KINETICS OF ACRYLAMIDE FORMATION IN POTATO CHIPS

A Thesis

by

CLAUDIA ESTHELA GRANDA

Submitted to the Office of Graduate Studies of
Texas A&M University
in partial fulfillment of the requirements for the degree of

MASTER OF SCIENCE

May 2005

Major Subject: Biological and Agricultural Engineering

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Approved as to style and content by:

Rosana Moreira (Chair of the Committee)	M. Elena Castell-Perez (Member)
J. Creighton Miller, Jr. (Member)	Gerald Riskowski (Head of Department)

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ABSTRACT

Kinetics of Acrylamide Formation

in Potato Chips. (May 2005)

Claudia Esthela Granda, B.S., Texas A&M University

Chair of Advisory Committee: Dr. Rosana Moreira

Acrylamide is considered a carcinogen in animals and a possible carcinogen in humans. It has been found in starch rich foods cooked at high temperatures. Vacuum frying (10 Torr) was studied as a possible alternative to reduce acrylamide formation in potato chips.

Seven potato cultivars were analyzed to determine their influence on acrylamide formation during traditional and vacuum frying. The White Rose cultivar produced the highest level of acrylamide during both traditional and vacuum frying. Vacuum frying (10 Torr, 118^{o}C, 8 min) produced chips with lower acrylamide content than those produced under traditional frying (165^{o}C, 4 min) for all cultivars.

The cultivar Atlantic was used to determine the kinetics of acrylamide formation during traditional and vacuum frying at different temperatures because it is a good chipping cultivar and it was the most abundant in the lab. Acrylamide accumulation under vacuum frying was modeled using first-order kinetics, and during traditional frying it was modeled using the logistic kinetic model. The behavior of the kinetics of acrylamide content in potato chips fried under the two processes was different mainly due to the different temperatures used. During traditional frying, higher temperatures are

used (150^{o}C to 180^{o}C) and acrylamide after some time is produced but also starts degrading, producing a constant level of acrylamide content at longer times. During vacuum frying (10 Torr), acrylamide increased exponentially (but at lower levels) for all frying times.

The effect of potato components was studied by infusing leached potato slices with predetermined amounts of glucose and asparagine. Increasing glucose and asparagine content in the slices increased the acrylamide content in the potato chips. Color could not be used as an indication of acrylamide content, since potato chips with similar color had very different acrylamide concentrations.

DEDICATION

To my parents, Beni and Martha.

It is really hard to find the words that

could express all my gratitude and my love for them.

I can just say that I owe them everything, and that

I feel like the luckiest person for having them as my parents.

Los admiro y los quiero muchisimo. Gracias por todo!

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CHAPTER I

INTRODUCTION

Eating potato chips may seem harmless. However, recent studies have shown that subjecting foods to high temperatures during cooking processes such as frying could give rise to the formation of acrylamide, a chemical that causes cancer in rats and may cause cancer in humans (IARC, 1994). Several factors including product composition and processing conditions may affect the rate of formation of this chemical in starch rich foods. Low reducing sugar and the amino acid asparagine content is desired when cooking, since the formation of acrylamide is attributed to the Maillard reaction that occurs between these food components (Zykak et al., 2003). Scientists are investigating ways of reducing the formation of acrylamide in foods without giving up flavor and quality. However, not many alternatives have been investigated. The main research focus has been on proposing pathways for the formation of acrylamide, and on the effects of some parameters such as product pH and processing temperature.

The long term goal of this area of study is to develop effective ways for the production of safe fried food products. This study focused on vacuum frying, a process that utilizes low temperatures and pressures. The effect of temperature and frying techniques (atmospheric vs. vacuum) was evaluated in terms of the formation of acrylamide.

This thesis follows the style and format of the Journal of Food Engineering.

Garayo and Moreira (2002) studied the effect of vacuum frying on the quality of potato chips, achieving better color and lower oil content than when using traditional (atmospheric) frying. The lower temperatures used in vacuum frying also maintain a better frying oil quality. This information supports the hypothesis that this process will not only yield a product with better quality, but also one with less acrylamide content.

The main objective was to evaluate the potential of vacuum frying as a technique to produce fried potatoes with lower acrylamide content.

The central hypothesis was that the lower temperatures achieved during vacuum frying together with lower content of reducing sugars and asparagine will hinder the formation of acrylamide.

The central hypothesis will be proven and the main objective achieved by accomplishing the following specific objectives:

(1) To determine the kinetics of acrylamide accumulation in potato chips fried under vacuum and atmospheric conditions at different frying temperatures.

(2) To determine the effect of potato cultivar on the acrylamide content in potato chips fried under vacuum and atmospheric conditions.

(3) To determine the influence of raw product composition (reducing sugars and the amino acid asparagine) on the acrylamide content in potato chips fried under atmospheric conditions.

The proposed research aims to decrease the level of acrylamide formation in potato chips while maintaining product quality. Product composition (reducing sugars and asparagine) and processing conditions (frying temperature and residence) were carefully studied as they are considered critical factors affecting final product properties and acrylamide formation.

CHAPTER II

LITERATURE REVIEW

Deep-fat frying is one of the oldest and most common unit operations used in the preparation of foods. This process results in products with unique flavor-texture combination. Some of the products that are deep-fat fried include potato chips, French fries, extruded snacks, doughnuts, fish sticks, and fried chicken products (Moreira et al., 1999). In the U.S., about 1.2 billion pounds of potato chips are consumed each year (USDA, 2002). However, consumer fears have started to arise as acrylamide, a possible carcinogen, has been detected in foods exposed to high temperatures, including fried and baked foods (Tareke et al., 2002).

2.1. Acrylamide

2.1.1. Background

Acrylamide (2-propenamide) is a chemical that has been produced since the 1950's by hydration of acrylonitrile to synthesize polyacrylamide (Friedman, 2003). Polyacrylamide has several applications in the cosmetic and packaging industries, in soil and conditioning agents, in treatment of sewage and wastewater, and in the purification of drinking water; it is also found in tobacco smoke (Smith et al., 2000). The acrylamide monomer is toxic, while its polymer is not (IARC, 1994).

The major ways of exposure to this chemical are in manufacturing of acrylamide and polyacrylamide, acrylamide grouting, and during preparation of polyacrylamide gels (Friedman, 2003).

Acrylamide is classified by the International Agency for Research on Cancer (IARC) as *probably carcinogenic to humans* and *carcinogenic in animals*. It is also considered by this same agency as *neurotoxic to humans*.

Rice (2005) indicated that acrylamide is carcinogenic to experimental mice and rats, causing tumors in both species when ingested. Epidemiologic studies of people exposed to acrylamide either through food or occupational exposure have not shown consistent evidence of increased cancer risk. However, a double risk for pancreatic cancer was observed in highly exposed workers.

LoPachin (2004) focused his investigation on the neurotoxicity of acrylamide. The author indicated that acrylamide acts mainly on the nerve terminal, and that eventual degeneration occurs due to membrane-fusion processes that impair neurotransmitter release.

The study on acrylamide in foods originated in Sweden, when a sealant called Rhoca-Gil, that was injected into cracks in tunnel walls, contaminated the ground and water with this toxic chemical. Workers suffered numbness due to neurotoxicity (Reynolds, 2002).

The tunnel accident in Sweden prompted researchers to measure an acrylamide reaction product bound to hemoglobin (adduct) to study people that were exposed to the monomer. Hemoglobin adducts are not an indication of toxic action but are used for

intake calculations and to measure exposure (Lingnert et al., 2002). The great surprise was that not only tunnel workers had the adduct N-(2-carbamoylethyl)valine (CEV) to the N-termini of hemoglobin (Hb), but also nonsmokers without occupational exposure (Tareke et al., 2002). To test the hypothesis that this compound was formed during cooking, control rats were fed with un-fried diet, and another group of rats with fried animal standard diet for 1 or 2 months. The animals fed the standard diet showed a level of the CEV adduct ten times lower than the ones fed the fried diet.

The U.S. Environmental Protection Agency (EPA) requires the limit for acrylamide content in water to be less than 0.5 ppb (EPA, 2004). Starch-rich products, such as those produced from potatoes (7.80-25.43% starch), have a much higher content of acrylamide (170-3700 ppb) than the level identified as safe by the EPA (Becalski et al., 2003). This data supports the need for finding ways of reducing the formation of this chemical in foods.

Several studies have been performed by different institutes such as the Center for Food Safety and Applied Nutrition (CFSAN) and the World Health Organization (WHO) to determine the level of acrylamide in commercially available foods. Table 2-1 shows some ranges for different products.

Other carcinogens in foods have been identified, such as those produced in grilled or fried meat, known as the polycyclic hydrocarbons (PAHs) (Ferguson, 2002).

According to the Harvard Center for Cancer Prevention (2004), 1.2 million Americans will be diagnosed with cancer and an estimated 556,500 will die from this disease annually, the nation's second-leading killer after heart disease. Diet is one of the

main factors influencing the development of the disease; in fact, approximately 35% of cancer deaths are thought to be linked to food (Ward, 1994).

Table 2-1. Acrylamide content of different foods (CFSAN/FDA, 2002; Becalski et al., 2003).

PRODUCT	ACRYLAMIDE CONTENT (ppb)
Potato chips	170-3700
Potato chips (sweet)	767-2762
French fries (baked)	356-1325
French fries	49-1900
Breads and Bakery products	ND-364
Cereals	47-266
Coffee	175-351

2.1.2. Theories for acrylamide formation

Since the release of information on the formation of acrylamide during food processing, several hypotheses have been proposed on its development. All these are possible routes for acrylamide formation that could take place in a food processing scenario.

2.1.2.1. Formation from acrolein

Acrolein (CH_2=CH-CHO) is a three-carbon aldehyde with a structure very similar to that of acrylamide. As reported by Grivas et al. (2002), acrolein is known to be formed by: 1) transformation of lipids, 2) degradation of amino acids and proteins, 3) degradation of carbohydrates, and 4) the Maillard reaction between amino acids or proteins and carbohydrates. The formation of acrylamide from acrolein could, in principle, be directly transformed to acrylamide by reaction with ammonia (NH_3) (Figure 2-1). This pathway has been proven in model systems (Tezer & Ozkan, 2001).

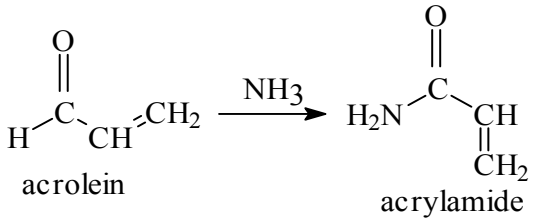


Figure 2-1. Formation of acrylamide from acrolein.

2.1.2.2. Formation from the Maillard reaction

Tareke et al. (2002) showed that moderate levels of acrylamide (5-50 ppb) were found in heated protein-rich foods, while higher levels (150-4000 ppb) originated from carbohydrate-rich foods. Raw or unheated foods did not exhibit any acrylamide formation. This indicates that some reaction taking place during cooking of carbohydrate-rich foods is responsible for the formation of acrylamide. For instance, the development of acrylamide as a by-product of Maillard browning is currently the most accepted theory (Stadler et al., 2002; Yaylayan et al., 2003).

The Maillard reaction is also known as non enzymatic browning, which is responsible for most of the flavor and aroma in heated foods. It consists of the chemical reaction between reducing sugars (mainly D-glucose) and a free amino acid (Fennema, 1996). An associated process is the Strecker degradation of amino acids by intermediates of the Maillard reaction. During the Strecker degradation, the amino acid is decarboxylated and deaminated to form an aldehyde, which is a proposed pathway for the formation of acrylamide: the amino acids asparagine and methionine go through Strecker degradation in the presence of dicarbonyl products from the Maillard reaction resulting in the formation of acrylamide (Mottram et al., 2002) (Figures 2-2 and 2-3).

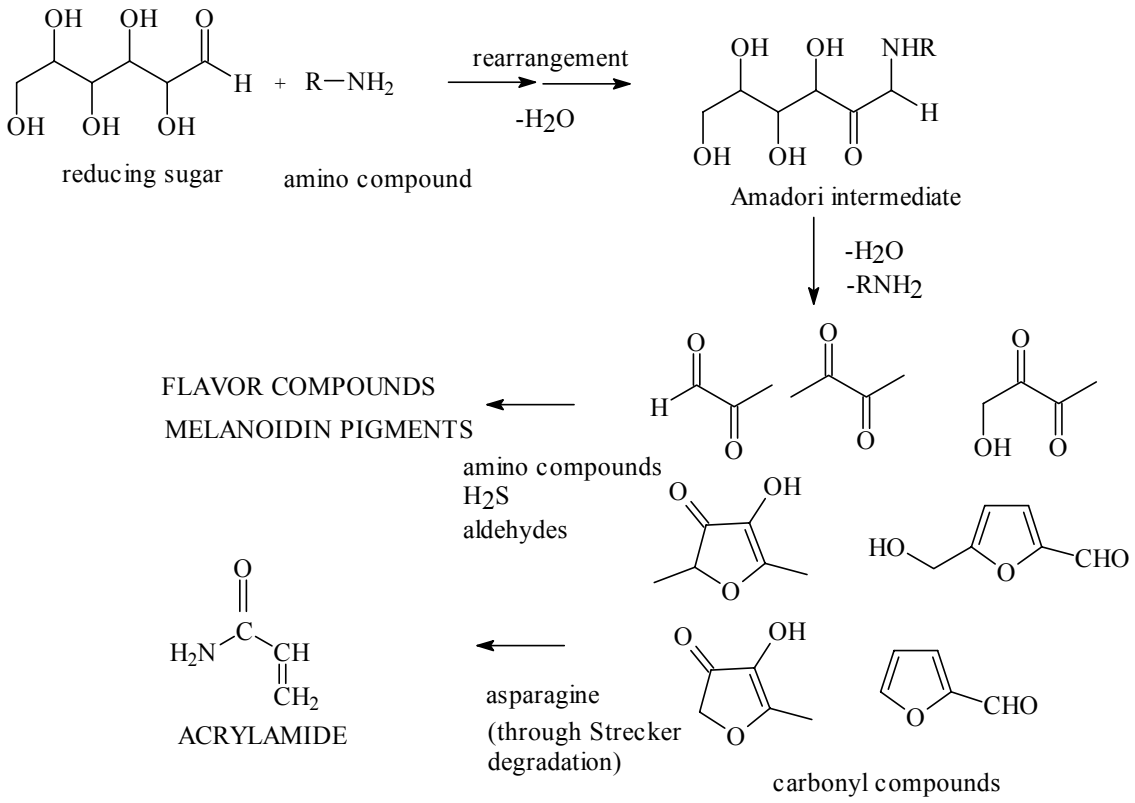


Figure 2-2. Schematic of production of acrylamide from the Maillard reaction (adapted from Mottram et al., 2002).

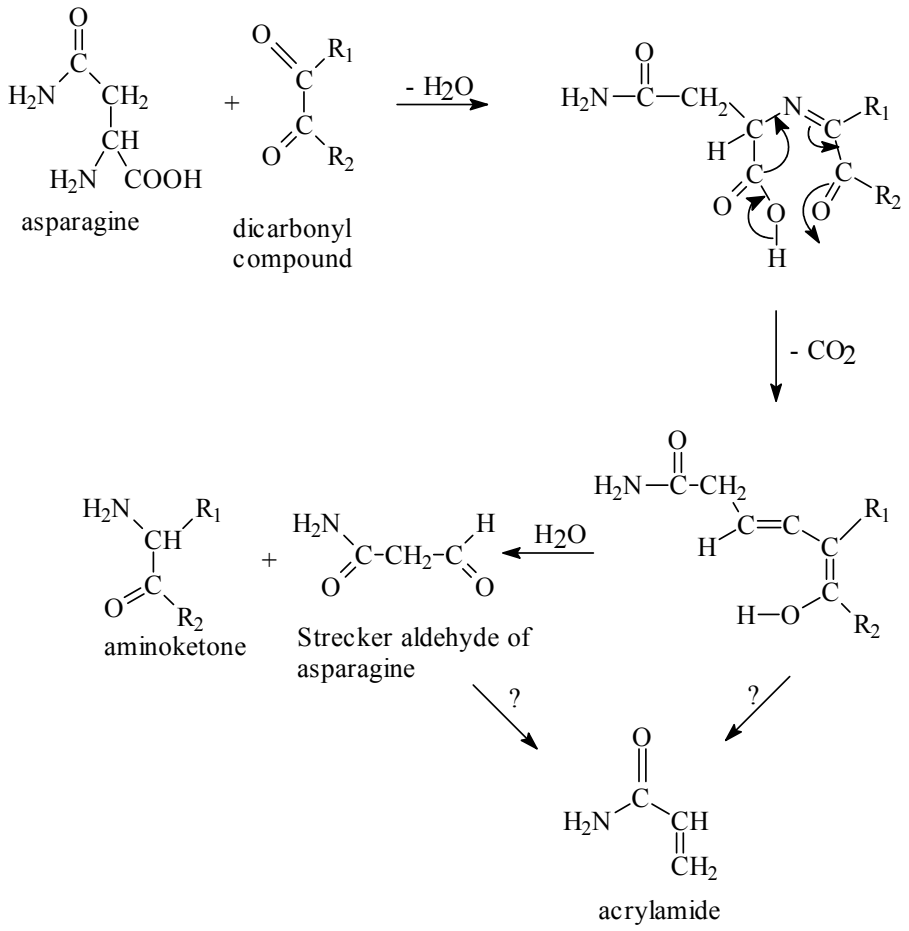


Figure 2-3. Proposed pathway for the formation of acrylamide after Strecker degradation of asparagine in the presence of dicarbonyl products from the Maillard reaction (adapted from Mottram et al., 2002).

Mottram et al. (2002) performed several Maillard reactions to see which ones resulted in the production of acrylamide. Glucose and several amino acids were tested. When asparagine reacted with glucose at 185^{o}C, significant amounts of acrylamide were formed (221 mg per mol of amino acid). When other amino acids were used (glycine, cysteine or methionine) no acrylamide was detected (less than 0.5 mg/mol); using glutamine and aspartic acid produced only trace amounts (0.5-1 mg/mol). The fact that asparagine was the only amino acid that produced significant amounts of acrylamide can be explained by its structure, which already has the correct backbone for the formation of this compound (Figure 2-4 and Figure 2-5).

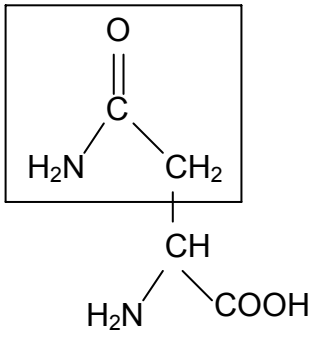


Figure 2-4. Asparagine structure

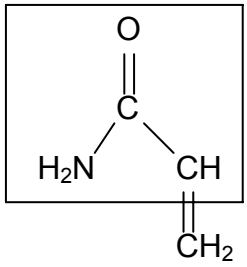


Figure 2-5. Acrylamide structure

Reducing sugars and amino acids are also the precursors of flavor components and browning (Fennema, 1996), which means that acrylamide is generated in addition to these attributes (Amrein et al., 2003).

2.1.3. Processing conditions affecting acrylamide formation in food

2.1.3.1. pH

Decreasing the pH is a way to reduce the Maillard reaction when it is undesirable (Schwartzberg & Hartel, 1992). Jung et al. (2003) proposed a theory of acrylamide reduction by lowering the pH in the raw product before cooking. In the Strecker degradation reaction, a Schiff base formation is the initial reaction step, which is an addition-elimination reaction. The first step is the addition of the nucleophiclic α-amino group of the asparagine to the partially positive carbonyl carbon of the dicarbonyl group, followed by the loss of a proton from the nitrogen and the gain of a proton by the oxygen (Figure 2-6). The authors propose that lowering the pH blocks the nucleophilic addition of the free nonprotonated amine ($-NH_2$) to the carbonyl group, by converting the free nonprotonated amine to nonnucleophilic protonated amine (NH_3^+), therefore avoiding the formation of acrylamide.

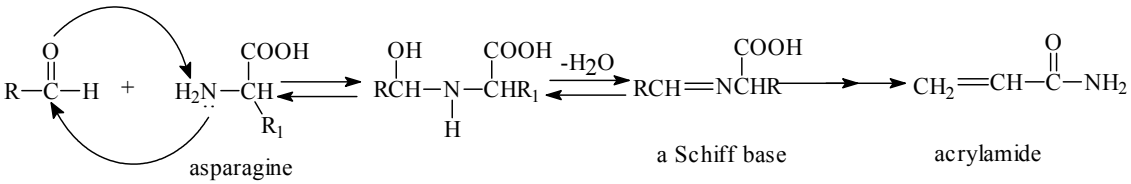


Figure 2-6. Schiff base formation in the course of acrylamide formation (adapted from Jung et al., 2003).

