the product was

bigger near the boundary rather than at the center of the product. Suknark et al. (1999) also observed increase in the expansion ratio after deep fat frying of extrudates.



Figure 4.1 Effect of mixture formulation on expansion ratio when TP was at 3.5% in the proportion

## (ii) Breaking stress:

Breaking stress is directly related to the hardness of the product as increased hardness lead to increase in the force required to bring a break in product. The minimum and maximum value calculated for extrudate breaking stress was in the range of 0.23 N/mm<sup>2</sup> to 0.47 N/mm<sup>2</sup> (Table 4.3). Increase in the value of breaking stress reduces the acceptability of the product as product with higher hardness is difficult to break in the mouth. Generally, the hardness of 200 N is considered undesirable (Forsido and Ramaswamy, 2011).

As observed from Figure 4.2, the value of breaking stress decreased with the increasing rice flour content whereas increment of lentil flour and tomato powder increased the hardness simultaneously increasing the value for breaking stress also. The correlation factor between expansion ratio and breaking stress was observed to be negative (r=-0.87), suggesting that increasing expansion ratio reduces breaking stress of product. It is because increase in the air cell weakens the cell of the product and hence when force is applied the structure of product snaps easily bringing down the breaking stress value (Barrett and Peleg, 1992). Hence, frying helped in producing a product with lower breaking stress by increasing air cell size.



Figure 4.2 Effect of mixture formulation on breaking stress when chickpea was at 20% in the proportion

#### (iii) Rehydration Ratio

The value of rehydration ratio ranged from 93.21 as the minimum value to 150.25 as the maximum value (Table 4.3). The rehydration ratio of the fried products was significantly affected (p<0.05) by proportion of rice flour, lentil flour as well as by the interaction effect of rice and chickpea flour (Table 4.7). The contour graph given in Figure 4.3 shows effect of individual product variables on rehydration ratio of the product. It was observed that value of rehydration ratio was higher in the products with higher expansion because of formation of air cells.

However, even with higher expansion ratio than the dried extrudates, maximum value of rehydration ratio was still observed to be less than dried extrudates. Saeleaw and Schleining (2011) reported that product while frying can absorb oil which is mainly driven by air cell size as well as by the amount of time taken for frying. As observed in this study also, frying resulted in increase of air cell size. High temperature resulted in evaporation of water and air cells absorbed oil while frying process. Now, even with higher air cell size, the sites were not available for water to enter, reducing absorption of water and ultimately decreasing the rehydration of product.



Figure 4.3 Effect of mixture formulation on rehydration ratio when chickpea was at 20% in the proportion

## 4.3.1.2 Color

Color is the one of the most important characteristic of the food and is the first property which affects the choices of a consumer. Measurement of color is of utmost importance especially for frying as it helps to provide insights on the extent of browning reactions that has occurred in the product. Color helps the food customers as well as manufacturers to asses the product qualitatively (Yu, 2011). Apart from own natural colors of the individual ingredients of product processing parameters also have significant effect and lead to changes in color due to non-enzymatic browning which are usually the result of Maillard reactions or caramelization because of reaction between amino acids and reducing sugars (Berset, 1989).

The color of the extrudates after frying was measured in terms of L\* (lightness), a\* (redness) and b\* (yellowness) values. To test the significance of formulation on color parameters analysis of variance (ANOVA) was used. Furthermore, the regression equation that shows the relationship between the evaluated parameters and independent variables is shown in the table 4.3.

## (i) L\* value:

The value of L\* ranged from 9.01 as the minimum value to 17.89 as the maximum value. The L\* of the fried products was significantly affected (p<0.05) by proportion of rice flour, lentil flour as well as by the interaction effect of rice and chickpea flour. Contour graph showing effect of individual product variables on the value of L\* is given in the Figure 4.4. It can be observed from the contour graph that increasing pulse flour decreased the value of L\* and higher rice flour content in formulation resulted in higher L\* values. This is because of the increment in protein content with increasing pulse flour especially the increasing availability of lysine. Lysine being the most reactive amino acid can readily react with reducing sugars and lead to non-enzymatic browning. Browning of product decreased its brightness and hence decreased the value of L\* also. Similar results and trends were also observed in the study of development of protein rich extrudates by (Forsido and Ramaswamy, 2011). The value of L\* was affected by increasing a\* values also (r= -0.77). Similar trend was found in the study by (Altan et al., 2008; Ilo and Berghofer, 1999) in their respective studies.

## (ii) a\* value:

The value of a\* ranged from 7.27 as the minimum value to 9.02 as the maximum value. The a\* of the fried products was significantly affected (p<0.05) by all the individual product variables in the formulation mixture and by the interaction effect of rice and lentil flour. The figure shows contour graph indicating effect of individual flours on the value of a\*. The maximum value of a\* was obtained with increasing proportion of tomato powder in the formulation. This is because of natural bright red color of tomato powder. The findings of this research had similar trends found in other previous research also (Dehghan-Shoar et al., 2010; Altan et al., 2008).

## (iii) b\* value:

The value of b\* which shows blueness or yellowness of the extrudate ranged from -2.35 to 5.16. Frying of the extrudates produced darker products, which led to observation of negative values in the extrudates. The increasing darkness could have masked the yellow color of products by chickpea flour. The value of b\* was affected most by proportion of rice flour in the sample. Increasing rice proportion lead to higher b\* value in the product.