2.1.3.2. Moisture

Stadler et al. (2002) observed that when pyrolyzed at 180°C, in the presence of glucose, asparagine formed significant amounts of acrylamide (368 ppm). The interaction of the reactants was improved accordingly when adding water to the reaction mixture, and there was an increase of the product (acrylamide) in the reaction (960±210 ppm) (Stadler et al., 2002).

2.1.3.3. Time and temperature

Schwartzberg and Hartel (1992) suggested that one of the measures to inhibit Maillard reaction in cases where it is undesirable is the maintenance of lowest possible temperatures. Tareke et al. (2002) conducted experiments to monitor the formation of acrylamide during heating of different foodstuffs. These studies showed that the formation of acrylamide was temperature dependent, increasing with increasing temperatures. Raw or unheated foods did not exhibit any acrylamide formation. Mottram et al. (2002) indicated that acrylamide formation increases with temperature from about 120°C to 170°C and then decreases.

The data published so far indicate that a temperature greater than 100°C is required for the formation of acrylamide (Becalski et al., 2003). Biedermann et al. (2002) also set 100°C as the threshold for acrylamide formation in potato chips, but also indicated that high temperatures (higher than 170°C) not only accelerate formation, but also elimination of acrylamide. They found that concentrations of acrylamide did not increase exponentially with higher temperatures due to the formation and elimination of acrylamide.

Acrylamide decomposes and polymerizes on melting above 175°C (Becalski et al., 2004). However, heating of acrylamide in sealed tubes at 120°C for 30 min, at 180°C for 10 min, and at 230°C for 5 min produced a 40-50% decrease in acrylamide content. This reduction was not altered in the presence of polymerization inhibitors, indicating that under these conditions the decrease of acrylamide content already occurs below the melting point and is mainly due to degradation rather than polymerization. Biedermann et al. (2002) found that some potato cultivars contained more acrylamide after heating to 180°C than after 120°C (Charlotte cultivar, 9000 ppb vs 3000 ppb, respectively), but others contained less (Sirtema cultivar, 9000 vs. 14000, respectively) maybe due to differences in raw product composition. The data suggested that the Sirtema cultivar was more active than Charlotte in both senses: acrylamide formation was faster, as observed at 120°C, but also its elimination.

Surdyk et al. (2004) found that not only temperature (above 200°C) but also heating time increased the acrylamide content in yeast-leavened wheat bread crust.

When bread was baked at 270°C for 18 and 32 min, acrylamide content increased from about 300 ppb to 1200 ppb, respectively.

2.1.4. Acrylamide reduction

Several investigators have reported methods to reduce acrylamide content in foods. Jung et al. (2003) achieved an acrylamide reduction of approximately 80% in fried and baked corn chips and in French fries by dipping them in citric acid before frying. The pH of corn grits was lowered from 5.7 to 4.2, and the pH of potatoes from 6.2 to 4.9. Kita et al. (2004) also lowered the pH in potatoes slices with a 90% decrease in acrylamide content in potato chips after blanching in acetic acid for 60 min at 20°C before frying, reducing the pH of the slices from 6.05 to 4.15. They also found that short-frying for 3-7 min at 160°C and for 2-5 min at 180°C followed by post-drying in a hot air oven (105°C) for 30-120 min produced a decrease in acrylamide content of ~80% and ~70%, respectively.

Other studies involved coating the product before applying heat. Fiselier et al. (2004) coated potato croquettes with a mixture of egg and breadcrumbs. Even though the product was browner than regular croquettes, the acrylamide content was reduced from 280 to 50 ppb.

Pedreschi et al. (2004) investigated the influence of acrylamide content in potato chips in relation to frying temperature and three different treatments: 1) soaking in distilled water for different time periods, 2) blanching in hot water at six different time-temperature combinations, and 3) immersing in citric acid solution. By soaking and

blanching, they proved that the glucose and asparagine content of the potato slices were on average 76% and 68% lower than the control. Blanched potato slices had very low acrylamide content (28 ppb) even when fried at very high temperatures. As also shown by Jung et al. (2003), citric acid immersion before frying reduced the acrylamide content by 70%. For all treatments, as the temperature of the oil increased from 150^{o}C to 190^{o}C, acrylamide content increased (Pedreschi et al., 2004).

Grob et al. (2003) also succeeded in removing asparagine and reducing sugars by blanching or rinsing the potatoes before frying. They produced French fries with 40-70 ppb acrylamide, which is 5-10 times lower than in normal fries. This study also focused on finding the optimum potatoes suitable for French fries, i.e., those that would become crispy before the on-set of substantial browning. To accomplish this objective, they selected the cultivar Agria, an important variety grown in Switzerland with very low reducing sugar content that has a yellow color and yields a golden rather than gray product.

Chuda et al. (2003) studied the effect of potato storage temperatures on the formation of acrylamide after frying. In their study, potatoes were stored at 2^{o}C and 20^{o}C for two weeks prior to processing. They achieved a ten-fold decrease (20 ppm to 2 ppm) in acrylamide in potato chips produced from tubers that were stored at higher temperatures due to the smaller concentration of reducing sugars.

Amrein et al. (2004) investigated the influence of ingredients, additives, and processing conditions on acrylamide formation in gingerbread. The addition of ammonium hydrogencarbonate enhanced acrylamide accumulation. On the other hand,

addition of sodium hydrogencarbonate as a baking agent reduced acrylamide concentration by more than 60%. Another method of reducing acrylamide formation in gingerbread was developed by replacing reducing sugars with sucrose or by adding organic acids. When honey, inverted sugar syrup, and caramel coloring were replaced by sucrose solutions, the acrylamide content was reduced by a factor of 20 (to 25 ppb). When 1% citric acid was added to the dough, acrylamide content was reduced from 501 ppb to 12 ppb.

Vattem and Shetty (2003) obtained reduced acrylamide formation in potato chips (from 930 ppb to 580 ppb) when potato slices were coated with chickpea batter before deep-fat frying (10 min). They hypothesized that legume proteins can prevent the breakdown of the starch complex, therefore decreasing the formation of reducing sugars that accelerate the Maillard reaction and hence acrylamide formation. They also indicated that acrylamide formation is a non-oxidative process because acrylamide content was not affected when phenolic antioxidants from cranberry and oregano extracts were used as coatings.

2.2. Deep-fat Frying

The purpose of deep-fat frying a product is to seal the food by immersing into hot oil so that all the flavors and juices stay inside the crust (Moreira et al., 1999). During deep fat frying, it is important that the product is completely covered by the oil by at least 2 cm. In addition, some products that do not have a high starch content need to be coated before frying (Moreira et al., 1999).

As explained by Mellema (2003), when a food product is added to hot oil in deep fat frying, the surface temperature of the food rises very rapidly and the water at the surface starts boiling. As boiling starts, convection in the oil will be increased by the turbulent water vapor. Explosive evaporation will lead to the formation of large pores. Water inside the food will be heated and the product will be cooked. When a product is deep-fat fried, several changes can take place, including starch retrogradation, Maillard reactions, and glass transitions.

Deep-fat frying can be accomplished under three different pressure conditions: atmospheric pressure, low (vacuum), and high pressure (Moreira et al., 1999). Under atmospheric (open) frying, the temperature of the oil is usually between 160^{o}C and 200^{o}C (Moreira et al., 1999).

Pressure fryers were developed mainly for frying chicken because the time is reduced when compared to open frying, and the food is more moist and uniform in color and appearance (Moreira et al., 1999). The temperature of pressure fryers can vary from 160^{o}C to 177^{o}C, and the pressure ranges can be 9-14 psi and 28-32 psi. The disadvantages of pressure frying are that the oil will degrade faster, and it has to be filtered more frequently (Moreira et al., 1999).

Vacuum frying is carried out at pressures below atmospheric levels, preferably below 6.65 kPa. When the pressure is lowered, the boiling point of the oil and the moisture of the foods are lowered as well (Garayo & Moreira, 2002). This property of reducing the boiling point gives rise to some advantages, which include: 1) reducing the oil content of the product; 2) preserving color and flavor; and 3) maintaining good oil

quality for longer times (Shyu et al., 1998). Vacuum frying is a process that could be a feasible alternative to produce chips with lower oil content and with good color and texture (Garayo & Moreira, 2002).

2.3. Raw and Processed Product Quality

2.3.1. Reducing sugars and asparagine

2.3.1.1. Reducing sugars

Aldoses are called reducing sugars because when oxidizing the aldehyde group of an aldose to the salt of a carboxylic acid group the oxidizing agent is reduced (Fennema, 1996). Ketoses are also called reducing sugars because under the alkaline conditions of the Fehling test, ketoses are isomerized to aldoses; the Benedict reagent will react with aldoses, but not with ketoses (Fennema, 1996).

Reducing sugar content is used by the food industry to predict potato tuber suitability for chip processing, since it is often an indicator of color development (Marquez & Añon, 1986). Some products, e.g. potatoes, may accumulate high levels of reducing sugars during storage at low but nonfreezing temperatures in a process known as "low-temperature sweetening" (ApRees et al., 1981). For instance, Brierly et al. (1996) reported that the reducing sugar content in the Pentland Dell cultivar ranged between 0.184%$\pm$0.039% and 0.335%$\pm$0.066% when stored at 5^{o}C for 40 weeks. However, when this same cultivar was stored at 10^{o}C for 40 weeks, the total reducing sugar content was significantly lower (0.001%$\pm$0.001% to 0.126%$\pm$0.043%).

Glucose reagent strips with a detection limit equivalent to approximately 0.1 mg/ml were used by Martin and Ames (2001) to confirm the removal of reducing sugars in potato slices; they also used capillary electrophoresis (CE) to determine how much reducing sugars were absorbed by the infused slices. High Performance Liquid Chromatography (HPLC) analysis has been used by other authors to determine the concentration of sucrose and reducing sugars (Rodriguez-Saona & Wrolstad, 1997). Marquez and Añon (1986) used Thin Layer Chromatography to study the influence of reducing sugar concentration in the color development of fried potatoes. Sowokinos et al. (1987) used an Industrial Analyzer which determines the concentration of *B*-D-glucose via a glucose oxidase-bound membrane system.

Colorimetric methods can also be used to determine reducing sugar content (Nourian et al., 2002). Dinitrosalicylic acid reagent oxidizes the aldehyde functional group present in reducing sugars (Miller, 1959). The different color intensities measured in a spectrophotometer are correlated to a standard curve to determine their concentration (Miller, 1959). Nourian et al. (2002) used ferrocyanide in a carbonate-phosphate buffer as the oxidizing agent for sugars. The ferrocyanide produced was measured by determining the absorbance in a spectrophotometer using the blue solution formed after the addition of Nelson's arsenomolybdate reagent.

2.3.1.2. Asparagine

α-amino acids are the basic structural units of proteins and consist of an α-carbon atom covalently attached to a hydrogen atom, an amino group, a carboxyl group, and a

side-chain R group. Amino acids can be classified into several categories, depending on how much their side chain interacts with water. For instance, asparagine is a polar (hydrophilic) and uncharged amino acid (Fennema, 1996).

Some raw products, such as potatoes, have a high level of asparagine (0.094-1.137%) that affects the physical and chemical characteristics of the final product (Martin and Ames, 2001; Becalski et al., 2004). Several authors have studied the asparagine content of potatoes, especially to investigate its effect on color due to the Maillard reaction.

Detection of amino acids has been extensively performed by pre-column derivatization. The most commonly used reagents for pre-column techniques have been ortho-phthalaldehyde (OPA), dansyl or dabsyl chloride and phenyl isothiocyanate (PITC) (Cohen, 1984). As explained by the manufacturer (Pierce, Rockford, IL), PITC reacts readily with amino acids at alkaline pH in 5 to 10 minutes at room temperature. After derivatizing, phenylthiocarbamyl derivatives (PTC-amino acids) are formed, which can then be quantified using reverse-phase HPLC with a UV detector at 254 nm.

Rodriguez-Saona & Wrolstad (1997) used PITC pre-column derivatization and coupled columns to find the amount of asparagine absorbed by infused potato slices. Similar procedures involving pre-column derivatization with PITC have been used by several authors (Bidlingmeyer et al., 1984; Lavi et al., 1986; Hagen et al., 1993). Fabiani et al. (2002) used 9-fluorenylmethyl-chloroformate for derivatization of 16 amino acids, after adjusting the pH of fruit juices to alkaline values.

2.3.1.3. Reducing sugar and asparagine content variability between products

Doyle (2002) compiled free asparagine levels of different raw products, showing wide differences among them. Even within the same product, large differences on free amino acid content can be seen. For instance, Brierley et al. (1997) reported differences on the free asparagine content in potatoes that were stored under different conditions: right after harvesting their content was 0.21%; after storage at 5^{o}C for 25 weeks, it increased up to 0.73%; finally, after storage at 10^{o}C for 25 weeks, the free asparagine content was 0.93%.

Davies (1977) studied 31 varieties of potatoes grown in England and Ireland. This study showed that there were large quantitative differences of free amino acids not only between varieties, but also due to location and year of growth. For instance, within the same cultivar, asparagine content varied from 1.022 to 2.068% for the cultivar Majestic, from 0.371 to 1.673% for Arran Victory, and from 0.483 to 1.567% for Kerr's Pink. Other authors (Brierley et al., 1997; Fitzpatrick & Porter, 1966; Racusen, 1983) have indicated that free amino acids increase with prolonged storage, and that this accumulation cannot be reduced by reconditioning. Brierley et al. (1997) reported that the total free amino acid content of Pentland tubers was 0.170% after storage at 5^{o}C for 32 weeks; reconditioning for 6 weeks at 20^{o}C resulted in the same asparagine content (~0.170%).

Reducing sugar content of potatoes also exhibits great variability. Williams & Cobb (1992) observed low temperature sweetening in potatoes stored at 5^{o}C and 10^{o}C. As storage temperature decreased and time increased, they found an increase in reducing

sugar content. Thus, potatoes stored at 5^{o}C for 40 weeks showed an increase in reducing sugar content from 0.2% to 2%. When they were stored at 10^{o}C, the reducing sugar content increased from 0.2% to 0.7%. Low temperature sweetening was reversed by periods of reconditioning for 6 weeks at 20^{o}C (Williams & Cobb, 1992).

Due to the great differences in asparagine and reducing sugar content in raw potatoes, several authors have developed model systems to study how these components affect the final product. Roe and Faulks (1991) studied the effects of amino acids and sugars on the color development of infused filter paper discs. However, this type of model system overlooks the effect of the food matrix (Bakker et al., 1996). Rodriguez-Saona & Wrolstad (1997) studied the contribution of potato components by using a potato model system that did take into account its structure. Soluble constituents of potatoes were removed, and different concentrations were infused to study the individual contributions of ascorbic acid, glutamine, asparagine, chlorogenic acid, sucrose, and reducing sugars in the color development of potato chips. Khanbari & Thompson (1993) also used a model system based on sliced raw potato to study the color development in chips by removing amino acids and sugars with water and ethanol and then soaking in solutions of the amino acids and various concentrations of glucose. A similar procedure was used by Martin & Ames (2001) to study the formation of Strecker aldehydes and pyrazines in a fried potato model system.

2.3.2. pH

pH can affect the reactions taking place in a food product, and also reactions taking place in a food product may change the pH (Jung et al., 2003). The pH of raw potatoes is usually around 6.0 (Nourian et al., 2002).

Kaaber & Martinsen (2002) decreased the browning in pre-peeled potatoes by decreasing the pH by adding CO_2 to the soaking water (pH in the CO_2 solution was 4.7). Jung et al. (2003) and Pedreschi et al. (2004) achieved a decrease in acrylamide formation by pre-treating potato slices with citric acid, thus decreasing the pH from about 6.2 to 4.9.

pH in raw potato has been measured directly in the filtered juice obtained after washing and crushing the samples with a pH meter (Pardo et al., 2000; Jung et al., 2003). Nourian et al. (2002) measured pH with in a supernatant obtained by homogenizing 100 g of diced potatoes with 100 g of distilled water and filtering.

2.3.3. Specific gravity

Specific gravity (total solids) is the first criterion used in the industry to choose a good potato chipping cultivar. High specific gravity is an indication that the raw potatoes will produce high chip volume due to high dry matter content. For instance, for every 0.005 increase in specific gravity, an additional pound of chips can be obtained per 100 pounds of potatoes. Moisture in raw potatoes is related to the specific gravity: the higher the moisture, the lower the total solids and the lower the specific gravity (Lusas &

Rooney, 2001). The specific gravity in potatoes is very variable, and may range between 1.050-1.106 (Snack Food Association, 1991).

There are several methods to determine the specific gravity of potato tubers. One method is the weight in air/weight in water method (Gould, 1995). Representative potato samples are weighed in air, then in water, and the specific gravity is calculated using the following equation:

$$S.G. = \frac{weight\ in\ air}{weight\ in\ air - weight\ in\ water} \qquad [2\text{-}1]$$

The brine method consists of determining the specific gravity by floating potatoes in brine solutions of known salt concentrations (Lusas & Rooney, 2001). Potatoes will float if they have low specific gravity, and sink if it is high; they will remain suspended in the brine if their specific gravity is the same (Gould, 1995b).

The potato hydrometer method consists of using a standard potato hydrometer available from the Snack Food Association (Lusas & Rooney, 2001). With this method, the reading is taken directly from the hydrometer to obtain an average value for 8 lbs of potato.

The specific gravity could also be estimated using composition data:

$$SG = \frac{\rho_{potato}}{\rho_{water}}; \frac{1}{\rho_{potato}} = \frac{1}{\Sigma \frac{x_1}{\rho_1} + \frac{x_2}{\rho_2} + .. + \frac{x_n}{\rho_n}} \qquad [2\text{-}2]$$

where SG is the specific gravity, ρ_{water} is 1000 kg/m^3, x_1, $x_{2\ldots}$ x_n are fractions of the components in the potato, and ρ_1, $\rho_2\ldots$ ρ_n are the densities of components 1, 2, … n (Choi & Okos, 1986).

2.3.4. Oil content

Potato chips are among the foods with the highest oil content, which can range between 33-38% (Moreira et al., 1999). Since a considerable amount of frying oil is absorbed by the food, the quality of this oil is very important for the final product quality (Moreira et al., 1999).

Frying oil quality can be affected by the high temperatures used in the process (Moreira et al., 1999). Frying, when compared to other processing or handling methods, has the greatest potential for causing chemical changes in fat. During frying, several compounds are produced from the oil, including: volatiles, nonpolymeric polar compounds of moderate volatility, dimeric and polymeric acids, dimeric and polymeric glycerides, and free fatty acids (Fennema, 1996).

Moreira & Barrufet (1998) suggested that the most important oil absorption conditions are product initial moisture content and cooling air temperature. Higher initial moisture content resulted in higher oil content chips. Thus, the final oil content of tortilla chips having an initial moisture content of 41.44% w.b. and 27.52% w.b. was 32% and 22.5%, respectively. Decreasing the cooling air temperature by 33% and by 67% resulted in 12% and 24% more oil uptake, respectively, for 120 s of cooling. In addition, they found that increasing the pore radius from 2 μg to 15 μg resulted in a 9% decrease in oil uptake. Also, decreasing the effective interfacial tension by 42% and by 55%, resulted in 3% and 1% decrease in oil absorption, respectively.

Moreira et al. (1997) studied the factors affecting oil uptake in tortilla chips during deep-fat frying by using different conditions (baking time, frying oil temperature,

particle size distribution, and oil quality). They reported that higher initial moisture content and smaller particle size produced a final product with higher oil content. Tortilla chips that had initial moisture contents of 111% and 38.26% d.b. had a final oil content of 46% and 30% d.b., respectively. Tortilla chips produced from 100% fine and from 100% coarse particles resulted in final oil contents of 46% and 28%, respectively. They also found that the ratio of water evaporated and the final oil content was independent of the oil temperature used. Oil quality affected its distribution in tortilla chips, but did not affect the final total oil content.

Some authors suggest that, since the surface of the product is important for fat uptake, the application of a coating is a good way to reduce this uptake (Mellema, 2003). Rimac-Brncic et al. (2004) achieved reduced oil content potato strips by blanching in water solutions of calcium chloride or citric acid (28% and 15% oil content reduction, respectively) and by immersion in some carboxymethyl cellulose derivatives solutions (32% oil content reduction). Garcia et al. (2002) coated potato strips with methylcellulose and hydroxypropylmethylcellulose, achieving up to 40 % reduction in oil uptake.

The treatment food receives after being removed from the frying medium is very important to achieve the desired final oil content; proper shaking and draining of the food and proper frying temperature can help reduce the oil uptake (Moreira et al., 1997).

Another way to reduce oil content in food products is by vacuum frying. Garayo and Moreira (2002) studied the effects of oil temperature and vacuum pressure on the final oil content of potato chips, concluding that the final oil content was higher in chips

fried under atmospheric conditions than in those fried under vacuum (66% d.b. vs. 37% d.b).

Oil content has been determined in fried foods by using several methods, which can be classified as extraction, hydraulic press, refractometric method, and NIR spectroscopy (Moreira et al., 1999).

Extraction methods consist on extracting the dried and ground material with a light petroleum fraction or diethyl ether in a continuous extraction apparatus, of which there are basically two types: the Bolton or Bailey Walker type and the Soxhlet type. In the first type, condensed solvent drips onto the sample in a porous filter container, around which passes the hot solvent vapor. The Soxhlet type, on the other hand, gives intermittent extraction with excess of fresh condensed solvent; the Soxhlet extraction method has been widely used for the determination of oil content in foods (Rimac-Brncic, S. et al., 2004; Moreira et al., 1997) The Soxtec apparatus is a combination of the two types previously described and provides a faster way of determining fat in foods (Kirk & Sawyer, 1991); Garayo and Moreira (2002) used this apparatus to determine the oil content in potato chips.

Extraction may also be achieved by pressing the sample with a hydraulic system; however, this method has to be calibrated against the Soxhlet method (Moreira et al., 1999).

The refractrometric method involves grinding the sample, mixing with a solvent, centrifuging twice, and reading the refractive index of the supernatant with a refractometer at 25^{o}C (Moreira et al., 1999).

Near Infrared Reflectance (NIR) takes advantage of the property of chemical bonds to absorb energy. The amount of energy that is reflected by the bonds can be correlated with the quantity and types of functional groups present in the product (Moreira et al., 1999). NIR has also been used to determine quality parameters in raw products such as dry matter and sugar content (Scanlon et al., 1999).

2.3.5. Color

Color in a food product influences consumer acceptability. It is the major, if not the most important, attribute of foods, since it is the first to be evaluated by consumers (Fennema, 1996).

Color in processed products such as potato chips can be affected by several factors including product composition and processing conditions (Rodriguez-Saona & Wrolstad, 1997). For instance, common browning of foods during heating occurs when reducing sugars and a free amino acid or amino group react in the Maillard reaction (Fennema, 1996).

Marquez and Añon (1986) studied color development during potato frying, finding that both reducing sugars and amino acids participated in the color development of fried potatoes, reducing sugars being the limiting factor.

Rodriguez-Saona and Wrolstad (1997) used a potato model system that consisted of infusing potato slices with predetermined amounts of sucrose, reducing sugars, ascorbic, chlorogenic and amino acids (asparagine and glutamine) to evaluate the influence of these potato constituents on browning of potato chips. They found that

reducing sugars had the biggest influence on lightness, producing the brighter colors when they were absent; however, reducing sugars did not entirely predict color quality when present at low concentrations. In fact, they found a positive correlation between ascorbic acid content and potato chip color development, and a negative association of ascorbic acid, fructose, a chlorogenic acid isomer, glucose and glutamine. Sucrose and chlorogenic acid did not affect the color of the chips, and amino acids had only minor effects.

Frying temperature and thickness of the potato strips can also affect the color of fried potatoes; higher temperatures and thicker strips intensify the color change (Krokida et al., 2001). Garayo and Moreira (2002) found that vacuum frying produced significantly lighter potato chips than when frying under atmospheric pressure.

Several methods can be used to assess the color in potato chips. Potato chip color has been mainly measured using a spectrocolorimeter (Rodriguez-Saona & Wrolstad, 1997; Pedreschi et al., 2005). Another widely used, somewhat subjective method for determining acceptable colors is the Snack Food Association Potato Chip Color Chart (Sowokinos et al., 1987). Chips are rated on a scale of 1 to 5, 1 being a light golden color, and 5 being very dark and bitter (Gould, 1995a). Video Image Analysis has also been used to measure color in potato chips (Segnini & Dejmek, 1999; Scanlon et al., 1994).

2.3.6. Texture

Texture is one of the most significant quality attributes in food products since it makes a dominant contribution to the overall quality and acceptability (Kayacier & Singh, 2003). Texture in a food product can be determined by instrumental analysis and by sensory evaluation (Steffe, 1996). Determining texture by instrumental analysis is easy to perform, simple to reproduce, and less time consuming than sensory analysis (McCormick, 1988). Texture Profile Analysis consists in generating and interpreting texture profile information with either instrumental or sensory means (Steffe, 1996). Hardness is one of the various parameters that can be identified from a texture profile curve, and it is defined as the force at maximum compression during the first bite. Popular terms describing hardness include soft, firm and hard (Steffe, 1996).

Several authors have used the Texture Analyzer (Texture Technologies Corp., New York) to determine texture properties of foods. Moreira et al. (1997) used a Texture Analyzer compression test to characterize changes in tortilla chips during frying: a cylindrical probe of 0.203 cm in diameter and a cylindrical base with an outside diameter of 25.5 cm and a hole of 19 mm was used in a bite compression test with a probe velocity of 10 mm/s. Kawas (2000) used a similar procedure to measure texture of tortilla chips: a ¼ inch ball probe traveled at a downward velocity of 0.1 mm/s until it broke the sample; the sample was located on an 18 mm diameter hollow cylindrical base. Fracturability was determined as the first peak of the force vs. distance curve. Garayo (2001) used a rupture test on fried potato chips. He used the same approach used by

Kawas (2000); hardness of the chips was determined by finding the maximum force at compression (Steffe, 1996).

2.3.7. Acrylamide determination

Acrylamide content of foodstuffs has been determined mainly by Gas Chromatography-Mass Spectrometry (GC-MS) and Liquid Chromatography-Mass Spectrometry/Mass Spectrometry (LC-MS/MS) methods (Wenzl et al., 2003).

Gas Chromatography can be conducted either with or without previously derivatizing acrylamide. Derivatization of acrylamide is accomplished mostly by bromination. This method has the advantage that a more volatile compound is produced and the selectivity of determination is increased. The disadvantage is that derivatization may be time consuming (Wenzl et al., 2003). Some authours have derivatized their samples through bromination by using potassium bromide, hydrobromic acid, and saturated bromine water (Castle et al., 1993; Tareke et al., 2002).

Extraction of acrylamide by LC/MS/MS involves mainly water at room temperature and the addition of an internal standard to keep track of any losses during extraction and clean up (Wenzl et al., 2003). Samples are then shaken and centrifuged before the clean-up step; centrifuge speed and time vary (Tareke et al., 2002; Roach et al., 2003; Yasuhara et al., 2003; Rosen, J. & Hellenas, K., 2002). Clean up has been accomplished by using combinations of solid-phase extraction cartridges (Wenzl et al., 2003). Reversed-phase chromatography has been commonly used for the chromatographic separation of acrylamide; an alternative to reversed-phase is ion-

exchange chromatography (Wenzl et al., 2003). After chromatographic separation, tandem mass spectrometry has usually been used (Rosen & Hellenas, 2002; Roach et al., 2003; Tareke et al., 2002; Becalski et al., 2004). This method is run in MRM mode (multiple reaction-monitoring), which has a high selectivity. In MRM, the transition from a precursor ion to a product ion is monitored (Wenzl et al., 2003). The product ion is generated by collision of the precursor ion that is generated in the first quadrupole with argon in the second quadrupole, and is monitored in the third quadrupole. In most cases, the transition 72→55 is monitored for acrylamide (Pedreschi et al., 2004; Becalski et al., 2004). Tareke et al. (2002) monitored transitions of m/z 72 to 54 and 55. Others have monitored the transitions of m/z 72 to 72, 55, 54, 44, and 27 to confirm acrylamide identity (Rosen & Hellenas, 2002; Ahn et al., 2002; Becalski et al., 2003). For quantitation of acrylamide, the transition 73→56 is used when the internal standard is [$^{13}C_1$]-acrylamide, and 75→58 is used for [$^{13}C_3$]- and [D_3]-acrylamide (Wenzl et al., 2003).

CHAPTER III

EFFECT OF CULTIVAR, FRYING SYSTEM AND FRYING CONDITIONS ON ACRYLAMIDE FORMATION IN POTATO CHIPS

3.1. Materials and Methods

3.1.1. Raw potato handling

All raw potatoes were handled in a similar way throughout the experiment. Seven different cultivars were used in this study: Atlantic, Shepody, Innovator, White Rose, ATX 84706-2Ru, NDTX 4930-5W, and ATX 85404-8W. Potatoes were provided by the Texas A & M University Potato Variety Development Program. Table 3-1 describes the main uses for these cultivars, which are pictured in Figure 3-1.

Table 3-1. Characteristics and uses of selected potato cultivars.

Cultivar	Characteristics	Uses
Innovator	Tubers long oblong; russeted tan colored skin; pale yellow to yellowish white flesh.	Suitable for chipping and fresh market.
NDTX4930-5W	Round White. Parentage (ND860-2 x A7961-1).	Good for chipping.
ATX85404-8W	Round White. Parentage (Gemchip x ND860-2).	Good for chipping.
Atlantic	Tubers round to oval; buff-colored skin; white flesh.	Excellent for chipping and French frying. Good for boiling and baking.
Shepody	Tubers oblong to long; smooth to light netted white skin; white flesh.	Unsuitable for chipping. Excellent for boiling, baking, and French frying.
ATX847806-2Ru	Oblong Russet. Parentage (A7938-1 x COA7906-5).	Good for boiling and baking. Excellent for French frying.
White-Rose	Tubers large, long, elliptical, flattened, usually irregular; smooth, white skin; white flesh.	Unsuitable for chipping. Good for boiling and baking.

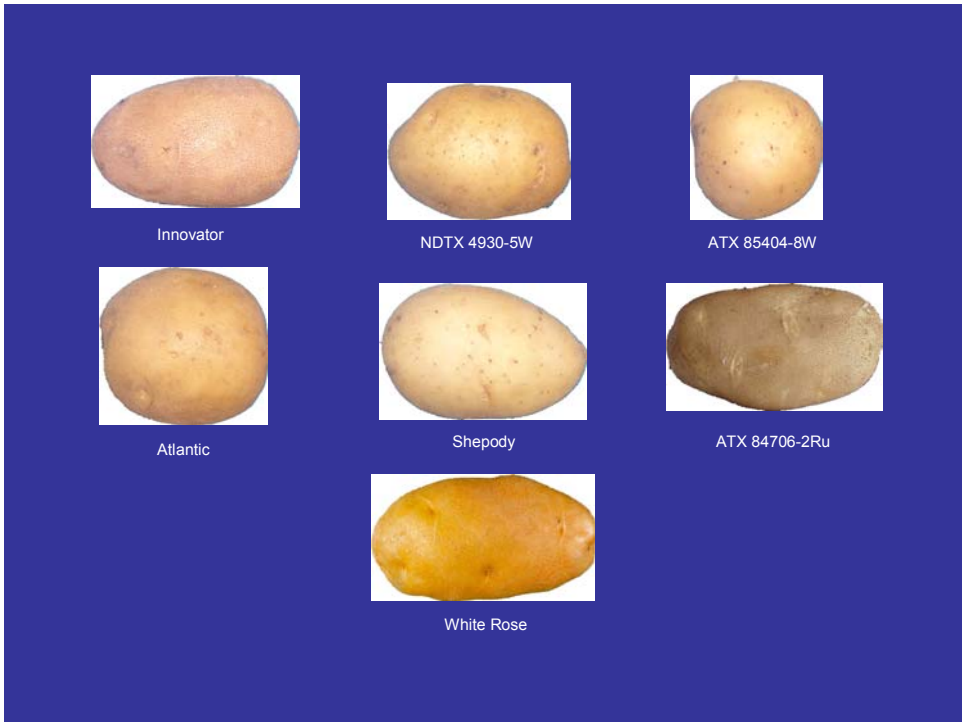


Figure 3-1. Selected potato cultivars.

After harvest, potatoes were stored at 10^{o}C. Before processing, they were left at room temperature for approximately 3-4 days to allow reconditioning and to lower the reducing sugar content. Potatoes were then peeled, sliced (1.5 mm average thickness), and given a round shape with a 4.5 mm diameter cutter mold. Before frying, the potato slices were soaked in tap water and blotted dry using paper towels.

3.1.2. Raw potato properties

Specific gravity, moisture content, reducing sugar content, and pH of raw potatoes were measured before processing.

3.1.2.1. Specific gravity

The specific gravity of the potatoes was measured using the weight in air/weight in water method (Gould, 1995b), Eqn (2-1).

Each potato was weighed individually in a system that consisted of a basket placed under water. The basket was connected by a string to a balance. The weight in air was obtained by placing the potato directly on the balance. The weight in water was determined by submerging the potato in the basket under water. The test was conducted in duplicate.

3.1.2.2. Moisture content of raw potatoes

The moisture content of the raw potatoes was determined by drying 5 g of the product in a convection oven to a constant mass for 72 hours at 105^{o}C (AACC, 1986). The test was performed four times. The weights were recorded before and after drying, and the moisture content, wet basis (MC_{wb}), was calculated as follows:

$$MC_{wb} = \frac{weight\ wet - weight\ dry}{weight\ wet} \qquad [3\text{-}1]$$

The moisture content, dry basis (MC_{db}), was defined as:

$$MC_{db} = \frac{weight\ wet - weight\ dry}{weight\ dry} \qquad [3\text{-}2]$$

3.1.2.3. pH

Three to four potato slices were mashed to extract their juice. The pH was then measured with a pH meter (Cole Parmer, Vernon Hills, IL) previously calibrated using buffers at pH of 4, 7, and 10. The test was performed in duplicate.

3.1.2.4. Reducing sugars

The same juice used for determining the pH was used for evaluating the reducing sugars. Glucose test strips (Precision Labs, Inc., West Chester, OH) were submerged in the solution for 1-2 seconds. Color changes in the strips were compared to a color chart, where values are correlated to reducing sugar content. The test was conducted in duplicate.

3.1.3. Vacuum frying experiments

The setup for the vacuum fryer used in this experiment was the one assembled by Garayo (2001). This fryer is located in the Food Engineering Laboratory of the Biological and Agricultural Engineering Department at Texas A & M University (Figure 3-2). An electric pressure cooker made of cast aluminum served as the vacuum vessel. This vessel can withstand an internal pressure of 2 atm. The maximum temperature that can be generated is 145^{o}C at maximum capacity of 6 L.

Vacuum is achieved in the vessel with a dual seal vacuum pump (model 1402 Welch Scientific Co., Skokie, IL) that can generate a vacuum up to 10 Torr. To prevent water from damaging the pump, a condenser that holds a 50-50 mix of ethanol and water was connected to the vessel.

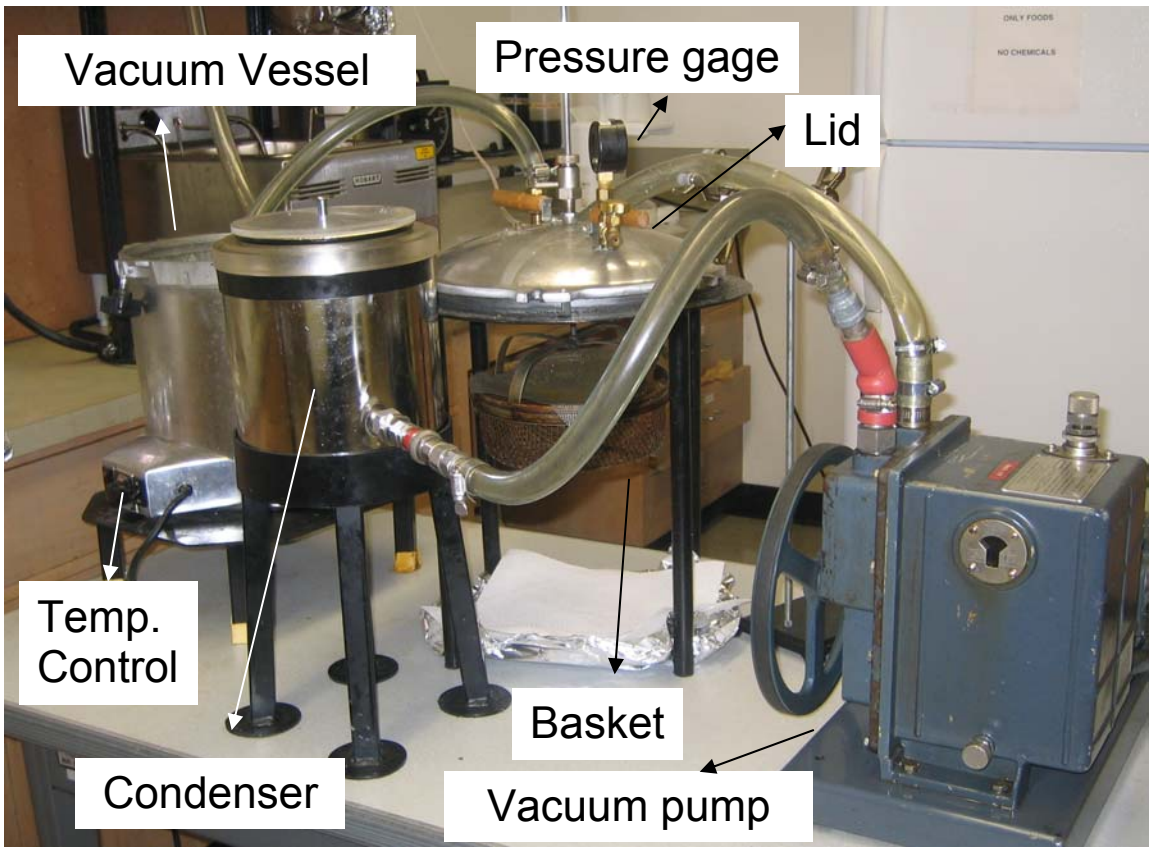


Figure 3-2. Vacuum frying system setup.

The frying process consisted of placing the potato slices into the basket, closing the lid, then depressurizing the vessel. When the pressure in the vessel achieved 10 Torr, the basket was lowered to a point where the slices were submerged in the hot oil. After the potatoes were fried, the basket was raised and the vessel pressurized. The potatoes

were then blotted dry with paper towels to remove excess oil. Table 3-2 shows the different treatments used for the vacuum frying experiments.

Table 3-2. Time and temperature conditions for the vacuum frying experiments.

VARIETY	TEMPERATURE (^{o}C)	TIME (s)
Innovator	118	480
NDTX4930-5W	118	480
ATX 85404-8W	118	480
Atlantic[a]	118	60,120,240,360,480,600
Atlantic	125	60,120,240,360,480,600
Atlantic	140	60,120,240,360,480
Shepody	118	480
ATX 847806-2Ru	118	480
White-Rose	118	480

[a]Cultivar chosen to study the effect of time and temperature on potato quality attributes (PQA).

3.1.4. Traditional frying experiments

An electric-fired type fryer (Hobart model HK3-2, Hobart Corp, Troy, OH) with an oil capacity of 7.5 L was used in this study. The frying process consisted of immersing 6-8 potato slices into the hot oil for a specified time. The experimental conditions for the traditional frying experiments can be seen in Table 3-3.

Table 3-3. Time and temperature conditions for traditional frying experiments .

VARIETY	TEMPERATURE (^{o}C)	TIME (s)
Innovator	165	240
NDTX4930-5W	165	240
ATX 85404-8W	165	240
Atlantic[a]	150	60,120,180,240,300,360
Atlantic	165	60,120,180,240,300,360
Atlantic	180	30,60,120,150,180,240
Shepody	165	240
ATX 847806-2Ru	165	240
White-Rose	165	240

[a]Cultivar chosen to study the effect of time and temperature on potato quality attributes (PQA).

3.1.5. Final product quality attributes (PQA)

3.1.5.1. Moisture content

About 3 g of ground potato chips were dried at 105^{o}C (AACC, 1986) in a convection oven for 24 hours. The weights were recorded before and after drying, and the final moisture content was calculated using Eqns (3.1) and (3.2). The test was performed in duplicate.

3.1.5.2. Oil content

The oil content of the potato chips was determined using the Soxtec System HT extraction unit (Pertorp, Inc., Silver Spring, MD) with petroleum ether (AACC, 1986). Three grams of ground potato were weighed (Wi), placed on a cellulose extraction timble (model 2800256, Whatman, England), and covered with a cotton ball. Six extractions were performed at each time. Aluminum cups were dried for 15 minutes at 105^{o}C and cooled in a dessicator for 20 minutes. The cup weight (W_1) was recorded, and 50 ml of petroleum ether was added to each cup.