Figure 4.4 Contour graph for L\* value at tomato powder proportion of 3.5 %



Figure 4.5 Contour graph for a\* value at chickpea flour proportion of 20 %



Figure 4.6 Contour graph for a\* value at tomato powder proportion of 3.5 %

## 4.3.2 Microwave roasting

Nowadays, oily food is considered bad for health especially because frying process adds to more calories coming from oil. But also, it is true that it is difficult to imitate the sensorial characteristics provided by deep fat frying. The reaction like Maillard reaction or caramelization do not occur in the microwave cooking and hence there is lack of that desirable flavor production in microwave roasting as compared to conventional frying or baking process (Bernussi et al., 1998). As snack in this study is being developed mainly for increasing the nutrition, rather than just addition of more calories, microwave roasting gives a good option to retain the product nutrients with comparable sensory characteristics.

Presence of microwaves in almost every home has made microwave roasting very popular. Industry has been interested in the application of microwave technology to improve conventional process (Ramaswamy and Van de Voort, 1990). Food products that are designed for microwave processing are more popular among people as operating microwave is very simple and processing takes very less time (Remmen et al., 1996). The process of heating a food during microwave cooking is due to non-ionizing energy generating heat by molecular friction by alternating the electromagnetic field (Lewandowicz et al., 1997). The interactions lead to the generation of heat. Studies by Ernoult et al. (2002) and Van Hulle et al. (1983) reveal that non-expanded pellets can expand with the help of microwave heating. The glassy polymeric matrix of non-expanded snack is transformed into rubbery state at process temperature with the help of moisture, now due to heating the steam bubbles are formed at nuclei resulting to expansion (Moraru and Kokini, 2003). Nebesny and Budryn (2003) reported that the expansion of amylopectin extrudates starts from the center as the heating is more at center and then expansion is seen in whole of the volume of extrudate with maximum expansion is obtained after 30 seconds. Hefnawy (2011) also reported that microwave cooking is a better option than conventional cooking for lentils as microwave cooking not only reduced nutritional losses but reduced processing time also. Microwave roasting gives a better way to preserve the antioxidants present in the food (Natella et al., 2010; Nebesny and Budryn, 2003).

The main factor effecting expansion of a cereal pellet or extrudate is moisture. Simultaneous loss of moisture is observed during microwave heating of an extrudate (Boischot et al., 2003). Ernoult et al. (2002) demonstrated that no expansion was observed in an extrudate which was dehydrated, whereas maximum expansion was obtained in the extrudates having moisture content of 10% (w/w). However, viscoelastic character or mechanical properties of the matrix also effect expansion of extrudate during microwave roasting. In the study by Chen and Yeh (2000) it was observed that addition of dextrin to rice pellets decreased the intrinsic viscosity leading to linear increase in the expansion ratio. The basis of formation of final structure is based on phase transition from glassy to rubbery state occurring during microwave cooking (Aguilera and Stanley, 1999; Kokini et al., 1992). The vibrational energy on moisture due to application of microwaves generates heat in the product. Heating generates superheated steam required for expansion, which is accumulated at the nuclei resulting in development of pressure locally (Boischot et al., 2003). Now, as a phase transition undergoes in the cereal matrix from glassy to rubbery state, the structure starts to expand due this pressure. After evaporation of moisture, structure cools down with an overall expansion in the product.

Microwave roasting induced several changes to the physical properties of the product. The main objective is to compare the physical properties, effect on antioxidants and sensory evaluation to that of the fried products.

Run	Expansion ratio	L*	a*	b*	Breaking stress (N/mm <sup>2</sup> )	Rehydration ratio (%)
1	$2.17 \pm 0.07$	22.18± 1.15	8.91±0.39	13.34±1.98	0.28±0.04	160.89±1.56
2	$2.23\pm0.05$	26.87±2.07	7.65±0.67	$12.05 \pm 1.31$	0.27±0.06	210.67±2.57
3	$2.19\pm\!\!0.07$	24.21±1.98	8.31±0.71	13.41±0.45	0.21±0.04	201.56±2.76
4	$2.31 \pm 0.08$	29.57±3.11	5.87±0.45	12.81±2.23	0.13±0.05	222.37±2.14
5	$2.23 \pm 0.05$	22.68±1.90	8.34±0.78	13.09±1.45	$0.28{\pm}0.04$	201.51±3.78
6	$2.18\pm0.11$	22.17±1.55	8.87±0.59	13.24±1.96	0.31±0.06	187.34±4.76
7	$2.12 \pm 0.03$	19.94±1.07	8.54±0.40	12.91±2.08	0.29±0.07	157.42±5.76
8	$2.06\pm\!\!0.06$	19.2±1.49	8.35±0.61	13.17±0.92	$0.3{\pm}0.05$	171.61±4.29
9	$2.17 \pm 0.5$	25.86±2.34	8.21±0.56	11.54±1.48	0.23±0.03	199.23±4.78
10	$2.38 \pm 0.07$	28.86±3.98	7.25±0.49	$11.02 \pm 2.8$	$0.19{\pm}0.08$	233.67±4.34
11	2.23 ±0.10	24.23±3.40	7.94±0.61	11.86±0.84	0.21±0.06	209.43±3.67
12	$2.26\pm\!\!0.06$	27.81±4.01	7.25±0.47	11.64±2.90	0.21±0.10	257.32±3.89
13	$2.29 \pm 0.06$	26.76±3.31	7.51±0.54	12.21±1.31	0.19±0.11	231.32±3.09
14	$2.28\pm\!\!0.07$	26.91±3.72	6.85±0.46	11.53±2.23	0.24±0.06	230.23±5.67
15	$2.45\pm\!\!0.08$	27.35±4.06	6.75±0.38	11.68±1.43	0.17±0.03	242.54±6.54
16	$2.14\pm\!\!0.07$	21.03±4.48	8.38±0.65	13.01±1.97	0.3±0.02	197.32±8.24
17	$2.18 \pm 0.04$	23.13±1.99	8.45±0.56	13.16±2.09	0.3±0.05	174.23±5.29
18	2.21 ±0.07	25.31±3.59	7.97±0.37	11.99±0.55	0.27±0.04	213.45±4.59
19	2.15 ±0.05	22.68±3.71	8.21±0.64	11.87±1.56	0.29±0.06	184.29±4.84
20	2.27 ±0.11	28.76±3.37	7.1±0.37	13.26±1.33	0.24±0.11	219.37±5.02