The samples were then subjected to extraction, and the oil from the chips collected. Petroleum ether was evaporated by turning the air on and setting the unit to the "evaporation" position. To make sure all the petroleum ether was evaporated, the cups were dried in a convection oven for 20 minutes at 105^{o}C. The cups were then cooled in a desiccator for 20 minutes, and the final cup weight was recorded (W_2). Finally, the oil content (wet basis) was found by the relationship:

$$OC\ (\%) = \frac{W_2 - W_1}{W_i} \times 100 \qquad [3\text{-}3]$$

Tests were conducted in triplicate.

3.1.5.3. Color

The color of the potato chips was measured using a Hunter Lab Colorimeter Labscan XE (Hunter Associates Laboratory, Reston, VA). The Hunter Lab notation was used, where *L* denotes levels of lightness or darkness (0 for black, 100 for white), *a* represents redness (positive values) or greenness (negative values), and *b* yellowness (positive values) or blueness (negative values). Ten (10) potato chips were evaluated per treatment, and two readings were recorded per potato chip by rotating them 180^{o}. The colorimeter was calibrated using a white and a black plate. The same background was used every time.

3.1.5.4. Hardness

The texture of the potato chips was determined by a rupture test to determine the maximum force at compression, which is defined as "hardness" (Steffe, 1996). The equipment used was the TA-XT2 Texture Analyzer (Texture Technologies Corporation, Scardale, NY).

This test consisted of applying a force to a potato chip placed on an 18 mm hollow cylinder. The speed at which the probe approached the sample was 0.1 mm/s. The force applied was 0.7 N, and the test was finished 4 mm after the potato chip fractured.

Hardness was measured for different levels of moisture content under each treatment and frying method. However, if the samples were not brittle enough, the texture values were discarded. Twenty (20) potato chips were analyzed each time.

3.1.5.5. Acrylamide Content

Acrylamide content in potato chips fried at different times and temperatures was determined using the protocol developed by Roach et al. (2003) with some modifications (Figure 3-3).

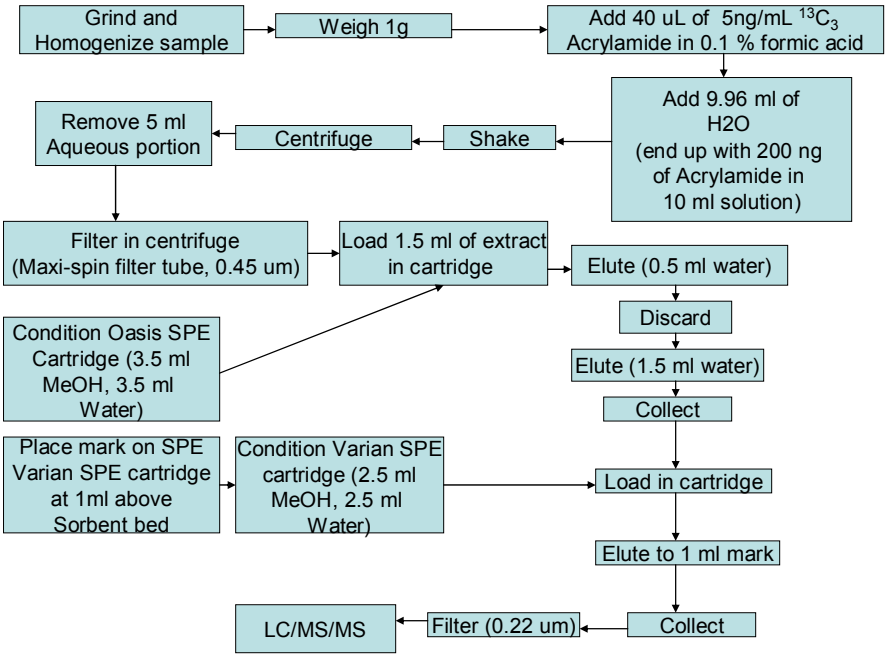


Figure 3-3. Acrylamide extraction flow chart (Roach et al., 2003).

About 1 g of ground potato chips was placed in a 50 ml conical tube. Then, 9.96 ml of H_2O and 40 µl of the internal standard (5 ng/ml of $^{13}C_3$ acrylamide in 0.1 % formic acid) were added to the sample. Samples were thoroughly mixed for 20 min with a rotating shaker. The samples were then centrifuged for 30 min at 9000 RPM. About 5 ml of the supernatant was immediately removed with a pipette, avoiding the oil on the top and the solids on the bottom. The supernatant was filtered by centrifuging (5000 RPM for 30 min) with Maxi-spin filter tubes (0.45 µm PVDF-Alltech, Alltech Assoc., Deerfield, IL). If the filters clogged, the remaining liquid on the top of the filter was poured into another tube and was centrifuged again until most of the liquid had gone through.

Solid phase extraction cartridges (Oasis HLB 6 cc solid phase extraction, Waters Corp., Milford, MA) were conditioned with 3.5 ml of methanol followed by 3.5 ml of water. The cartridges were loaded with 1.5 ml of the supernatant and allowed to go through the sorbent material, and 0.5 ml of H_2O was used for clean-up. The column was then eluted with 1.5 ml of water, and the eluant was collected.

A mark was placed at a height of 1 ml above the sorbent bed of the Varian SPE cartridge (Bond Elut – Accucat, mixed mode, C8, SAX and SCX; 3-ml solid-phase extraction cartridge, Varian Inc., Harbor City, CA). Varian SPE cartridges were conditioned with 2.5 ml of methanol followed by 2.5 ml of water. They were then loaded with the eluant previously collected, and were allowed to elute until the 1 ml mark. Portions were collected and filtered with 0.22-μm filters (Fisherbrand, Fisher Scientific Intl., Pittsburg, PA) and were analyzed by LC/MS/MS at room temperature using a Waters C-48 column (Waters Corporations, Milford, MA). LC/MS/MS analysis was performed by Dr. Shane Tichy, from the Department of Chemistry at Texas A & M University.

3.1.6. Data analysis

Determination of non-linear and linear regression parameters was conducted using the Marquard Compromise iteration method in the graphics software package PlotIt (version 3.2, 1999). Differences among treatments were detected with the SPSS software (version 12.0.1 for Windows, 2003) using Duncan's multiple range tests. Statistical significance was expressed at the $P<0.05$ level.

3.2. Results and Discussion

3.2.1. Effect of different potato cultivars on final product quality attributes (PQA)

3.2.1.1. Effect of cultivars and frying methods on acrylamide formation in potato chips

Table 3-4 shows the raw potato properties of the seven potato cultivars. The different concentrations of acrylamide in the varieties processed under vacuum and atmospheric conditions can be found in Table 3-5 and Figure 3-4.

Table 3-4. Raw potato properties.

Cultivar	Specific gravity	Sugar (%)	pH	MC_i [a] (%wb)
Innovator	1.07 ± 0.01	0.28 ± 0.11	6.05 ± 0.06	83.7 ± 1.5
NDTX4930-5W	1.06 ± 0.01	0.08 ± 0.04	6.13 ± 0.07	82.2 ± 2.3
ATX 85404-8W	1.07 ± 0.01	0.03 ± 0.04	6.22 ± 0.07	81.5 ± 0.7
Atlantic	1.08 ± 0.01	0.22 ± 0.09	6.06 ± 0.10	78.1 ± 1.0
Shepody	1.07 ± 0.01	0.18 ± 0.11	6.04 ± 0.05	81.9 ± 1.2
ATX 84706-2Ru	1.06 ± 0.01	0.13 ± 0.04	6.19 ± 0.03	81.9 ± 1.4
White Rose	1.07 ± 0.01	0.38 ± 0.18	6.12 ± 0.03	79.4 ± 1.4

[a]Initial moisture content in potatoes. All tests were performed in duplicate.

Table 3-5. Acrylamide content of different potato cultivars fried under vacuum and atmospheric conditions.

Frying Method	Traditional[a]		Vacuum[b]	
Cultivar	**MC_f[c] (%wb)**	**Acrylamide[d] (ppb)**	**MC_f (%wb)**	**Acrylamide (ppb)**
Innovator	0.97 ± 0.08	552 ± 24 b,A	1.81 ± 0.30	35 ± 3 b,c,B
NDTX4930-5W	1.55 ± 0.02	372 ± 50 a,A	2.25 ± 0.22	ND[e] a,B
ATX85404-8W	1.30 ± 0.21	391 ± 25 a,A	1.79 ± 0.01	25 ± 3 b,B
Atlantic	1.14 ± 0.02	466 ± 34 a,b,A	1.68 ± 0.01	75 ± 13 d,B
Shepody	1.57 ± 0.01	536 ± 21 b,A	1.54 ± 0.01	38 ± 2 b,c,B
ATX84706-2Ru	1.04 ± 0.01	1554 ± 99 c,A	1.56 ± 0.01	44 ± 9 c,B
White Rose	1.56 ± 0.05	3899 ± 111 d,A	1.38 ± 0.28	242 ± 30 e,B

[a]Fried for 4 min at 165°C, atmospheric conditions. [b]Fried for 8 min at 118°C, vacuum conditions (10 Torr). [c]Final moisture content in potato chips, run in duplicates. [d]Acrylamide values are means of 4 replicates; means followed by the same letter are not significantly different ($P<0.05$). Uppercase letters refer to frying method; lowercase letters refer to cultivar effect. [e]Acrylamide content not detectable.

The White Rose cultivar showed the highest acrylamide content (3899 ppb) when fried under the traditional treatment (atmospheric pressure at 165°C for 4 min), followed by ATX84706-2Ru (1554 ppb). Innovator, Shepody, and Atlantic follow with 552, 536, and 466 ppb, respectively. The cultivars with the lowest acrylamide content were NDTX4930-5W (372 ppb) and ATX85404-8W (391 ppb). Under the vacuum frying treatment (10 Torr at 118°C for 8 min), all varieties showed significant lower acrylamide contents ($P<0.05$) compared to potato chips fried under traditional frying. Once again, White Rose showed the highest acrylamide content (242 ppb) among the cultivars fried under vacuum conditions.

There were no significant differences ($P<0.05$) among raw potato characteristics such as pH, specific gravity, and initial moisture content for the different cultivars. However, the reducing sugar content of the White Rose cultivar (0.38%) was

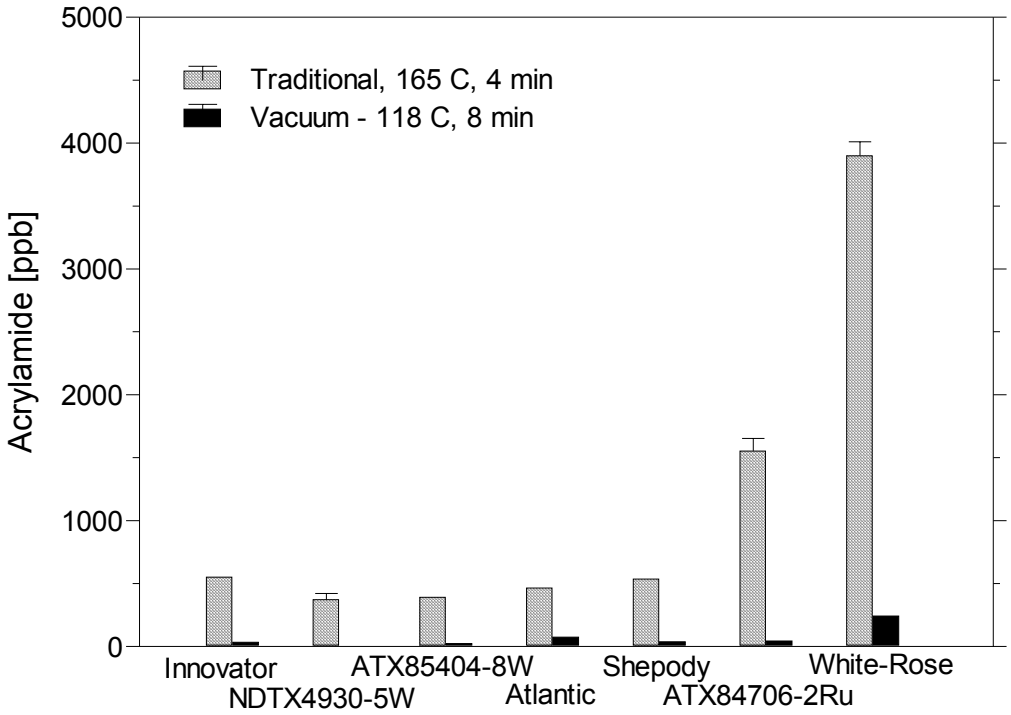


Figure 3-4. Effect of potato cultivar and frying method (vacuum and traditional) on acrylamide formation in potato chips.

significantly higher ($P<0.05$) than the reducing sugar content of the others. Because reducing sugars play an important role in the Maillard reaction, and hence on acrylamide formation (Mottram et al., 2002), the high accumulation of reducing sugars explains the higher content of acrylamide in White Rose under both frying conditions. However, this cultivar is used primarily for boiling and baking, but is unsuitable for chipping (Table 3-1) because it develops dark colors during frying.

Table 3-5 shows that the two cultivars with the lowest reducing sugar content, NDTX4930-5W and ATX85404-8W, also had the lowest acrylamide concentrations under both frying conditions. These cultivars are good for chipping (Table 3-1) because they can handle high temperatures without excessive browning. Atlantic, Shepody, and Innovator had intermediate reducing sugar contents, and consequently intermediate acrylamide levels when compared to the other cultivars. ATX84706-2Ru had a high acrylamide level (1554 ppb) when fried under atmospheric conditions, in contrast to the other cultivars with similar sugar content. This cultivar may have had higher asparagine content than the other cultivars that had similar reducing sugar concentration, hence the higher acrylamide content. On the other hand, ATX84706-2Ru had very low acrylamide content (44 ppb) when fried under vacuum conditions, which is comparable to the acrylamide content of potato chips produced from the other cultivars due to the lower temperature used (140^{o}C), since acrylamide formation increases as temperature increases above 100^{o}C (Taubert et al., 2004; Mottram et al., 2002).

3.2.1.2. Effect of potato cultivar and frying method on color changes in potato chips

The effect of potato cultivar and frying technique on color of potato chips is shown in Table 3-6, and in Figures 3-5 to 3-7. Figure 3-5 shows the effect of these two variables on the *L* parameter, which may range from 0 to 100 (0= black to 100= white). For most cultivars, traditionally fried potato chips had a higher *L*-value, which indicates a lighter color. The only cultivar that did not show any significant differences on *L*-value between traditional and vacuum frying was ATX85404-8W. For White Rose, the *L*-value for the traditionally fried chips (38.3) was significantly lower ($P<0.05$) than the *L*-value for the vacuum fried product (46.2).

A high *a*-value is an indication of overcooked potato chips, which in Hunter Lab notation may range from -30 to 30. Table 3-6 and Figure 3-6 show that all potato chips, except for traditionally fried White Rose which had a positive value of 0.309, had a negative *a*-value or a more greenish than reddish color. It is interesting to note that White Rose was also the one that had the highest acrylamide level (3899 ppb) and the highest reducing sugar content (0.38%). High reducing sugar content contributes to dark fry colors due to enhanced Maillard reaction (Khanbari & Thompson, 1993). The *a*-value for vacuum fried chips was lower (more greenish) than for the traditionally fried potatoes (0.309 and -2.718 for White Rose, respectively)

The *b*-value was significantly different ($P<0.05$) for the chips produced under different frying methods (Table 3-6 and Figure 3-7). This value refers to the blue-yellow chromaticity, which may range from -40 to 40. For all potato cultivars, the traditionally

fried chips had a higher *b*-value than the vacuum fried chips. Also, differences in *b* values could be observed among cultivars fried using the same technique.

Table 3-6. Effect of frying method and potato cultivar on potato chip color.

Frying Method	**Traditional[a]**			**Vacuum[b]**		
Cultivar	**Color[c]**			**Color**		
	L	*a*	*b*	*L*	*a*	*b*
Innovator	51.12 d,A	-3.32 a,A	16.26 d,A	44.86 b,c,B	-4.05 a,B	15.09 e,B
NDTX4930-5W	50.34 c,d,A	-2.93 a,b,A	14.76 b,A	42.88 a,B	-2.95 c,A	8.29 a,B
ATX85404-8W	49.77 c,d,A	-3.16 a,b,A	15.50 c,A	49.64 e,A	-3.77 b,B	10.59 c,B
Atlantic	47.33 b,A	-2.82 b,A	15.16 b,c,A	43.61 a,b,B	-2.67 b,d,B	10.15 b,c,B
Shepody	48.91 b,c,A	-1.76 c,A	16.60 d,A	45.08 c,d,B	-3.11 c,B	9.71 b,B
ATX84706-2Ru	50.09 c,d,A	-1.72 c,A	17.25 e,A	43.87 a,b,c,B	-2.95 c,B	10.14 b,c,B
White Rose	38.33 a,A	0.31d,A	13.61a,A	46.25d,B	-2.72d,B	12.38 d,B

[a]Fried for 4 min at 165°C, atmospheric conditions. [b]Fried for 8 min at 118°C, vacuum conditions (10 Torr).[c]All color values (*L*,*a* and *b*) are means for 20 replicates. Means followed by the same letter are not significantly different (<0.05). Uppercase letters refer to frying method; lowercase letters refer to cultivar effect.

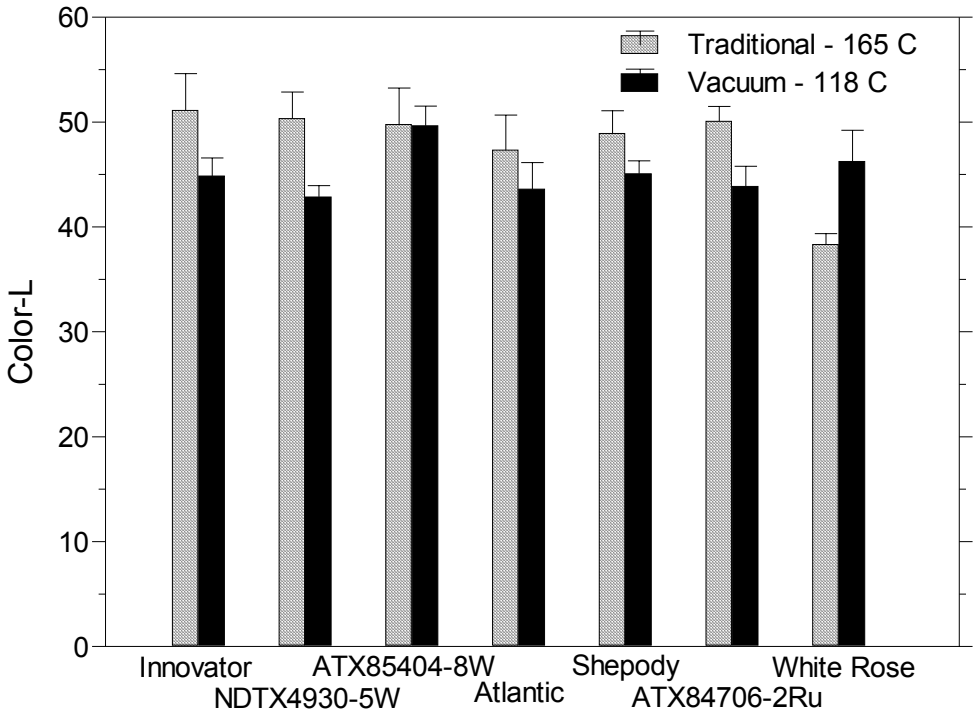


Figure 3-5. Effect of potato cultivar and frying method on lightness (*L*-value) of potato chips.

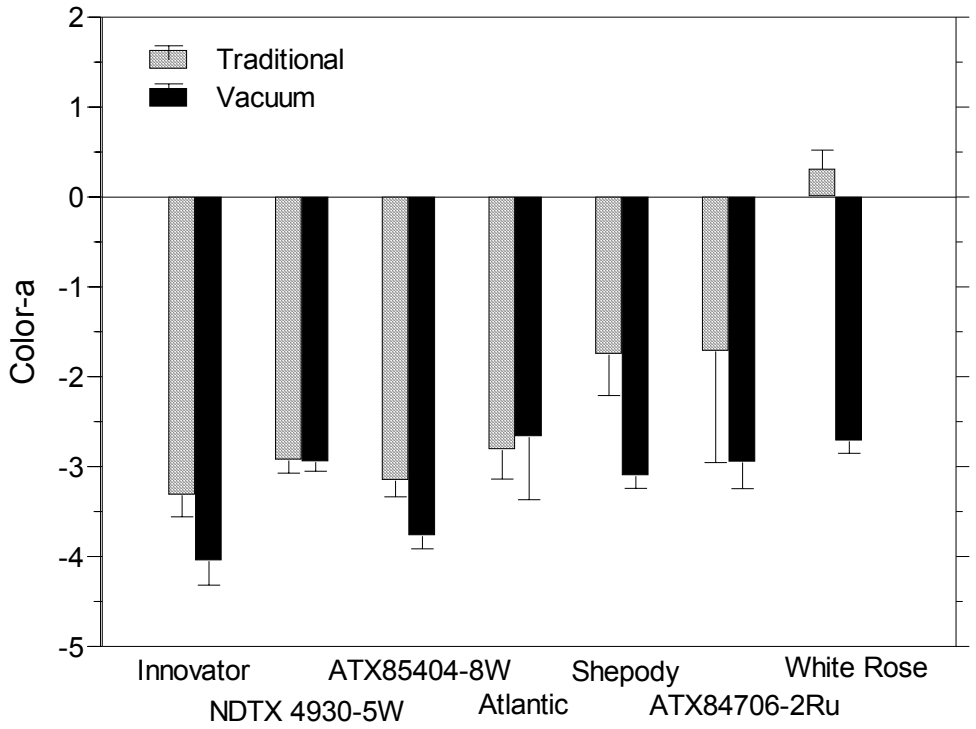


Figure 3-6. Effect of potato cultivar and frying method on the green-red chromaticity (*a*-value) of potato chips.