Table 4.8 Responses obtained for physical characteristics of microwave roasted extrudates

## Table 4.9 ANOVA obtained by fitting models of results obtained for physical

		Sum or	וע	Mean	<b>F</b> -value	P- value
		squares		Squares		
R	egression	168.72	3	56.24	80.77	< 0.0001
L	ack of fit	8.22	11	0.75	1.28	0.4165
L* P	ure error	2.92	5	0.58		
I	Residual	11.14	16	0.70		
	Total	179.86	19			
R	egression	11.02	9	1.22	15.06	0.0001
L	ack of fit	0.36	5	0.073	0.81	0.5885
<b>a</b> * P	ure error	0.45	5	0.090		
I	Residual	0.81	10	0.081		
	Total	11.83	19			
R	egression	10.53	9	1.17	18.63	< 0.0001
L	ack of fit	0.33	5	0.066	1.09	0.4628
<b>b</b> * P	ure error	0.30	5	0.060		
I	Residual	0.63	10	0.063		
	Total	11.16	19			
R	egression	0.14	9	0.016	11.57	0.0003
Expansion L	ack of fit	8.294E-003	5	1.659E-003	1.56	0.3176
ratio P	ure error	5.300E-003	5	1.060E-003		
I	Residual	0.014	10	1.359E-003		
	Total	0.16	19			
R	egression	13160.09	9	1462.23	16.32	< 0.0001
<b>Rehydration</b> L	ack of fit	250.10	5	50.02	0.39	0.8394
Ratio P	ure error	645.73	5	129.15		
I	Residual	895.83	10	89.58		
	Total	14055.93	19			
R	egression	0.039	3	0.013	19.87	< 0.0001
Breaking L	ack of fit	9.609E-003	11	8.735E-004	4.60	0.0523
Stress P	ure error	9.500E-004	5	1.900E-004		
I	Residual	0.011	16	6.599E-004		
	Total	0.050	19			

## characteristics of microwave roasted extrudates

\* significant at P < 0.05, df: degrees of freedom

## Table 4.10 ANOVA obtained after fitting of data for microwave roasted extrudates

Source	L*	a*	b*	ER	RR	BS
R2	0.9381	0.9313	0.9437	0.9124	0.9363	0.7884
Adj R-Squared	0.9264	0.8695	0.8931	0.8335	0.8789	0.7487
Pred R-Squared	0.8972	0.7078	0.7744	0.6613	0.7657	0.6901
Adeq Precision	23.707	12.700	11.254	11.837	11.390	11.516

Physical properties	Equations (Actual value)	Mean Standard Error
L*	$Y_{L*} = (0.394548 * R) + (0.0775806 * L) + (0.216131 * C) + (0.0544558 * TP)$	2.81
a*	$ \begin{array}{l} Y_{a*} = -(0.0413211 * R) - (0.029593 * L) + (0.171804 * C) + \\ (1.43869 * TP) + (0.00489261 * R * L) - (0.000432804 * R * \\ C) - (0.011237 * R * TP) + (0.00058949 * L * C) - (0.0150145 \\ * L * TP) - (0.016162 * C * TP) \end{array} $	0.54
b*	$ \begin{array}{l} Y_{b*} = (0.168397 * R) + (0.185963 * L) - (0.0222306 * C) - \\ (1.46728 * TP) - (0.00349467 * R * L) + (0.00229621 * R * C) \\ + (0.0135696 * R * TP) \\ + (0.00330117 * L * C) + (0.0222078 * L * TP) + (0.0133043 * C * TP) \end{array} $	1.64
Expansion ratio	$ \begin{array}{l} Y_{ER} = (0.029064 * R) + (0.0204972 * L) + (0.030622 * C) + \\ (0.0890001 * TP) - (0.000134554 * R * L) - (0.000268175 * R \\ * C) - (0.000326483 * R * TP) + (8.44943e-006 * L * C) - \\ (0.00105524 * L * TP) - (0.00128291 * C * TP) \end{array} $	0.09
Rehydration Ratio	$ \begin{array}{l} Y_{RR} = (2.15925 * R) - (0.059789 * L) - (1.86193 * C) + \\ (132.369 * TP) + (0.0453131 * R * L) + (0.0564364 * R * C) - \\ (1.35375 * R * TP) + (0.0584818 * L * C) - (1.544 * L * TP) - \\ (1.30444 * C * TP) \end{array} $	4.38
Breaking Stress	$Y_{BS}$ = 0.00048127 * R + 0.00529262 * L + 0.00277038 * C + 0.000645885 * TP	0.06