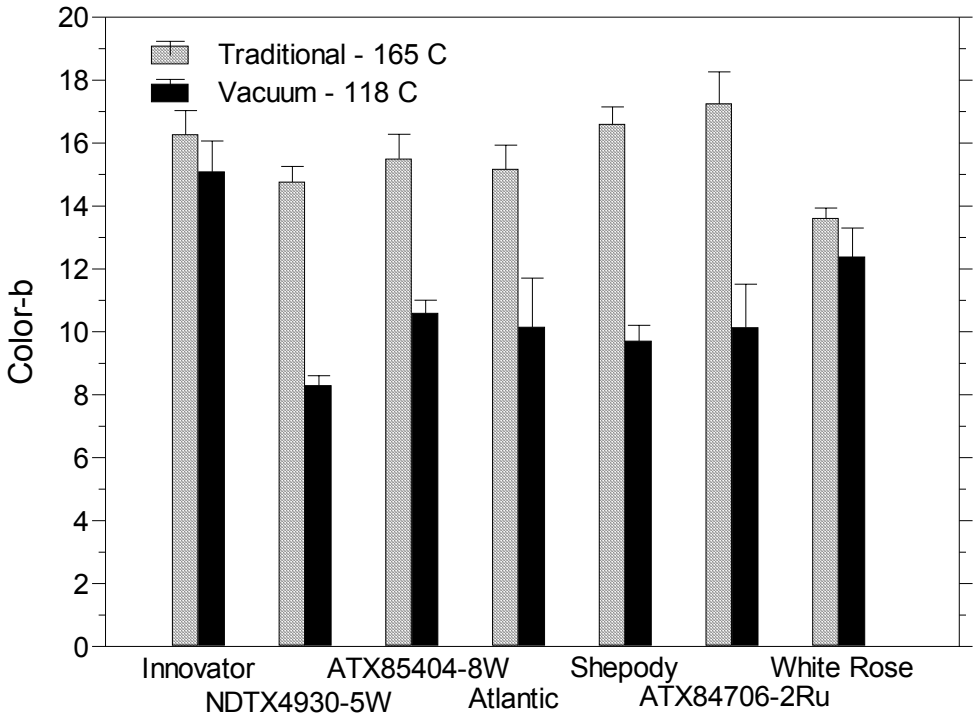


Figure 3-7. Effect of potato cultivar and frying method on the blue-yellow chromaticity (*b*-value) of potato chips.

3.2.1.3. Effect of potato cultivar and frying method on texture of potato chips

Texture varied slightly among cultivars, but there were no significant differences ($P<0.05$) when different frying methods were used (Table 3-7). This result confirms the findings of Garayo and Moreira (2002). White-Rose potato chips were the hardest under atmospheric and vacuum conditions among all the cultivars (3.0 N and 3.9 N, respectively).

Table 3-7. Effect of frying method and cultivar on texture of potato chips.

Frying Method	**Traditional[a]**	**Vacuum[b]**
Cultivar	**Hardness (N)**	**Hardness (N)**
Innovator	2.9 b,A	2.5 a,b,A
NDTX4930-5W	2.3 a,A	2.0 a,A
ATX85404-8W	2.7 a,b,A	2.6 a,b,A
Atlantic	2.2 a,A	2.9 b,A
Shepody	2.5 a,b,A	2.8 b,A
ATX84706-2Ru	2.4 a,A	2.4 a,b,A
White Rose	3.0 b,A	3.9 c,A

[a]Fried for 4 min at 165°C, atmospheric conditions. [b]Fried for 8 min at 118°C, vacuum conditions (10 Torr).[c]All values are means of 10 replicates. Means followed by the same letter are not significantly different (<0.05). Uppercase letters refer to frying method; lowercase letters refer to cultivar effect.

3.2.2. Effect of frying time and oil temperature on fried PQA

3.2.2.1. Effect of oil temperature and frying method on moisture loss of potato chips during frying

Figure 3-8 and Figure 3-9 show the moisture loss of potato slices (Atlantic) fried at different pressures (vacuum and atmospheric) and oil temperature. These curves exhibit typical drying profiles for food products. This finding confirms the observations of other authors that the loss of moisture during frying follows a typical drying profile (Garayo, 2001; Gamble et al., 1987; Shyu & Hwang, 2001). As explained by Van Arsdel et al. (1973), the first period during drying of food products is the "warm-up" period. The second period is characterized by a steady rate of evaporation per unit area of the exposed surface, and is known as the "constant-rate" phase. The moisture content of the product at which the constant-rate ends is known as the "critical" moisture content. The third phase is the "falling-rate", which continues until the equilibrium moisture content is reached.

The diffusion coefficient is a measure of molecular mobility. Hence, a higher moisture diffusion coefficient in potato slices means a faster drying rate (Datta, 2002). The diffusion coefficient of the potato slices or chips fried at different temperatures under vacuum and traditional frying was calculated using the method proposed by Brooker et al. (1992). The moisture diffusion equation for a flat plate can be described by:

$$MC_{db} = (M_o - M_e) * \left(\frac{8}{\pi^2}\right) \exp\left(-\frac{\pi^2 D_e t}{4a^2}\right) + M_e \qquad [3\text{-}4]$$

where MC_{db} is the moisture content in decimal dry basis (dec. d.b), M_o is the initial moisture content (dec. d.b.), M_e is the equilibrium moisture content (dec. d.b.), t is the frying time, and a is the thickness of the potato slice.

The values of D_e (diffusion coefficient) were obtained by using non-linear regression to fit the experimental drying rate curve (Table 3-8, Figure 3-8 and Figure 3-9).

As reported by Gamble et al. (1987) and Garayo and Moreira (2002), potato chips fried under atmospheric conditions resulted in an initial rapid fall, followed by a continuous drying period. Hence, the higher the temperature, the higher the diffusion coefficient (D_e) (Table 3-8). However, when comparing potato slices fried under atmospheric and vacuum frying, the drying rate did not depend only on temperature but on frying method. In potato slices fried under vacuum (10 Torr) at 140^{o}C the D_e was higher than in potato slices fried under traditional frying at 150^{o}C and 165^{o}C (2.26 x10^{-8} versus 1.70x10^{-8} and 2.09x10^{-8} m^2/s) (Table 3-8). This is due to the fact that during vacuum frying the boiling point of water is lowered, therefore it evaporates faster (Garayo & Moreira, 2002).

Table 3-8. Diffusion coefficients for potato chips fried under traditional and vacuum (10 Torr) frying at different temperatures.

Frying Method	**Traditional[a]**			**Vacuum[b]**		
Temperature (^{o}C)	**150**	**165**	**180**	**118**	**125**	**140**
M_o **d.b. (dec.)**	3.85	3.85	3.85	3.93	3.93	3.93
M_e **d.b. (dec.)**	0.007	0.007	0.007	0.010	0.010	0.010
D_e **(m^2/s)**	1.70×10^{-8}	2.09×10^{-8}	2.83×10^{-8}	1.15×10^{-8}	1.31×10^{-8}	2.26×10^{-8}
R^2	0.99	0.99	0.96	0.98	0.98	0.98

[a]Fried for 4 min at 165^{o}C, atmospheric conditions. [b]Fried for 8 min at 118^{o}C, vacuum conditions (10 Torr). Moisture tests were run in triplicates. Thickness of the potato slices for *De* calculations was 1.5×10^{-3}m.

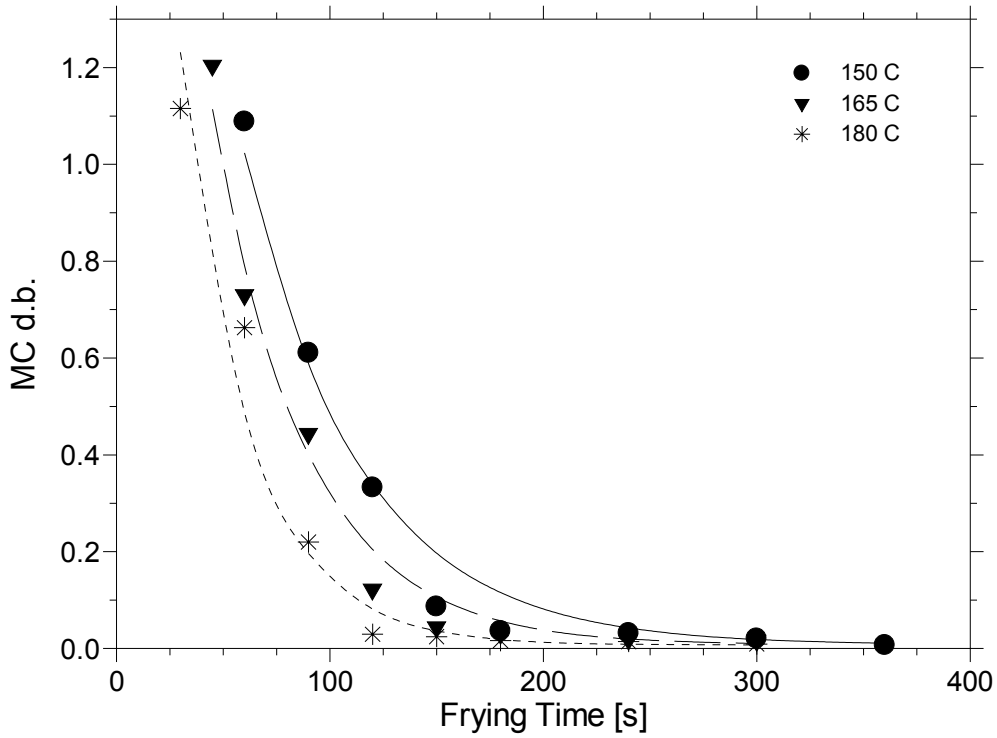


Figure 3-8. Drying rate of potato chips fried under traditional frying at different frying oil temperatures [150^{o}C, MC(t)=3.111*exp(-0.01864*t)+0.0071, R^2=0.99; 165^{o}C, MC(t)= 3.111*exp(-0.02296*t)+0.0071, R^2=0.99; 180^{o}C, MC(t)= 3.111* exp(-0.03108*t)+0.0071, R^2=0.96].

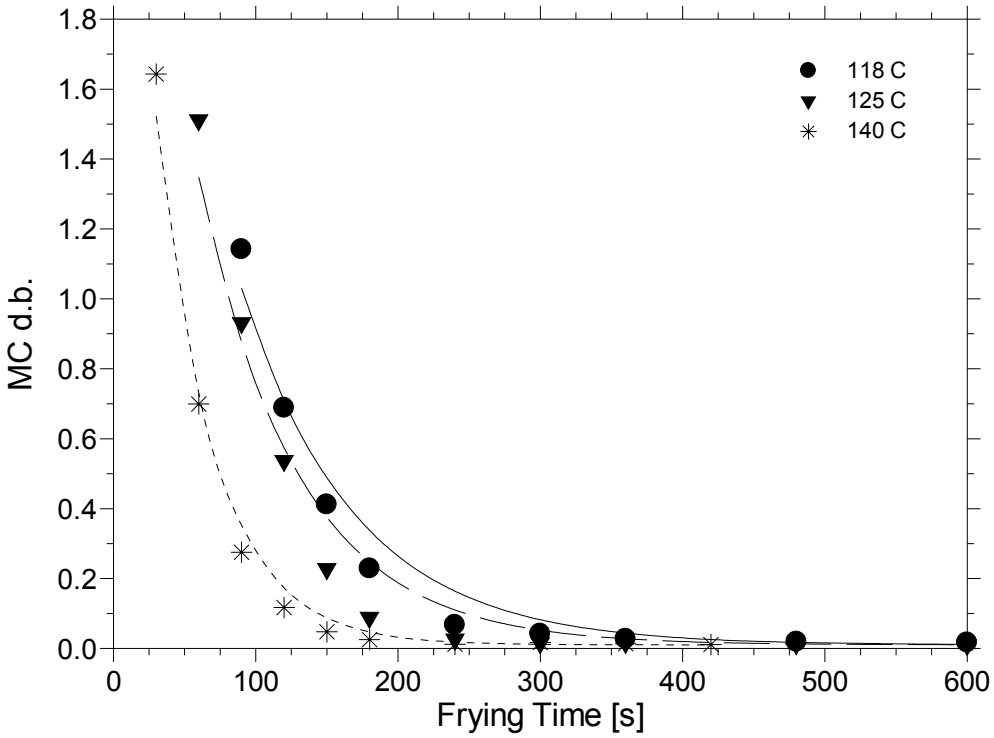


Figure 3-9. Drying rate of potato chips fried under vacuum frying (10 Torr) at different frying oil temperatures [118^{o}C, MC(t)=3.176*exp(-0.01261*t)+0.0102, R^2=0.98; 125^{o}C, MC(t)= 3.176*exp(-0.01441*t)+0.0102, R^2=0.98; 140^{o}C, MC(t)= 3.176* exp(-0.02473*t)+0.0102, R^2=0.98].

The influence of the frying oil temperature on the diffusion coefficient during traditional and vacuum frying was modeled using an Arrhenius-type equation:

$$D_e(T) = A \exp \frac{-E_a}{RT} \quad [3\text{-}5]$$

where A is the pre-exponential factor (1/s), E_a is the activation energy, T is the absolute temperature (K), and R is the universal gas constant (8.314 J/molK).

Equation (3.5) can be linearized as:

$$\ln D_e = \ln A - \frac{E_a}{RT} \quad [3\text{-}6]$$

The following relationships were found for traditional frying (Eqn 3-7 and Figure 3-10) and for vacuum frying (Eqn 3-8 and Figure 3-11):

$$\ln D_e = -10.2075 - 3257.4 \frac{1}{T},\ R^2 = 0.98 \quad [3\text{-}7]$$

$$\ln D_e = -5.3112 - 5086.15 \frac{1}{T},\ R^2 = 0.98 \quad [3\text{-}8]$$

The pre-exponential factors (A) for potato chips fried under traditional and vacuum frying were 3.7×10^{-5} 1/s and 4.94×10^{-3}(1/s), respectively. The activation energies (E_a) were 27,082 J/mol (for $150^oC \leq T \leq 180^oC$) and 42,286.2 J/mol (for $118^oC \leq T \leq 140^oC$), respectively. A higher E_a means that D_e responds faster to changes in temperature. Hence, changing the temperature will have a greater influence on D_e during vacuum frying than during traditional frying. This is expected, since the boiling point of water during vacuum frying is significantly reduced, and water starts evaporating even before the product is immersed in the oil (Garayo & Moreira, 2002).

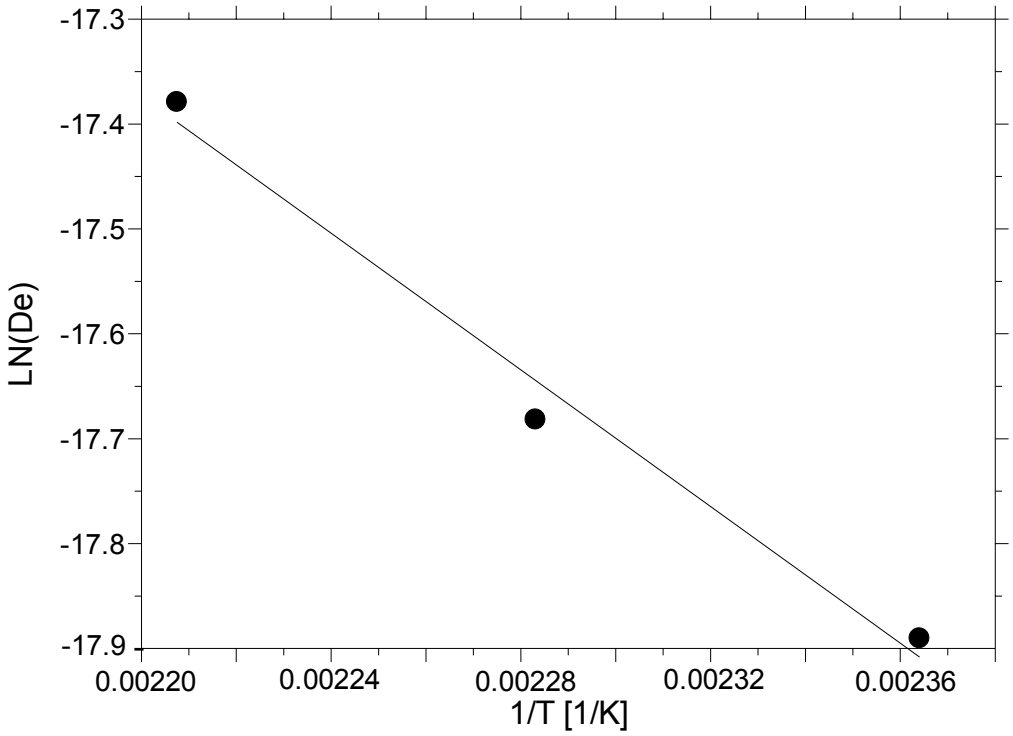


Figure 3-10. Effect of frying temperature on the diffusion coefficient (D_e) of potato chips fried under traditional frying.

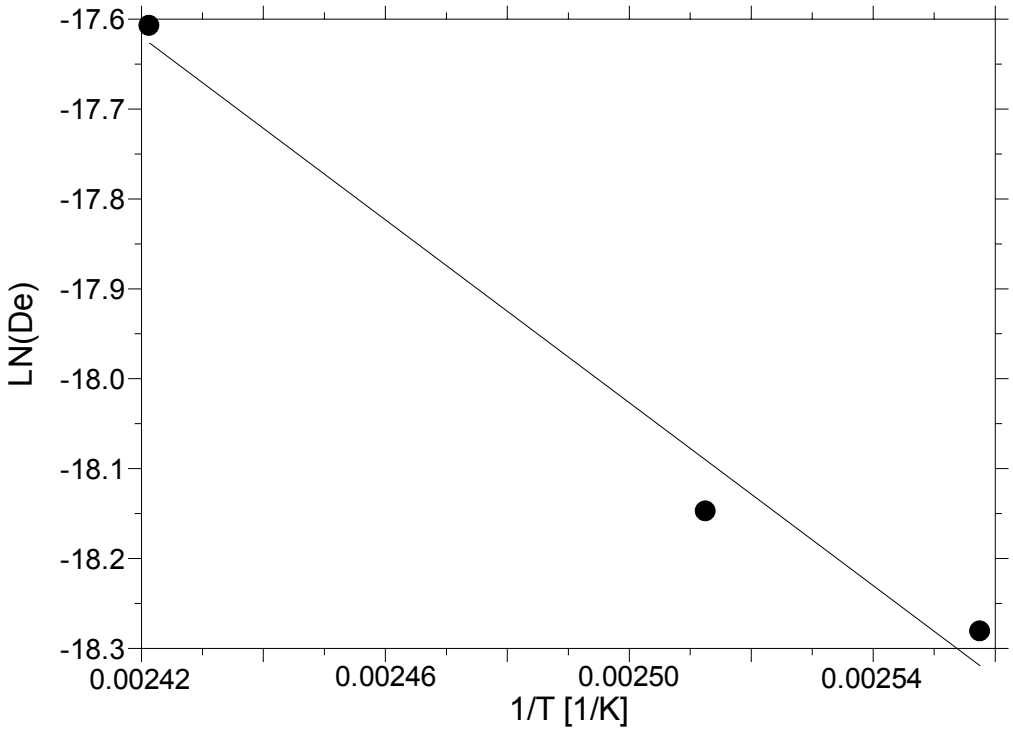


Figure 3-11. Effect of frying temperature on the diffusion coefficient (D_e) of potato chips fried under vacuum (10 Torr) frying.

3.2.2.2. Effect of oil temperature and frying method on oil content of potato chips

The final oil content of potato chips was not significantly different ($P<0.05$) for the samples fried under traditional or vacuum conditions at different oil temperatures (Figures 3-12 and 3-13).

For chips fried under traditional frying (Figure 3-12), the final oil content was about 59% d.b. (37% w.b.) for all the frying temperatures considered in this study. The oil absorption rate was not significantly affected ($P<0.05$) by the frying oil temperature.

During vacuum frying (Figure 3-13) at higher temperatures, the chip's oil content reached its maximum value early in the frying process, then decreased as frying time increased, i.e., the oil absorption rate increased as temperature increased. For instance, after 150 s at 140^{o}C the oil content was about 53% d.b. (35% w.b.); beyond 360 s, the content went down and remained fairly constant (39.93% - 40.40% d.b., 28.54%-28.77% w.b.) to the end of frying. The highest oil content during vacuum frying at 140^{o}C coincides with the period at which water evaporated from the potato slices at the fastest rate at this temperature (Figure 3-9). So, the higher the oil temperature, the higher the oil uptake by the potato chips during the first 150 s of frying, as also observed by Shyu & Hwang (2001) and Garayo & Moreira (2002). As the frying temperature decreased, the peak shifted to the right, in accordance to the period with the fastest drying rate at the given temperature (Figure 3-9). For example, at 125^{o}C, the oil content increased very fast during the first 360 s of frying (41.37% d.b., 29.26% w.b.), reached the maximum oil content at about 480 s (44.73% d.b., 31.91% w.b.), and decreased to 37.88% d.b (27.47% w.b.) after 600 s. This indicates that the final oil content of the potato chips at

constant pressure is not a function of temperature, but rather a function of time and remaining moisture, which increases with decreasing temperature (Garayo and Moreira, 2002).

It can be seen in Figure 3-13 that the behavior of oil content for chips fried under vacuum at 140^{o}C is different from the other temperatures (118^{o}C and 125^{o}C). This difference is produced during the pressurization period, which can decrease or increase oil absorption depending on the amount of free water and surface oil in the product. When the chips fried at 140^{o}C are removed from the oil at the beginning of frying (less than 150 s) the water vapor in the pores condenses, and the pressure difference ($P_{surroundings}$-P_{pore}) drives the oil into the product. The amount of oil absorbed during pressurization will increase until a critical level. At this point, oil absorption during the pressurization process decreases, since $P_{surroundings}$-P_{pore} is negligible. So, when frying under vacuum (10 Torr) at 140^{o}C, the highest oil content is reached when free water is still available to the product, then it decreases until the equilibrium moisture content is reached. At temperatures (118^{o}C and 125^{o}C), the higher free water content makes it difficult for the oil to enter the pore spaces, so the peak observed at 140^{o} C was not seen at lower temperatures at 150 s of frying. Thus, the higher the temperature (i.e. 140^{o}C), the higher the oil content of the chips during the initial frying period, when free water is still available in the product (Garayo & Moreira, 2002)

Figure 3-14 shows the influence of pressure on the final oil content of potato chips at 140^{o}C (vacuum) and 165^{o}C (traditional). Vacuum frying at different temperatures resulted in very similar final oil content. However, at 140^{o}C, the frying

time was reduced to about 420 s, and chips with nice light color and good texture were obtained. The final oil content for the chips fried under vacuum at 140^{o}C (40.40% d.b., 28.77% w.b.) was significantly lower ($P<0.05$) than the final oil content of potato chips fried under atmospheric conditions at 165^{o}C (58.84% d.b., 37.04% w.b.).

Mass transfer in atmospheric frying can be described by two periods: the frying period and the cooling period. During the frying period, the temperature of the product is raised from the ambient temperature to the boiling point of water (100^{o}C). The capillary pressure is negligible during this period, so almost no oil is absorbed (Moreira et al., 1999). During the cooling period, the surface oil is absorbed through the pores of the chips. This occurs due to the difference in pressure caused by an increase in the capillary pressure (Moreira & Barrufet, 1995).

During vacuum frying, the mass transfer mechanisms can be divided in three periods: frying, pressurization, and cooling (Garayo & Moreira, 2002). During frying, the boiling point temperature of the water is lowered; therefore, water evaporates faster under vacuum than under traditional conditions. Capillary pressure during this period is negligible; therefore, no oil absorption occurs. The next period is pressurization, when the pressure in the vessel is brought to atmospheric conditions at a constant temperature (under closed lid). During this stage, gas diffuses much faster to the pores, obstructing the passage of oil to the product. During the last period (cooling), oil on the surface of

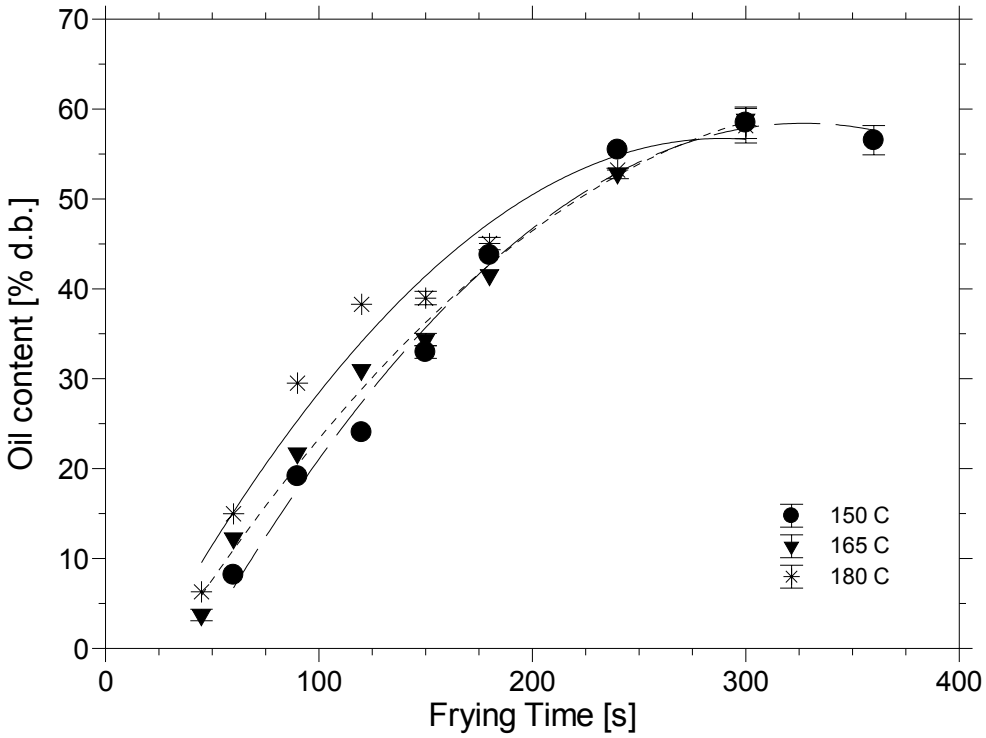


Figure 3-12. Oil absorption rate for potato chips fried under traditional frying at different frying oil temperatures [150^{o}C, OC(t)=-19.05+0.4733*t-0.000723*t^2, R^2=0.99; 165^{o}C, OC(t)=-10.81+0.3964*t-0.0005505*t^2, R^2=0.99; 180^{o}C, OC(t)=-9.448+0.4582*t-0.0007931*t^2, R^2=0.97].

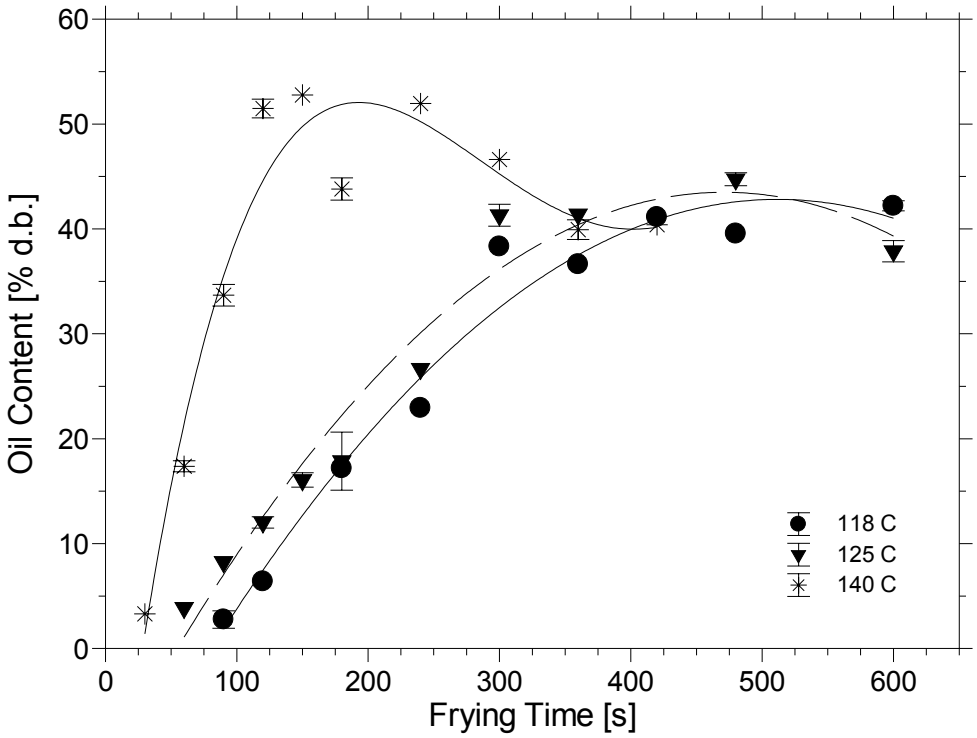


Figure 3-13. Oil absorption rate for potato chips fried under vacuum frying (10 Torr) at different frying oil temperatures [118^{o}C, OC(t)=-19.05+0.4733*t-0.000723*t^2, R^2=0.99; 125^{o}C, OC(t)=-12.19+0.2365*t-0.0002511*t^2, R^2=0.97; 140^{o}C, OC(t)=-25.96+1.046*t-0.004734*t^2+8.188x10^{-6}*t^3-4.656x10^{-9}*t^4, R^2=0.93].

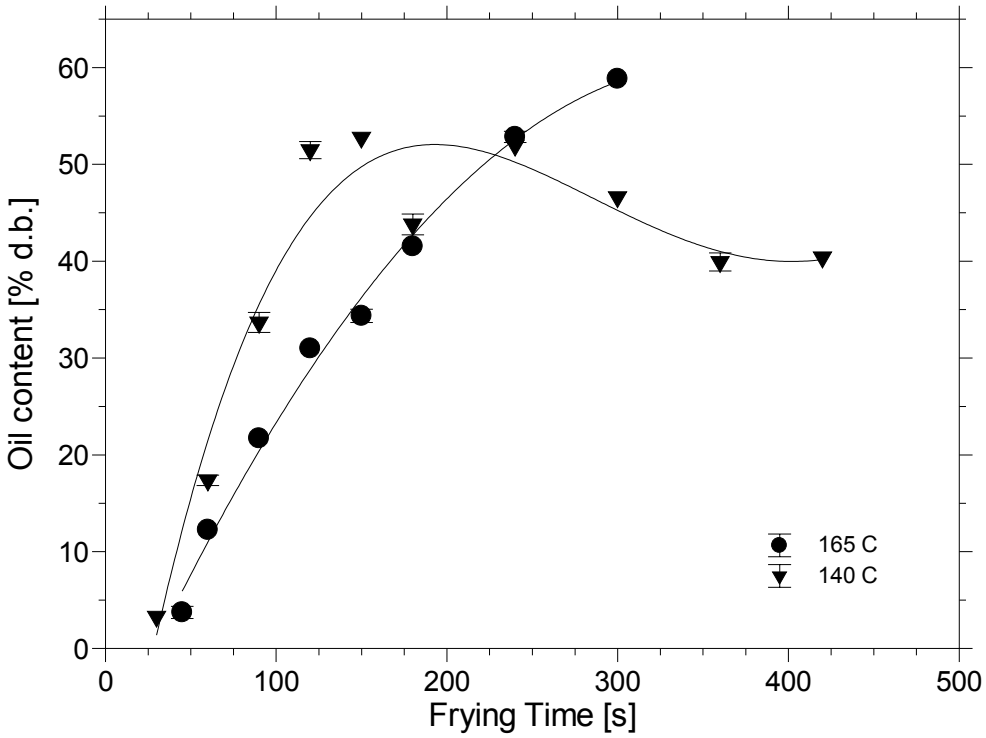


Figure 3-14. Comparison of oil absorption rates for potato chips fried under traditional (165^{o}C) and vacuum (10 Torr, 140^{o}C) frying [165^{o}C, OC(t)=-10.81+0.3964*t-0.0005505*t^2, R^2=0.99; 140^{o}C, 10 Torr, OC(t)=-25.96+1.046*t-0.004734*t^2+8.188x10^{-6}*t^3-4.656x10^{-9}*t^4, R^2=0.93].

the chips penetrates the pore spaces. However, since less oil remains on the surface of potato chips during vacuum frying, less oil is absorbed during cooling, which explains why the final oil content during traditional frying is higher than during vacuum frying (Garayo & Moreira, 2002).

The rate of oil content change was modeled using a fractional conversion kinetic model (Chen & Ramaswamy, 2002):

$$OR = \frac{OC(t) - OC_e}{OC_o - OC_e} = A\exp(-kt) \qquad [3\text{-}9]$$

where OR=oil ratio, OC_e=equilibrium oil content, OC_o=initial oil content, A and k are the regression coefficients, and t =frying time.

During vacuum frying at 140^{o}C, two regions were defined for the kinetic model: from 0 to 240 s, and from 240 to 420 s, since these two regions showed different behavior. Table 3-9, Table 3-10, and Figures 3-15 and 3-16 show the values of the kinetic parameters.

Table 3-9. Oil absorption rate constant (k) values (Eqn 3-9) for potato chips fried under traditional frying.

Oil Temperature (^oC)	**OC_o[1] (% w.b.)**	**OC_e[2] (% w.b.)**	***A***	***k* (1/s)**	**R^2**
150	7.57	36.11	2.159	0.012	0.97
165	3.59	37.04	1.639	0.012	0.99
180	5.93	36.76	1.851	0.014	0.99

[1]Initial oil content. [2]Equilibrium oil content (final oil content).

Table 3-10. Oil absorption rate constant (k) values (Eqn 3-9) for potato chips fried under vacuum frying.

Oil Temperature (^oC)	**OC_o[1] (% w.b.)**	**OC_e[2] (% w.b.)**	***A***	***k* (1/s)**	**R^2**
118	2.68	29.68	2.157	0.008	0.97
125	3.74	27.47	1.803	0.009	0.93
140 (0-240 s)	3.20	34.19	2.026	0.022	0.95
140 (240-420 s)	34.19	28.78	51.03	0.016	0.92

[1]Initial oil content. [2]Equilibrium oil content (final oil content).

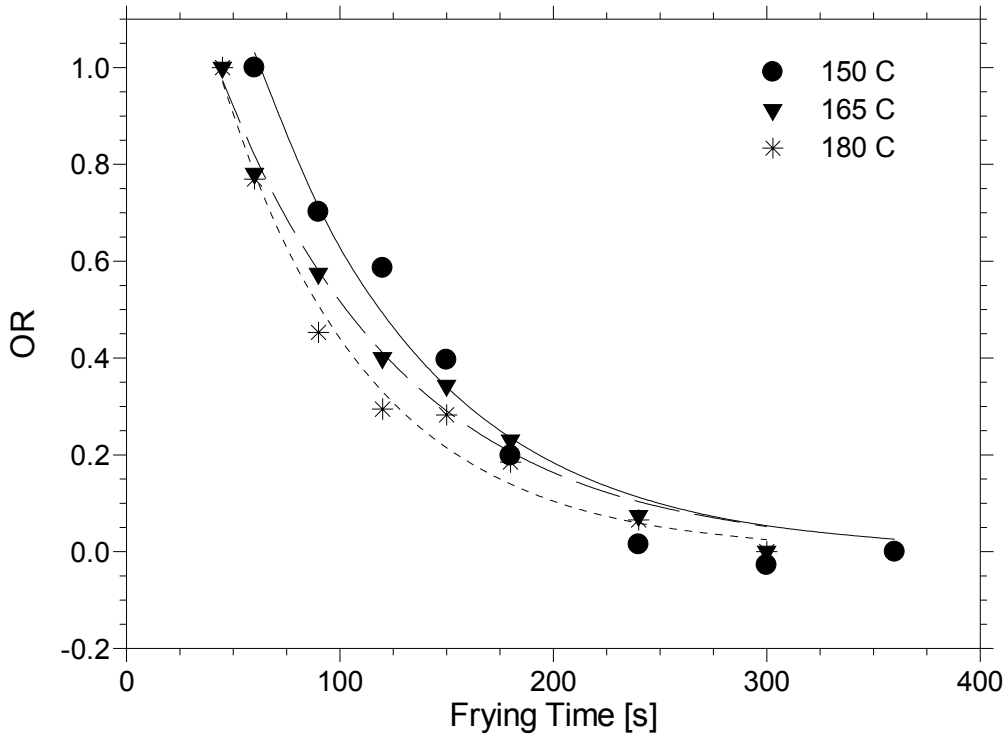


Figure 3-15. Oil absorption kinetics in potato chips fried under traditional frying at different frying oil temperatures [$OR=(OC(t)-OC_e)/(OC_o-OC_e)$], [150$^oC$, $OR(t)=2.159*\exp(-0.01234*t)$, $R^2=0.97$; 165^oC, $OR(t)=1.634*\exp(-0.0115*t)$, $R^2=0.99$; 180^oC, $OR(t)=1.851*\exp(-0.01435*t)$, $R^2=0.99$].

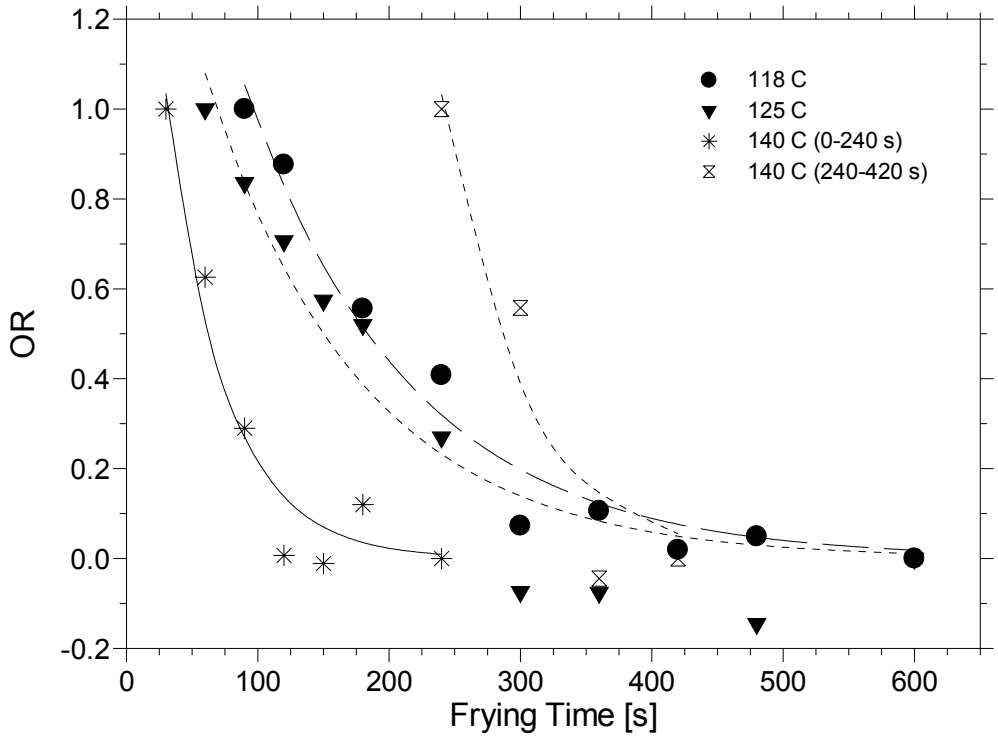


Figure 3-16. Oil absorption kinetics in potato chips fried under vacuum (10 Torr) frying at different frying oil temperatures [$OR=(OC(t)-OC_e)/(OC_o-OC_e)$], [118°C, $OR(t)=2.157*\exp(-0.00796*t)$, $R^2=0.97$; 125°C, $OR(t)=1.803*\exp(-0.00854*t)$, $R^2=0.93$; 140°C (0-240 s), $OR(t)=2.026*\exp(-0.02240*t)$, $R^2=0.95$; 140°C (240-420 s), $OR(t)=51.03*\exp(-0.01625*t)$, $R^2=0.92$].

3.2.2.3. Effect of oil temperature and frying method on texture of potato chips

Figure 3-17 shows that there was no specific trend in texture versus frying time when frying under either method. No significant textural differences ($P<0.05$) were found among the chips produced by the two frying methods (Table 3-7).