4.11	Regression	equations	(actual	value)	) for	microwave	roasted	extrudates
			<b>`</b>					

Table 4.12 Analysis of Variance (ANOVA) for	the individual variables and interactions

## among them

Source	L*	a*	b*	RR	ER	Breaking
						Stress
Model	< 0.0001	0.0001	< 0.0001	< 0.0001	0.0003	< 0.0001
<sup>1</sup> Linear	< 0.0001	< 0.0001	< 0.0001	< 0.0001	< 0.0001	< 0.0001
Mixture						
AB		0.0043	0.0138	0.3300	0.4532	
AC		0.8058	0.1587	0.3449	0.2544	
AD		0.3894	0.2448	0.0085	0.8439	
BC		0.7805	0.0982	0.4123	0.9753	
BD		0.2535	0.0688	0.0038	0.5251	
CD		0.2337	0.2628	0.0116	0.4547	

*Note:*  $p \le 0.05$ - significant; A- Rice flour; B- Green Lentil Flour; C- Chickpea Flour; D- Tomato powder

## 4.3.2.1 Texture

As discussed above, the analysis of physical properties was divided into two sub categories: texture and color, wherein texture expansion ratio, hardness and rehydration ratio were analyzed.
#### (a) Expansion ratio:

Expansion ratio was affected significantly (p<0.01) by the proportion of rice flour, lentil flour and chickpea flour in the formulation mixture. The products gave expansion ratio in the range of 2.06 to the maximum value of 2.45 (Table 4.8). The expansion ratio value obtained from the extrudates processed with microwave roasting processing was higher than the extrudates which were deep fried. Hence, microwave roasting process was able to give extrudates with higher puffiness.



Figure 4.7 Effect of mixture formulation on expansion ratio when chickpea flour was at 30% in the proportion

The increase in the expansion ratio of the extrudates with microwave roasted is higher because, during processing the expansion takes place throughout the whole structure of extrudate as compared to frying which induces major expansion in the outer parts of extrudate. The contour graph indicating the effect of independent individual variables on expansion ratio is shown in the Figure 4.7. It was observed that higher expansion was obtained when proportion of rice starch increased in the mixture. However, good expansion was observed even when the total proportion of rice decreased to its lowest value. Therefore, a product with almost half the proportion of rice flour can give expansion ratio equal to the maximum expansion ratio provided with deep frying of extrudate with higher rice flour content.

## (b) Breaking stress:

The value of breaking stress ranged from  $0.13 \text{ N/mm}^2$  to the highest value obtained as  $0.31 \text{ N/mm}^2$  (Table 4.8). Breaking stress was affected significantly (p<0.05) by proportion of all the individual independent variables in the formulation mixture. It was observed from the coefficients of linear regression equation that each variable was related positively to the value of breaking stress (Table 4.11).



Figure 4.8 Effect of mixture formulation on breaking stress (BS) when tomato powder was at 3.5% in the proportion

From the contour graph indicating the effect of variables on breaking stress, it was observed that breaking stress increased with increasing lentil flour in the formulation, whereas increasing rice flour decreased force required to bring a break in the extrudate. Lentils are rich in proteins which have the ability to form more strong molecular bonds than starch and can lead to more hard products. Also, increasing proteins lead to compact, less expanded and rigid products increasing value of breaking stress.

## (c) Rehydration ratio:

The rehydration ratio of the product ranged from 157.42 to 257.32 (Table 4.8). Rehydration ratio was significantly affected by the (p<0.05) by the proportion of all the independent variables as well as by the interaction effect of R-TP and C-TP.



Figure 4.9 Effect of mixture formulation on rehydration ratio (RR) when chickpea flour was 20% in the proportion

The rehydration ratio of above 200 is considered undesirable. As can be seen from the results value of rehydration ratio for some formulations surpassed 200 and hence it resulted in the breakage of product when put in liquid or the product becoming too soggy. The increase in rehydration ratio is mainly because of high expansion ratio achieved in extrudates. As expansion was observed throughout the matrix, high number of air cells were available for water to enter increasing rehydration ratio of product. The effect of variables used on the rehydration ratio is given in Figure 4.9 through contour graph. The contour graph indicates that high rehydration ratio was observed with increasing rice flour proportion. The rehydration ratio was observed above 200 with rice flour content of above 45%.

## 4.3.2.2 Color

The color of the extrudate was analyzed by measuring L\* (lightness), a\* (redness), and b\* (yellowness) values. The values varied with different proportions of formulation mixtures.

# (a) L\* value:

The value of L\* ranged from 19.2 to 29.57 (Table 4.8), which indicates that extrudates had good brightness and were light in color. L\* value was significantly affected (p<0.05) by the proportion of all the individual raw materials present in the formulation. The regression equation of actual values (Table 4.11) showed that coefficients of independent variables had positive effect on the value of L\*. Also, effect of all the individual variables on L\* is shown in Figure 4.10 with the help of contour graph. Contour graph indicates that higher L\* values were observed with higher rice content along with accompanied by higher proportion of chickpea flour. The lighter values of product were obtained as there was no significant non-enzymatic browning occurring in the extrudate.

# (b) a\* value:

The value of a\* indicates the redness or greenness in the extrudate. Value of a\* ranged from 5.87 being lowest to 8.91 being the highest value obtained. The data obtained for a\* values suggest the product showing redness. This was expected because of inclusion of tomato powder in extrudate. However, the contour graph shown in Figure 4.11 indicates that increasing proportion of chickpea flour in extrudate also increased the value of a\*. This can be due to the reason that yellow and red colors both complement each other for brightness and increasing chickpea flour is intensifying the redness of extrudate.

# (c) b\* value:

The value of b\* obtained was high and it ranged from 11.02 being the lowest value and 13.41 being the highest value obtained (Table 4.8). The proportion of rice flour, lentil flour and chickpea flour and interaction effect of rice-lentil flour had significant effect (p<0.01) on the value of b\*. The contour graph showing effect of individual flour proportion on b\* value is shown in Figure 4.12. It can be observed from the figure that increasing chickpea flour imparted more yellowish color to extrudate. Bright yellow color in extrudate is desired attribute. Chickpea has a yellowish color of its own and its increasing proportion in formulation produced snacks with higher yellowness.



Figure 4.10 Contour graph for L\* value at tomato powder proportion of 3.5 %



Figure 4.11 Contour graph for L\* value at chickpea flour proportion of 3.5 %



Figure 4.12 Contour graph for b\* value at chickpea flour proportion of 3.5 %

## 4.3.3 Antioxidant activity (AA)

The values of antioxidant activity obtained after frying of the product as well as after microwave roasting is given in the Table 4.15. It was observed from the table that extrudates with significant antioxidant activity potential were produced with the help of extrusion followed by post extrusion processing which included frying or microwave roasting of the extrudates. It was also observed that loss of antioxidant activity was higher in fried extrudates whereas microwave roasting came up as a better processing method to reduce destruction of antioxidants present in product.

 Table 4.13 ANOVA obtained for antioxidant activity values of extrudates processed with

 frying or microwave roasting

Source	AA of fried extrudates		AA of microwave roasted extrudates		
	SOS df		SOS	df	
Model	1710.45 3		5274.27	3	
Residual	123.20	16	152.64	16	
R <sup>2</sup>	0.9	328	0.9719		
Standard Error	3.	.09	2.95		

### Table 4.14 Regression equations obtained for AA of extrudates for both the

#### processing types

Processing Method	Regression Equation
Frying	Y <sub>AA</sub> = 0.0909706 * R+ 0.533287 * L+ 0.296023 * C+ 3.33007 * TP
Microwave roasting	Y <sub>AA</sub> = 0.114445 * R + 0.950508 * L + 0.433249 * C + 5.73416 * TP

### Table 4.15 Antioxidant activity obtained after post-extrusion processing methods

Run	AA (μmol TE/ 100 g db) after Frying	AA (μmol TE/ 100 g db) after Microwave Roasting
1	45.38±2.67	67.79±2.78
2	28.32±3.89	49.47±1.17
3	26.72±2.51	47.61±1.91
4	34.39±3.09	53.75±3.59
5	31.83±1.49	60.83±3.01
6	33.91±2.59	56.92±2.98
7	52.47±3.01	89.12±1.03
8	55.12±3.98	91.29±4.74
9	37.38±3.51	65.42±3.90
10	38.62±2.91	67.31±5.17
11	45.73±4.15	79.01±2.87
12	24.82±3.17	37.82±4.72
13	43.84±3.18	74.74±0.94
14	26.78±3.71	38.77±4.05
15	46.38±4.59	76.81±3.96
16	31.57±3.78	54.29±2.87
17	41.63±3.09	66.29±2.93
18	53.82±3.89	85.39±1.17
19	44.95±1.75	77.92±1.74
20	24.81±0.78	35.68±3.52

As compared to frying which retained only 50%-55% of the extrudates, microwave roasting retained more than 80% of the total antioxidants present in extrudates. Temperature can have significant effect on the antioxidant properties of a product. Frying was done at a high temperature of 200° C for 70 seconds whereas microwave roasting was done at a power level of 75% for 50 seconds only. The difference in time-temperature for both processing methods play a major role in difference in retention of antioxidant properties. Lycopene from tomato is a major antioxidant in the studied product. Mayeaux et al. (2006) studied the effect of microwave and frying on

lycopene and found that even processing of tomato slurry for 1 minute at 100% power microwave roasting retained more than 64% of the lycopene, whereas frying even at lower temperature of 145° C retained only 36.6% of the total lycopene present in tomato slurry. During microwave roasting of a product, heat is generated throughout the body of food whereas during frying outer surface of the product faces vigorous heating and hence heat is transferred from outer surface to inner part of the product leading to higher losses especially at surface (Gowen et al., 2006). The high frying temperature of oil itself has a negative effect on antioxidants as oil can produce hydroperoxide free radicals and then increase the rate of oxidation process reducing antioxidants (Gordon and Kourkimskå, 1995). The value of antioxidant activity ranged from 35.68 to 91.29 µmol TE/ 100 g db in case of microwave roasted extrudates and 24.81 to 55.12 µmol TE/ 100 g db for Fried extrudates. The highest antioxidant activity was shown by Run 8 and lowest was shown by Run 20 for both the processing methods.