For both methods, the potato chips became more brittle rather than rubbery earlier in the frying process as temperature increased, due to the lower moisture content.

The surface of the potato chips fried under both methods was quite different, as also observed by Garayo and Moreira (2002). The surface of the potato chips produced by vacuum frying contained several small bubbles, while the potato chips fried under atmospheric conditions had less, but larger bubbles. This difference occurred because, under vacuum, water vapor in the chip's pores expands with little resistance during the evacuation process because the crust has not yet been formed (Garayo & Moreira, 2002). During traditional frying, the gelatinized starch (and then the crust) will form a barrier for the saturated vapor to escape, forming large but few bubbles during frying (Kawas & Moreira, 2001).

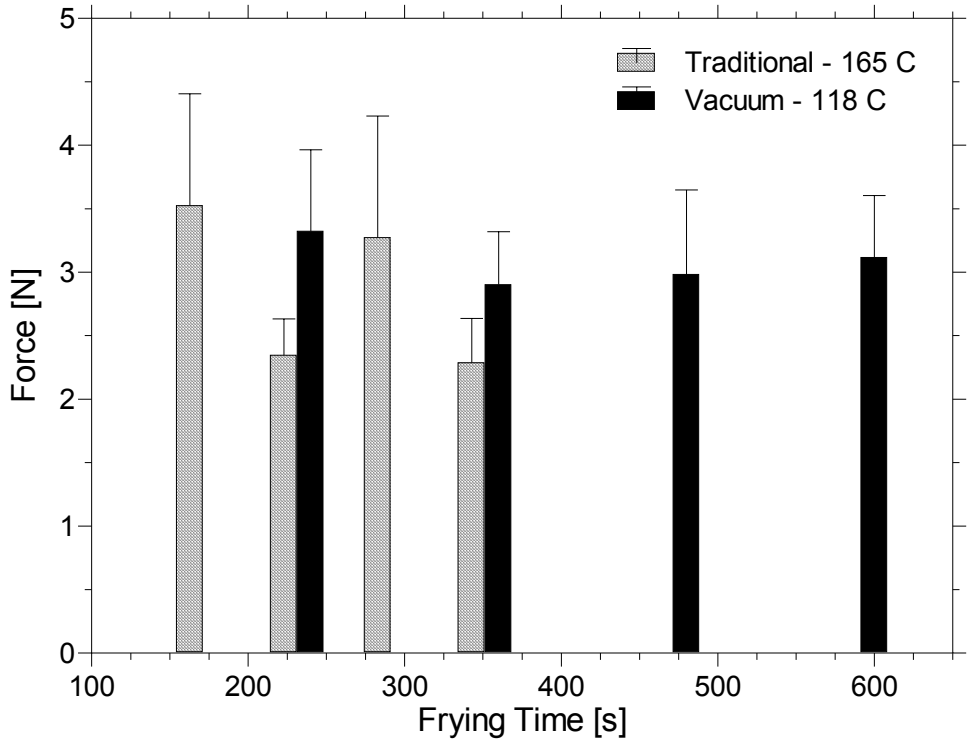


Figure 3-17. Effect of frying time on the texture (hardness) of potato chips fried under traditional and vacuum (10 Torr) frying.

3.2.2.4. Effect of frying time and temperature on color of potato chips

The most obvious effect on the *L*-value was found during traditional frying (Figure 3-18). The *L*-value denotes lightness, and ranges from 0 to 100 (0=black, 100=white). This value decreased as time increased, an indication that the potato chips were becoming darker. This same behavior was observed when frying potato chips at higher temperatures. For instance, at 120 min of frying, the *L*-values were 50.632, 48.802, and 46.748 for 150^{o}C, 165^{o}C, and 180^{o}C, respectively. No trend was found during vacuum frying (Figure 3-19).

The *a*-value (green-red chromaticity) did not follow a clear trend for either frying method (Figure 3-20 and Figure 3-21). However, towards the end of vacuum frying (after 360 s) the *a*-value for the chips fried at 140^{o}C was significantly higher ($P<0.05$) than those fried at 118^{o}C and 125^{o}C (Figure 3-21).

The most obvious differences in color were found in the *b*-values (blue-yellow chromaticity). As frying time and temperature increased, the *b*-value also increased for both frying methods (Figure 3-22 and Figure 3-23). When comparing traditional frying at 165^{o}C with vacuum frying at 118^{o}C, it was found that the *b*-values for the traditional frying method were significantly higher ($P<0.05$) than those for vacuum frying at all times (Figure 3-24).

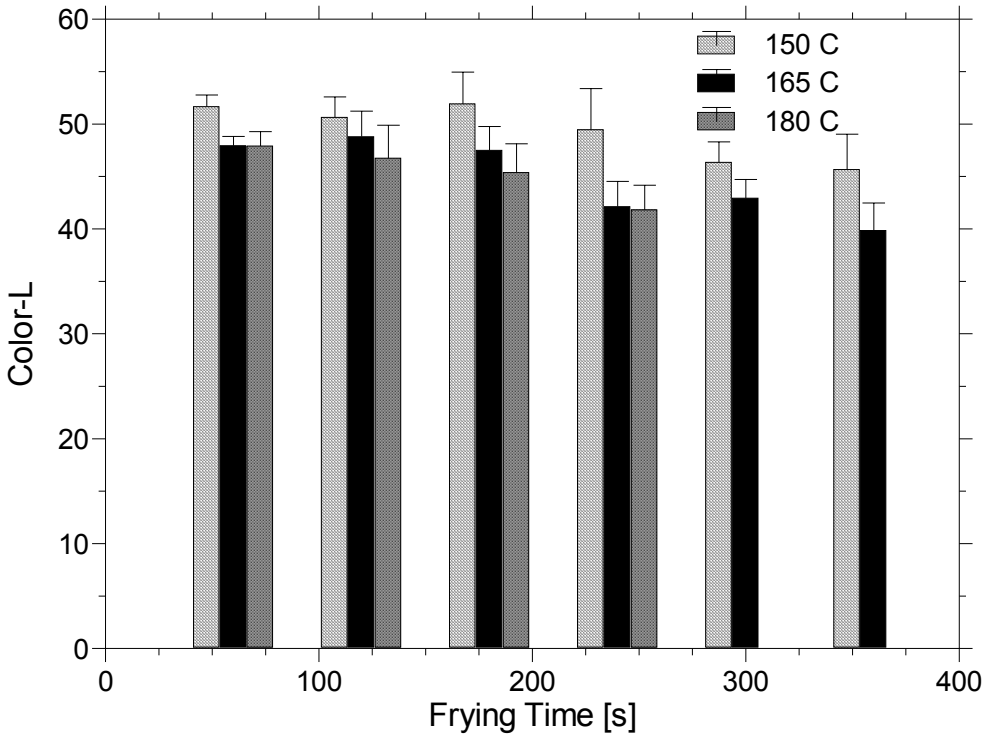


Figure 3-18. Effect of frying time and oil temperature on lightness (*L*-value) of potato chips fried under traditional frying at different frying oil temperatures.

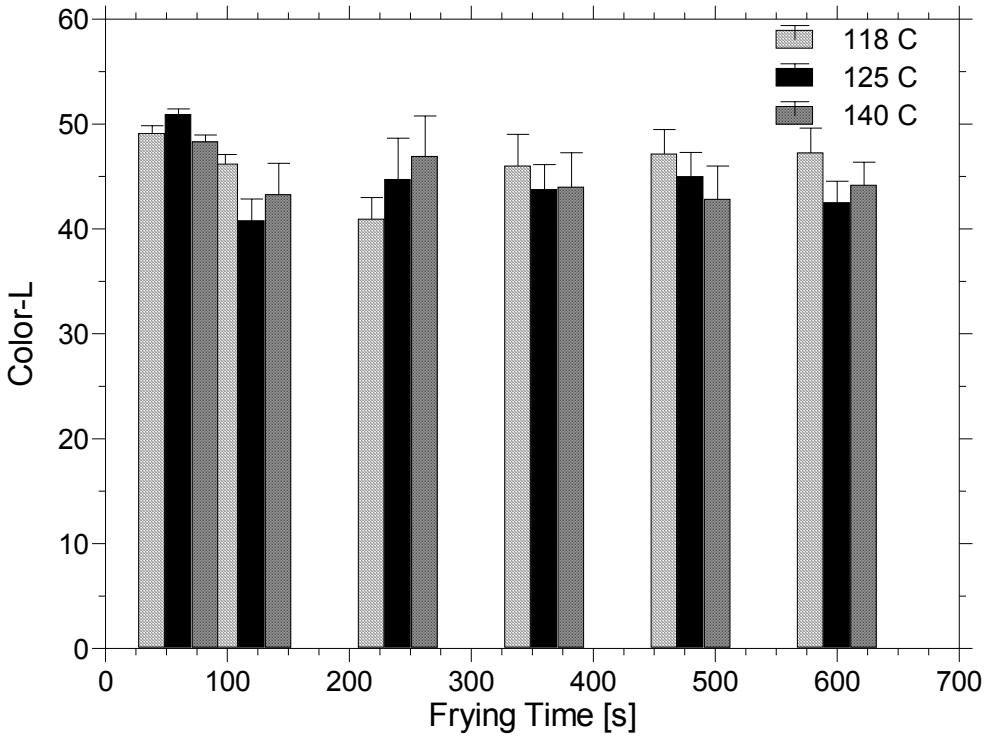


Figure 3-19. Effect of frying time and oil temperature on lightness (*L*-value) of potato chips fried under vacuum (10 Torr) frying at different frying oil temperatures.

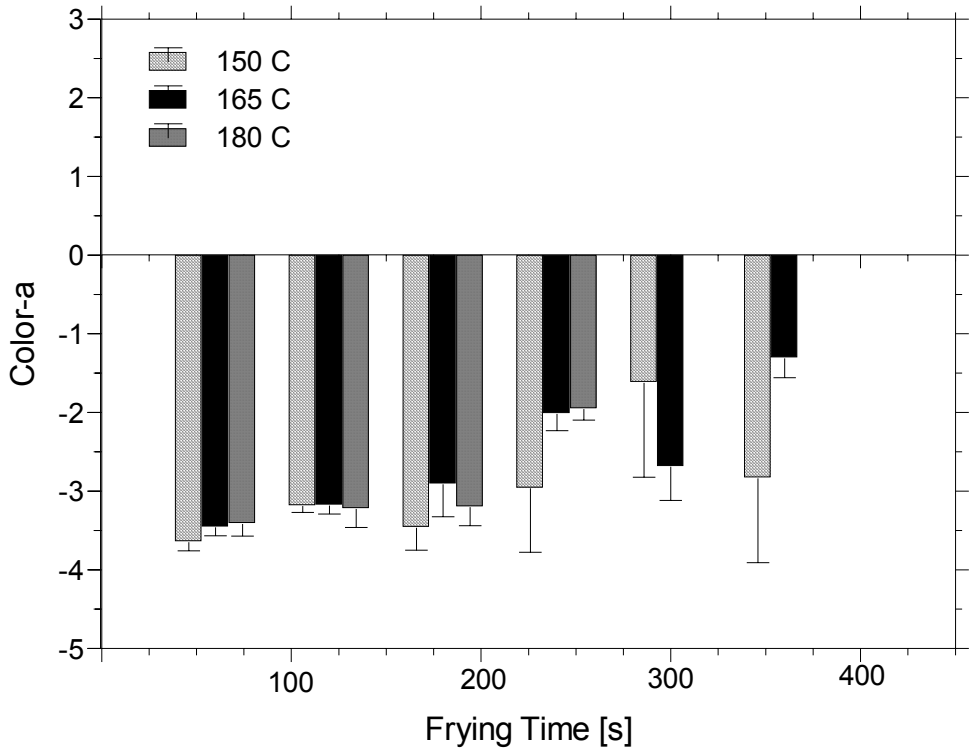


Figure 3-20. Effect of frying time and oil temperature on the green-red chromaticity (*a*-value) of potato chips fried under traditional frying at different frying oil temperatures.

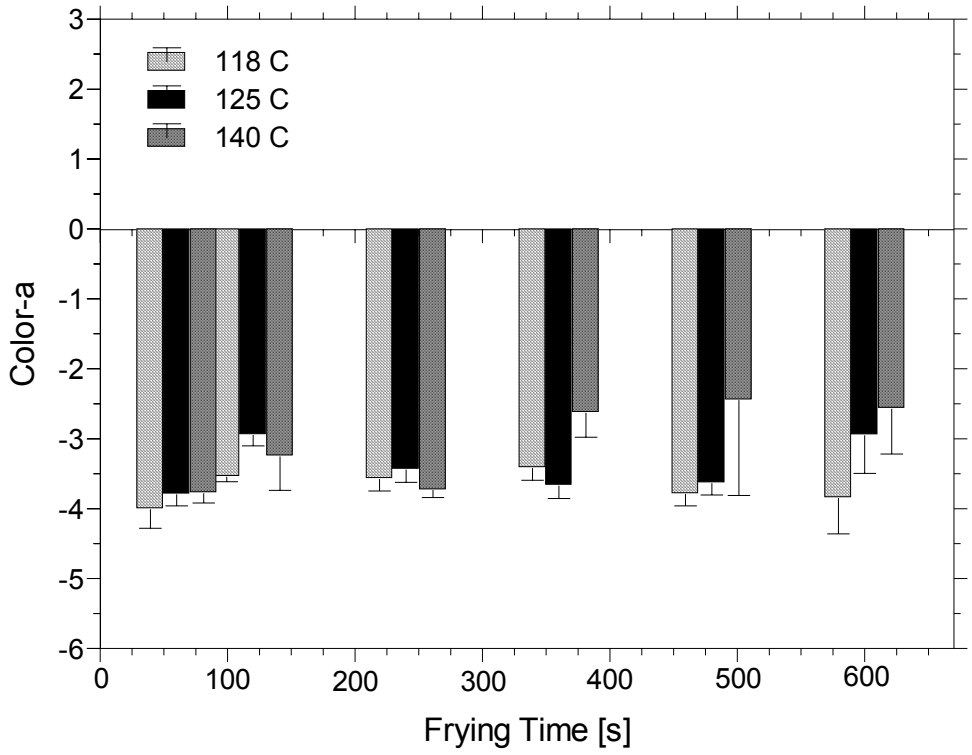


Figure 3-21. Effect of frying time and oil temperature on the green-red chromaticity (*a*-value) of potato chips fried under vacuum (10 Torr) frying at different frying oil temperatures.

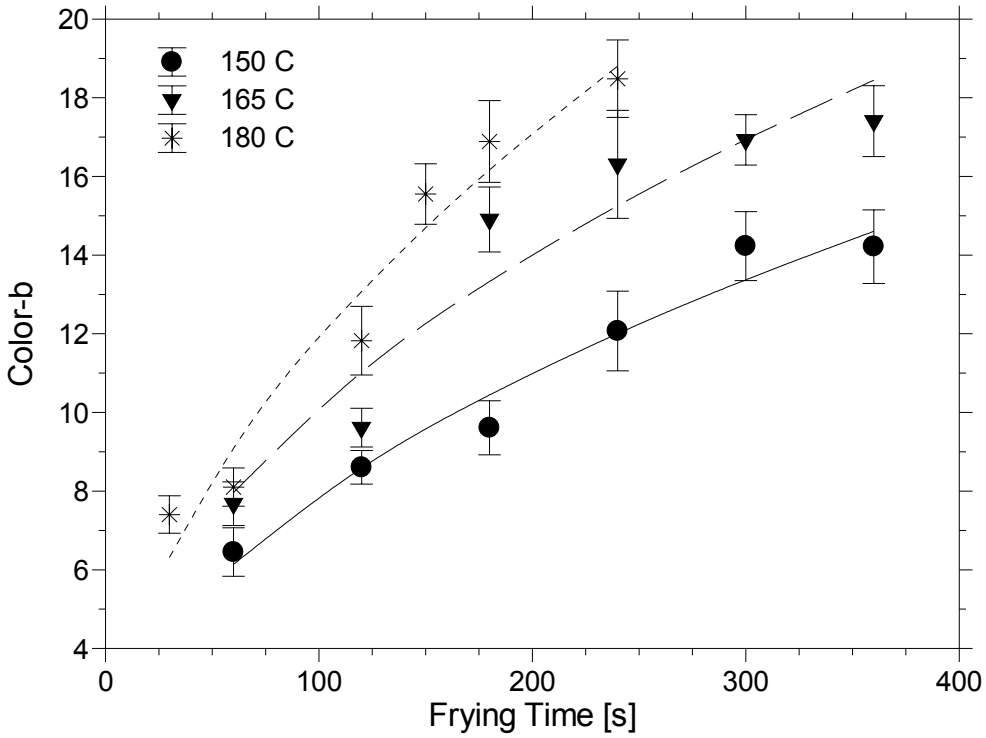


Figure 3-22. Effect of frying time and oil temperature on the blue-yellow chromaticity (*b*-value) of potato chips fried under traditional frying at different frying oil temperatures [150^{o}C, $b(t)=0.8477*x^{0.4832}$, $R^2=0.97$; 165^{o}C, $b(t)=1.168*x^{0.4688}$, $R^2=0.92$; 180^{o}C, $b(t)=1.0600*x^{0.55248}$, $R^2=0.95$] .

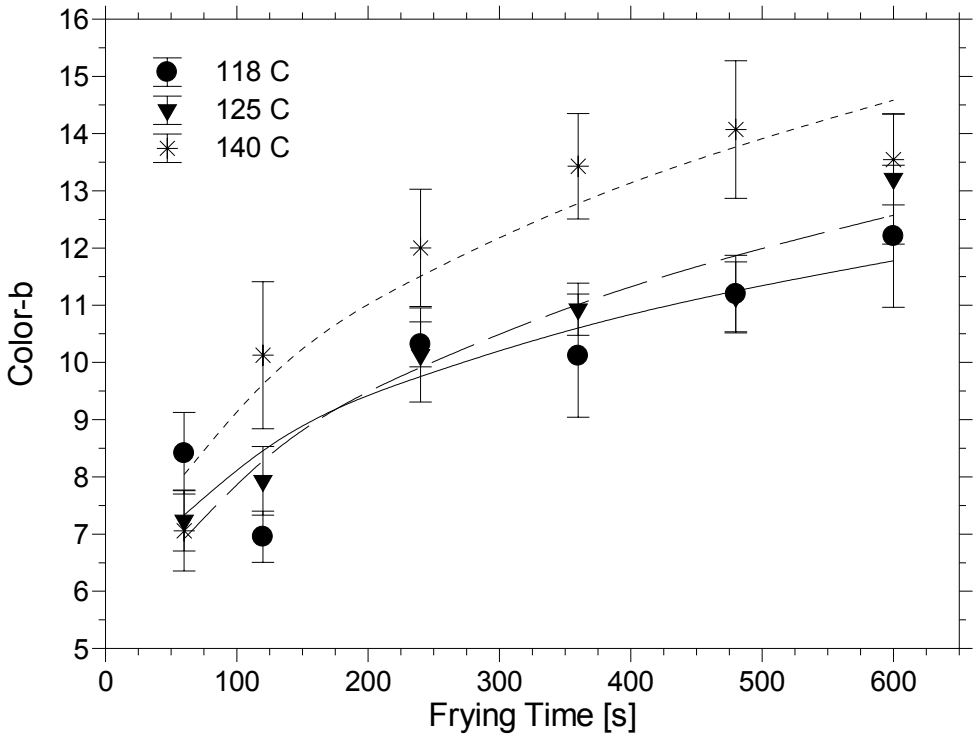


Figure 3-23. Effect of frying time and oil temperature on the blue-yellow chromaticity (*b*-value) of potato chips fried under vacuum (10 Torr) frying at different frying oil temperatures [118^{o}C, $b(t)=3.159*x^{0.2057}$, $R^2=0.77$; 125^{o}C, $b(t)=2.391*x^{0.2595}$, $R^2=0.95$; 140^{o}C, $b(t)=2.7900*x^{0.2585}$, $R^2=0.92$].