#### 4.5 Sensory evaluation

The competition in outside world is enormous, hence the product or quality someone is offering needs to have attributes desired buy the purchaser or consumer. Testing the sensory properties has become the most important aspect that is now being currently used in the food industry. Use of sensory properties to help in the marketing of a food product has effective results. Information of sensory properties for a product are necessary to enhance what already is known about the product or to change the formulation or flavor in a way that it is more desired by consumer. Hence, sensory information is a crucial part of making marketing strategy of any product. In order to predict likes or dislikes of a product a sensory evaluator has an array of techniques (Stone and Sidel, 2004). It is necessary to identify the key driver and modelling of those drivers be done quantitatively. So, sensory tests when combine with product development lead to betterment of the product. Sensory practitioners clearly know the importance of consumer likes, dislikes and their preferences (Stone al., 2012).

Quality rating is generally included in the rating methods as it rates the food as per the likeness and dislikes by consumers. The major challenge to sensory evaluation is that most of the times evaluation is carried out in laboratory in which participating members usually have some knowledge about the food being evaluated and thus the response of such members may not always translate into same manner in which consumers will behave. It is because a member with lesser

experience as sensory practitioner rate food according to his own likes or dislikes and ignore the difference in inherent quality (Schutz, 1965).

Quality rating test is a part of broad category of rating methods which has been used mainly for doing sensory evaluations with smaller number of panelists. The success of a snack depends on its acceptance by the consumer. Even with better nutrition provided to snack, the desirability of a snack is driven by its organoleptic properties. For an extruded or expanded product, these properties largely depend on the number as well as size of the air cells (Morsy et al., 2015). Descriptive statistics analytical tool is a tool that has the ability to draw a relationship between sensory and intended properties that has been measured. Table 4.16 provided shows the relationship between sensory and measured properties through correlation factor (r).

Here;

- Positive linear correlation is denoted by a positive value;
- Negative linear correlation is denoted by a negative value;
- No linear correlation is denoted by a value of 0;
- The closeness of value to 1 or -1 indicates higher strength of linear correlation.

Hence, to show the strength of correlation guide suggested by (Evans, 1996) to show absolute value of r is shown:

- 0.00-0.19 suggests "very weak" correlation
- 0.20-0.39 suggests correlation is "weak"
- 0.40-0.50 suggests strength of correlation is "moderate"
- 0.60-0.79 suggests a "strong" correlation
- 0.80-1.00 suggests a "very strong" correlation

# Table 4.16 Correlation type and effect of one variable on the other correlatedcorresponding variable (source: Taylor 1990)

Correlation Type	Corresponding effect		
Correlation with positive value	Correlated variable will have tendency to increase		
Correlation with negative value	Correlated variable will have tendency to decrease		
No correlation (value of r=0)	The variable has no effect on correlated variable		

Table 4.17 shown below indicates the average scores (after rounding off) of the sensory evaluation for each run. The results obtained from sensory evaluation of the extrudates showed that except one of the microwave roasted sample of extrudate (Run 8) all others were in range of acceptable products.

Run	Quality Rating Test (QRT)							
		Fried Extrudates			Microwave roasted Extrudates			
	Color	Crispiness	Oiliness	Overall Acceptability	Color	Crispiness	Oiliness	Overall Acceptability
1	5	4	5	4	5	5	7	6
2	6	6	4	7	6	6	6	7
3	7	5	5	5	7	5	7	5
4	6	6	3	6	7	4	5	6
5	6	4	4	5	6	6	6	5
6	6	4	5	4	7	5	6	4
7	5	3	3	4	6	5	5	4
8	5	3	3	4	4	5	6	3
9	6	7	4	6	4	6	7	7
10	7	7	3	5	6	5	7	7
11	5	4	5	5	6	6	7	5
12	6	7	4	6	5	7	7	6
13	7	6	5	7	7	5	7	7
14	6	7	3	5	6	7	7	6
15	7	7	5	7	6	7	7	6
16	6	4	5	4	6	5	6	5
17	6	4	4	4	5	5	7	6
18	6	5	3	4	5	7	7	4
19	6	5	4	5	5	5	6	5
20	6	5	5	6	7	6	6	7

Table 4.17 Results obtained from sensory evaluation of extrudates

Also, three sample runs (Run 2, 13 and 15) from fried extrudates scored high overall acceptability (7) as compared to other samples of extrudates that undergo frying process. Even though all the three runs scored overall acceptability score of 7, the sample runs had different scores for color crispiness as well as oiliness which were effected by different by different formulations of product variables. For microwave, roasted products run (2,9,10,13 and 20) scored a score of 7 for overall acceptability. Hence, greater number of microwave roasted extrudates exhibited desirable properties than fried extrudates. In the Table 4.18, the signs '+' refers to "positive" linear correlation whereas sign '-' means the correlation between the variables is negative. 'FR' and 'MW' are the abbreviations for fried extrudates or microwave roasted extrudates. Response variable such as L\* value, expansion ratio and rehydration ratio of both Fried

extrudates as well as for microwave roasted products showed a high degree of positive correlation with sensorial scores viz. color, crispiness, oiliness and overall acceptability of the product.

Instrumental Response			Value of correlation r				
			Color	Crispiness	Oiliness	Overall Acceptability	
C	L*	FR	0.52	0.77	-0.15	0.81	
0		MW	0.35	0.37	0.35	0.76	
1	a*	FR	-0.40	-0.66	0.36	-0.59	
0		MW	-0.34	-0.27	0.01	-0.49	
r	b*	FR	0.57	0.84	-0.07	0.75	
		MW	0.24	-0.43	-0.35	-0.40	
Rehydration ratio		FR	0.48	0.86	-0.13	0.84	
		MW	0.31	0.53	0.40	0.51	
Expansion ratio FF		FR	0.57	0.74	-0.08	0.77	
		MW	0.43	0.35	0.34	0.60	
Breaking stress FR		-0.67	-0.89	-0.01	-0.85		
MW		MW	-0.38	-0.08	-0.20	-0.52	

 

 Table 4.18 Analysis of correlation coefficient between physical properties and sensory evaluation

However, even though rehydration ratio also showed positive correlation with sensorial attributes, microwave roasted extrudates showed rehydration ratio too high which resulted in the breaking apart of structure of extrudates with absorption of water. Furthermore, the L\* value and Expansion ratio both have a strong positive correlation with the overall acceptability of the extrudates. Hence, response variables L\* value and expansion ratio having high positive correlation factor with sensory attributes are considered and can be used as a criterion for monitoring quality after post extrusion processing. Almost all the microwave roasted products scored high for oiliness of the extrudates, with both L\* value and expansion ratio showing positive correlation for oiliness in extrudates as compared to fried extrudates. Both expansion ratio as well as L\* value had negative correlation factor with oiliness of fried products but positive correlation with microwave roasted products which had no oil uptake, this confirms that uptake of oil by the extrudates play a significant role in decreasing the acceptability of extrudates. For both the extrudates b\* value showed significant positive correlation with color score indicating that increasing b\* value increased the acceptability of extrudates color. Although b\* value was low for fried products because of non-enzymatic browning, increase in b\* value was observed with increase in chickpea flour especially for microwave roasted products. Increasing breaking stress

value was negatively correlated with sensory attributes of the processing methods and hence reducing breaking stress produced extrudates with higher overall acceptability.

Processing	Run	Composition	Expansion Ratio	Hardness	Antioxidant Activity (μmol TE/ 100 g db)	Proximate protein % (raw ingredients)
Frying	2	R (52.83%) L (28.33%) C (17.08%) TP (1.75%)	2.07	0.26	28.32	14.42
	3	R (41.5%) L (28.5%) C (30%) TP (0%)	1.97	0.32	26.72	16.08
	15	R (60%) L (23%) C (10%) TP (7%)	2.14	0.23	12.53	12.53
Microwave Roasting	2	R (52.83%) L (28.33%) C (17.08%) TP (1.75%)	2.23	0.27	44.47	14.42
	9	R(50.75%) L(35.75%) C(10%) TP(3.5%)	2.17	0.23	65.42	15.55
	10	R(60%) L(23%) C(10%) TP(7%)	2.38	0.19	67.31	12.53
	13	R(51.5%) L(20%) C(21.5%) TP(7%)	2.29	0.19	74.74	13.19
	20	R(50%) L(20%) C(30%) TP(0%)	2.27	0.24	35.68	13.75

 Table 4.19 Selected properties for extrudates that scored high score for overall acceptability



**(a)** 



**(b)** 

Figure 4.13 Extrudates after (a) Frying (b) Microwave roasting

## 4.6 Conclusions

The post extrusion processing enhanced all the physical properties of the product resulting in better sensorial attributes. Extrudates with comparable sensory characteristics were produced by using microwave roasting. Even though sensory properties of microwave roasted products were different from fried extrudates because of lesser formation of Maillard browning, the microwave roasted products scored better for sensory evaluation. Microwave roasted products use no oil which has been a matter of concern for most of the people as increasing oil content not only leads to additional calories but also expanded products being porous absorb oil significantly that alters sensory profile of these products, whereas microwave roasting resulted in exhibiting the natural sensory profile of ingredients used. Microwave roasting of extrudates produced higher expansion ratio in extrudates as compared to frying. Expansion of extrudates made the extrudate structure more crisp rather than brittle by reducing the breaking stress. Expansion ratio had strong positive correlation with overall acceptability and breaking stress also had significant negative correlation hence confirming that products with higher expansion ratio were the ones that had higher desirability in the sensory evaluation. The expansion ratio of the products achieved by microwave roasting ranged from 2.06 to 2.45, whereas frying produced expansion ratio in the range of 1.97 to 2.21. The value of breaking stress for microwave roasted extrudates was in range of 0.13 N/mm<sup>2</sup> to 0.31 N/mm<sup>2</sup> as compared to frying which resulted in products with higher breaking stress range of 0.23 to 0.47. Color also plays important role in deciding the sensory desirability of an extrudate. With the increase in frying time and temperature value of L\* decreased making the products darker. This happens because of presence of reducing sugars and amino acid in a food matrix which at high temperature react. Oiliness was negatively correlated to L\* value and expansion ratio for fried products whereas all other sensorial attributes showed positive correlation. Hence, oil absorption by fried product affected negatively for sensorial acceptance of snack.

The incorporation of pulse flours and tomato powder enhanced the nutritional aspect of the product, which ultimately increase the acceptability of product. Incorporation of lentil flour increased the protein content by large amount and chickpea flour not only provided protein but also helped imparting bright yellow color to product along with essential fatty acids to the product. Tomato powder was incorporated successfully and help enhanced antioxidants in snack.

The defining test to decide the acceptability of the snack was its sensory evaluation. The degrees of correlation between quality parameters measured instrumentally and sensory tests were observed to be strong. The average score for the overall of acceptability of products given frying process came out to be 5.15, whereas for the products roasted in microwave scored an average of 5.55. The sensory evaluation indicated that produced snacks were acceptable. Microwave roasting produced snacks with better sensorial as well as nutritional properties. Hence, it is accepted as the preferred technique of processing for producing rice based extrudates incorporated with lentil, chickpea and tomato powder.

#### **CHAPTER 5**

# SUMMARY AND CONCLUSIONS

#### **Summary**

In this research, a nutritious snack with enhanced antioxidant properties was developed using extrusion-drying technique followed by frying or microwave roasting for improving the sensorial properties. In the first part of research, effect of extrusion product variables on physical properties and drying kinetics was studied and regression equations for predetermination of physical properties were obtained. The employed processing significantly reduced antinutrients while on the other hand helped retain antioxidants in snack. However, the snack produced in the first part needed necessary enhancement in sensorial properties which was done with post extrusion-drying processing (Microwave roasting or Frying). Both the techniques improved the sensory properties, but microwave roasting produced better overall sensory scores among the two and also retained antioxidants better than frying. Overall the research resulted in successful incorporation of pulses and tomato powder in a rice based extruded snack which can improve utilisation of plant nutrients.

#### **General Conclusions**

1. The proportion of product variables i.e. rice flour (R), lentil flour (L), chickpea flour (C) and tomato powder in the formulation had significant effect (p<0.05) on the drying process of extrudates, it's water activity as well as on the time taken to achieve moisture content of 18% (dry basis). The diffusion coefficient calculated for the extrudates was affected the most by increasing lentil flour in the formulation. Increasing lentil flour decreased diffusion coefficient thereby increasing the time to achieve drying of 18% in the extrudate whereas increasing rice flour in the formulation helped achieve drying faster.

2. The physical properties were affected significantly (p<0.05) by the proportion of product variables. Increasing lentil flour and tomato powder increased breaking stress and bulk density of the extrudate. Increasing chickpea flour increased b\* value of the product because of its characteristic natural yellow color, however its increase in proportion, decreased expansion ratio producing less puffed extrudates. All products produced were of lighter color, where lightness increased with increasing rice flour proportion.

3. Increasing proportion of lentil and chickpea flour produced extrudates with protein content of approximately 12% to 19% with lentil flour being the highest contributor of increasing protein content. Tomato powder containing lycopene along with lentil flour increased antioxidant activity exhibited in snacks thus making it more nutritious. Extrusion processing helped reduce antinutrient content (tannin, phytic acid) of the snack by more than 80% as compared to its content in raw formulation, hence potentially increasing bioavailability of certain nutrients.

4. The application of processing steps to improve sensorial attributes with the help of microwave roasting or frying was done. The expansion ratio was increased from 1.67-1.92 for extrusion-drying; it was in the range of 1.97-2.21 for fried extrudates and it was in the range of 2.06-2.45 for microwave roasted products. Higher expansion in microwave roasted extrudates decreased the hardness (breaking stress) of the product also. Fried products were darker due to non-enzymatic browning whereas microwave roasting produced brighter yellow colored products.

5. Microwave roasting produced higher average for overall acceptability of the products along with better retention of antioxidants in snacks. Regression equations obtained by fitting suitable models for microwave roasting and frying process were useful to predetermine physical characteristics of the product to tailor make changes in product for improving its attributes.

### **Future Recommendations**

The extrusion of rice based snack enriched with pulse flour and tomato powder resulted in number of important findings. Extrusion is a complex process in itself, combining it with post extrusion processing makes it more complex but on the same time, it opens door for a lot of possibilities. With better understanding of process and factors influencing processing and production of expanded snacks, the research also showed some possibilities of doing further research and development, summarized as follows:

1. Alternative sources of plant protein on the product characteristics can be studied to measure the effect on product characteristics, such as flour from lupin bean can also be used as an alternative.

2. Similarly, there is possibility of replacing rice as a starch source with other novel starch sources such as buckwheat, tef etc. to study their effect on product properties.

3. Other techniques of finished processing for improving sensory attributes of product can also be explored such as air frying, pressure frying etc.

4. Pulse flours have high amount of resistant starch. Effect of processing on resistant starch was not studied in this research. Hence, effect of extrusion and post extrusion processing on a formulation of resistant starch from different sources can provide valuable data on nutritional aspect of products.

5. Porous structure of extrudates results in higher oil absorption which leads to lower acceptability of the product. The possibility of coating the product with a layer of sugar or with some other ingredient can be explored to decrease oil absorption during frying.

6. Microwave roasting of food extrudates has a high potential, however to develop a flavor profile like fried products a technique can be developed to spray oil before processing of extrudates or during processing inside the microwave to achieve desired results or instead of oil spraying the product with a flavor substance can add to its desirability.

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