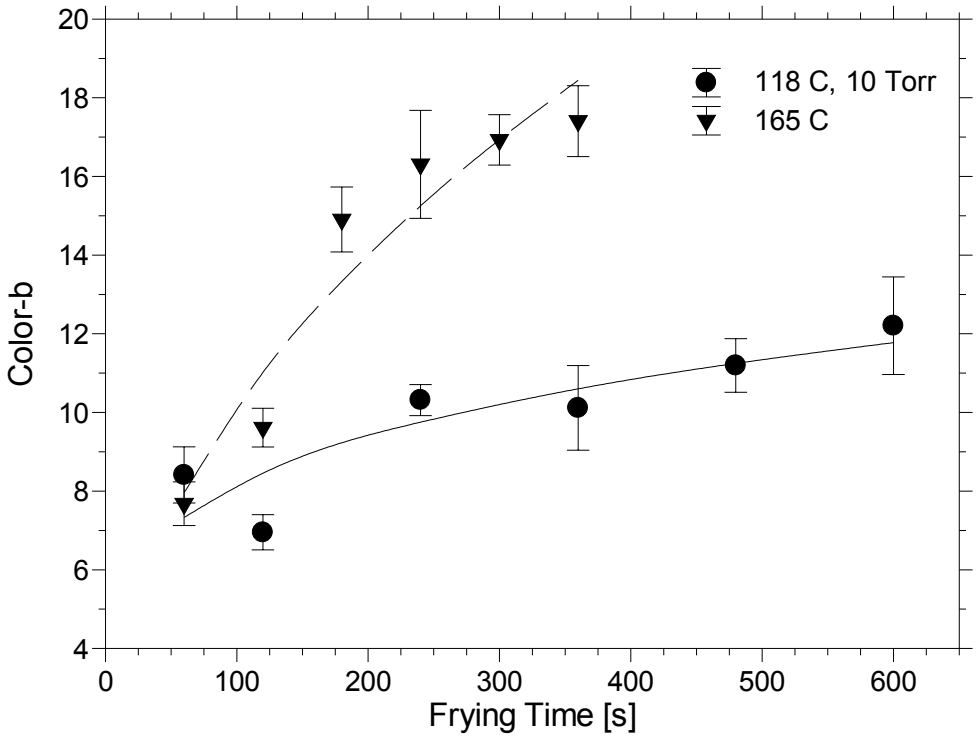


Figure 3-24. Comparison of the blue-yellow chromaticity (*b*-value) changes in potato chips fried under vacuum (118^{o}C, 10 Torr) and traditional (165^{o}C) conditions [118^{o}C, 10 Torr, $b(t)=3.159*x^{0.2057}$, $R^2=0.77$; 165^{o}C, $b(t)=1.168*x^{0.4688}$, $R^2=0.92$].

3.2.2.5. Effect of frying method and oil temperature on acrylamide formation

Figure 3-25 and Figure 3-26 show that acrylamide formation increases with time for all temperatures, for both methods. When frying potato chips to the same final moisture content under traditional frying (1.5%±0.3% w.b.), acrylamide content decreased by 23% when temperatures changed from 180°C to 165°C, 51% from 165°C to 150°C, and 62% from 180°C to 150°C (Figure 3-25).

Figure 3-27 shows the effect of temperature on the formation of acrylamide in potato chips fried under traditional frying. Acrylamide increases with temperature for all frying times. In fact, when reducing the oil temperature from 190°C to 150°C at 240 s of frying, the acrylamide content reduction in potato chips was about 94%. This result was also observed by Pedreschi et al. (2004), who found that the reduction of the frying oil temperature from 190°C to 170°C and to 150°C decreased acrylamide formation in potato chips by 68% and 88%, respectively. Decreasing the temperature from 190°C to 150°C caused a decrease in acrylamide content of about 95% (Pedreschi et al., 2004). Haase et al. (2003) reported that by lowering the frying temperature from 185°C to 165°C, and from 190°C to 150°C, it was possible to reduce the acrylamide content in potato chips by half, and by two thirds, respectively. Kita et al. (2004) also reported a decrease of approximately 75% in acrylamide content of potato chips when reducing the frying oil temperature from 185°C to 160°C. Gertz and Klostermann (2002) found a decrease of 85% in acrylamide content in French fries when lowering the oil temperature from 185°C to 160°C.

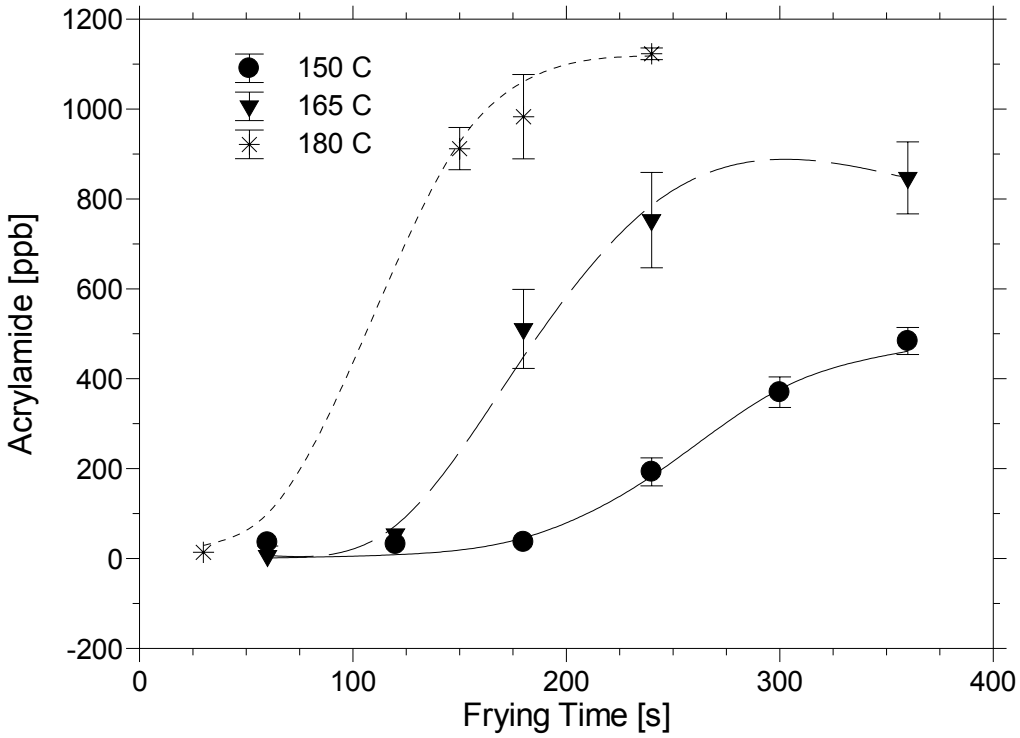


Figure 3-25. Effect of frying time and oil temperature on acrylamide content in potato chips (Atlantic cultivar) fried under traditional frying [150^{o}C, AA(t)=484/(1+exp(-0.02921*(t-257))), R^2=0.99; 165^{o}C, AA(t)=847/(1+exp(-0.04056*(t-424))), R^2=0.99; 180^{o}C, AA(t)=1123/(1+exp(-0.04308*(t-562))), R^2=0.99].

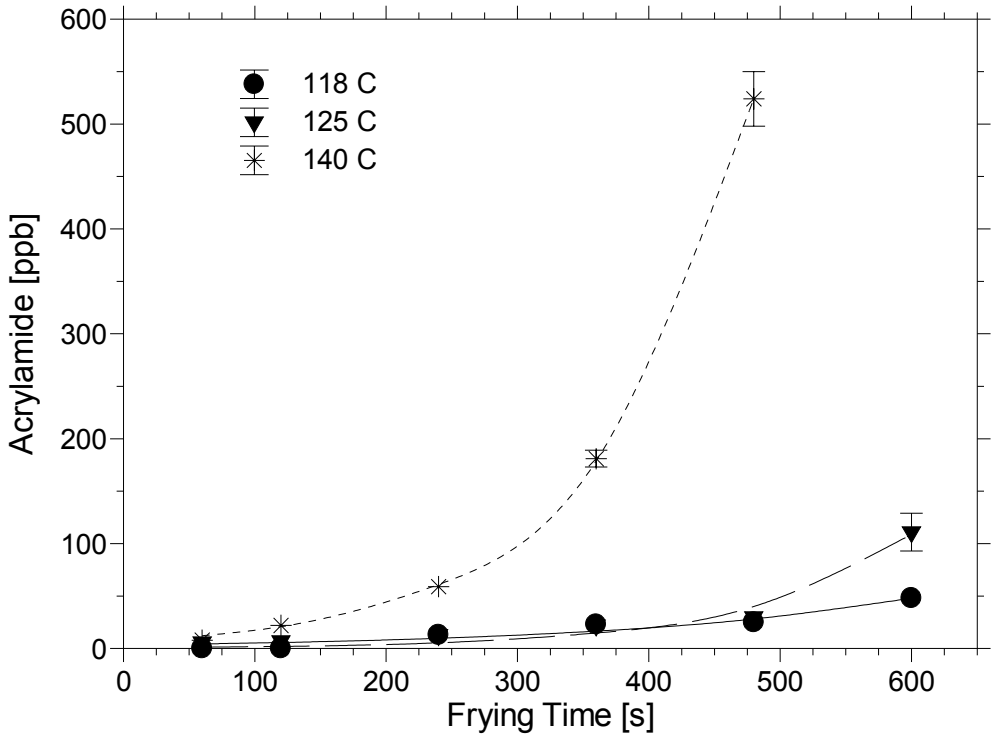


Figure 3-26. Effect of frying time on acrylamide content in potato chips fried under vacuum (10 Torr) frying at different frying oil temperatures [118^{o}C, AA(t)=5.860*exp(0.00339*t), R^2=0.94; 125^{o}C, AA(t)=3.688*exp(0.00514*t), R^2=0.96; 140^{o}C, 5.671*exp(0.00956*t), R^2=0.99].

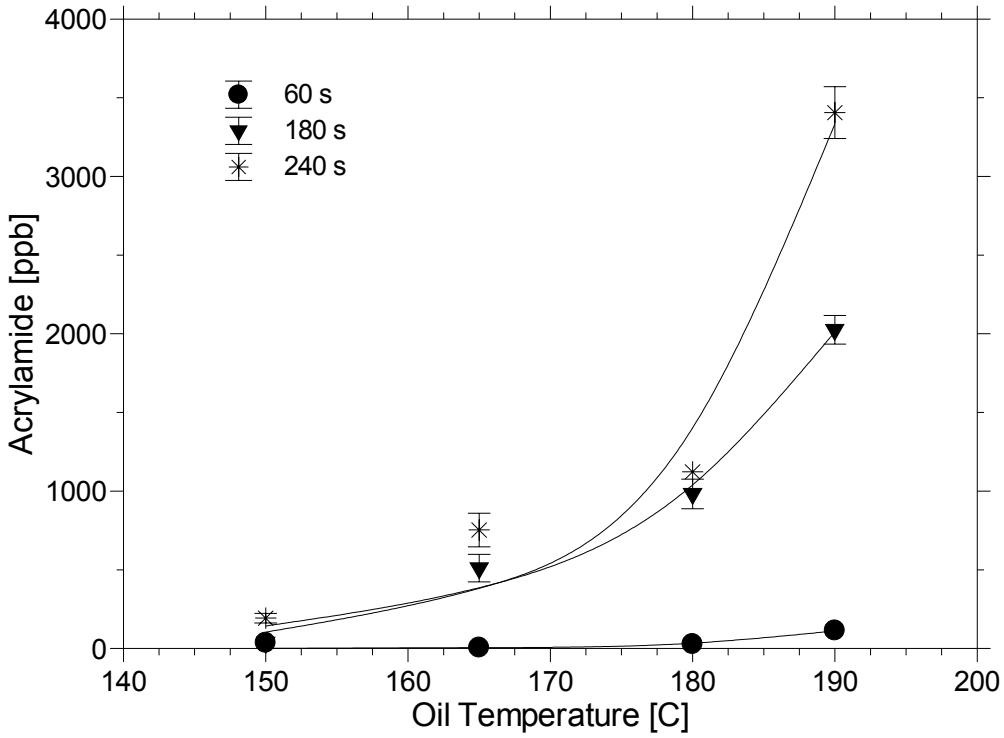


Figure 3-27. Effect of oil temperature on acrylamide content in potato chips fried under traditional frying after different frying times [60 s, $AA(T)=1.643x10^{-8} * \exp(0.11920*T)$, $R^2=0.87$; 180 s, $AA(T)=0.007192*\exp(0.06601*T)$, $R^2=0.99$; 240 s, $AA(T)=0.000237*\exp(0.08663*T)$, $R^2=0.97$].

According to Taubert et al. (2004), the effect of temperature on the formation of acrylamide depends on the surface-to-volume-ratio (SVR) of the food product. In this study, potato products with low SVR showed an increase in acrylamide formation as the temperature increased. They also found that acrylamide formation in potato cylinders (30 mm in diameter, 15 mm in height) continuously increased with increasing time and temperature. At 180^{o}C and at 200^{o}C, acrylamide concentration in the slices at 10 min of frying was 2000 and 2500 ppb, respectively. Conversely, high SVR potato slices showed maximum acrylamide levels at 160-180^{o}C. For example, in cylindrical potato slices of 30 mm diameter and 3 mm height, the acrylamide content after frying for 7 min at 180^{o}C and 200^{o}C was about 12000 and 8000 ppb, respectively.

A decrease in acrylamide formation with temperature has been reported in model systems (Mottram et al., 2002). Taubert et al. (2004) and Biedermann et al. (2002) suggested that this decrease in acrylamide concentration may be due to acrylamide degradation that occurs with increasing time and after a certain temperature. Acrylamide formation did not decrease with temperature in the present study (Figure 3-27); higher temperatures may be needed to see the decrease in acrylamide formation.

The degradation of acrylamide with time can be seen in Figure 3-25: at the beginning of frying, acrylamide formed rapidly, and then it remained constant; this may be due to the fact that, at the beginning of frying, acrylamide is only forming. After some time (about 300 s, 240 s, and 140 s at 150^{o}C, 165^{o}C and 180^{o}C, respectively), acrylamide is forming but also degrading, producing a counteracting effect. The same curve behavior was observed by Amrein et al. (2004) for gingerbread. Acrylamide

content increased exponentially up to 1000 ppb during the first 20 min of baking at 200^{o}C, then it remained constant.

The increase in acrylamide formation in potato chips fried under vacuum frying was more subtle than in those fried under traditional frying (Figure 3-26). The acrylamide content increased from not detectable (ND) to only 48 ppb at 118^{o}C during the first 600 s of frying. At 125^{o}C, the acrylamide content in the potato chips increased from 6 to 111 ppb. However, when fried at 140^{o}C, the potato chips showed an acrylamide content increase from 8 to 524 ppb during 480 s of frying. These results indicate that acrylamide content increases with increased temperature, as expected. The mechanism of acrylamide accumulation during vacuum frying is different than during traditional frying (See section 3.2.3.2 for an explanation).

Figure 3-28 shows the influence of acrylamide accumulation in potato chips fried under traditional (165^{o}C) and vacuum (118^{o}C, 10 Torr) frying. There was a 94% decrease in acrylamide content when potatoes were fried to the same final moisture content (1.5%$\pm$0.3% w.b.) under vacuum. This decrease in acrylamide content is due to the lower temperature used during vacuum frying, since acrylamide formation increases as temperature increases above 100^{o}C (Taubert et al., 2004; Mottram et al., 2002).

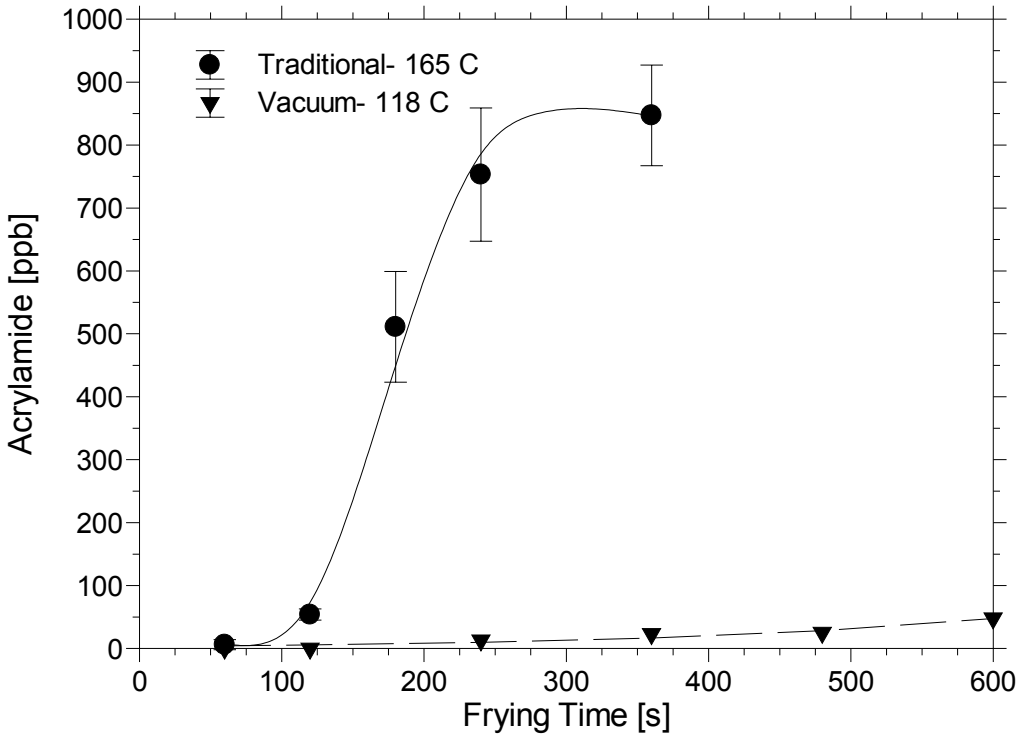


Figure 3-28. Effect of frying method and time on acrylamide content in potato chips.

3.2.3. Kinetic studies of acrylamide formation in potato chips fried under traditional and vacuum frying

3.2.3.1. Kinetics of acrylamide formation in traditionally fried potato chips

Kinetics of acrylamide formation in potato chips fried under traditional frying was modeled by using a special case of the logistic kinetic model (Chen & Ramaswamy, 2002) that accurately describes product qualities that increase exponentially and eventually level-off:

$$C(t) = A_o + \frac{A}{1 + \exp[-k(t - t_o)]} \qquad [3\text{-}10]$$

where C is the acrylamide content (ppb) at time t (s), A_o is the acrylamide content before frying (A_o=0 ppb), A is a constant value related to the equilibrium acrylamide content (ppb), and t_o (s) is the time constant value when the acrylamide content increases to half of A value.

Table 3-11 shows the values of the parameters in Eqn (3-10) for different frying temperatures (traditional frying). Figure 3-25 shows the experimental and fitted values for acrylamide content using this model. It can be seen from Table 3-11 that the values for A (equilibrium acrylamide content) increased with increasing temperature. This was expected since higher temperatures produce higher levels of acrylamide due to increased Maillard reaction. Also, as temperature increased, t_o (the time when the acrylamide content increases to half of its equilibrium value) decreased, i.e., at higher temperatures the acrylamide content will reach half of its maximum value earlier in the frying process.

As temperature increased, the acrylamide formation rate constant *k* increased as expected, since higher temperatures enhance the rate of acrylamide formation.

Table 3-11. Regression coefficients of the time dependent logistic model (Eqn 3-10) for acrylamide formation in potato chips during traditional frying.

Temperature (^{o}C)	***A* (ppb)**	***t*$_o$ (s)**	***k* (1/s)**	**R^2**
150	484	257	0.0292	0.99
165	847	178	0.0406	0.99
180	1123	114	0.0431	0.99

The influence of temperature on the rate constant (*k*) during traditional frying was modeled by using the Arrhenius equation:

$$k(T) = A\exp\frac{-E_a}{RT} \qquad [3\text{-}11]$$

where A is the pre-exponential factor, E_a is the activation energy, T is the absolute temperature (K), and R is the universal gas constant (8.314 J/molK).

Equation (3.11) can be linearized:

$$\ln k = \ln A - \frac{E_a}{RT} \qquad [3\text{-}12]$$

The following relationship was found by plotting ln(*k*) vs 1/T (Figure 3-29):

$$\ln k = 2.701 - 2625.8\left(\frac{1}{T}\right),\ R^2 = 0.89 \qquad [3\text{-}13]$$

From Eqn (3-13), A=14.9 1/s and E_a=21,830.9 J/mol. The dependence of k on temperature in traditionally fried chips is finally given by:

$$k(T) = 14.9(1/s)\exp\frac{-2625.8\ K}{T} \quad [3\text{-}14]$$

Finally, acrylamide accumulation during traditional frying can be described as:

$$C(t) = \frac{A}{1+\exp\left[-14.9\exp\frac{-2625.8}{T}(t-t_o)\right]} \quad [3\text{-}15]$$

As explained in the previous section (3.2.2.5), the behavior of these curves (Figure 3-25) may be due to the fact that, during traditional frying, acrylamide content increased exponentially for all temperatures at the beginning of frying. However, as time progresses (about 300 s, 240 s, and 140 s at 150^{o}C, 165^{o}C and 180^{o}C, respectively), acrylamide may not only be forming but also degrading, causing acrylamide content to remain constant. Another possible explanation of this behavior is that available amounts of reactants (asparagine and reducing sugars) in the potato chips are exhausted at a certain point, and acrylamide content remains constant (Taubert et al., 2004).

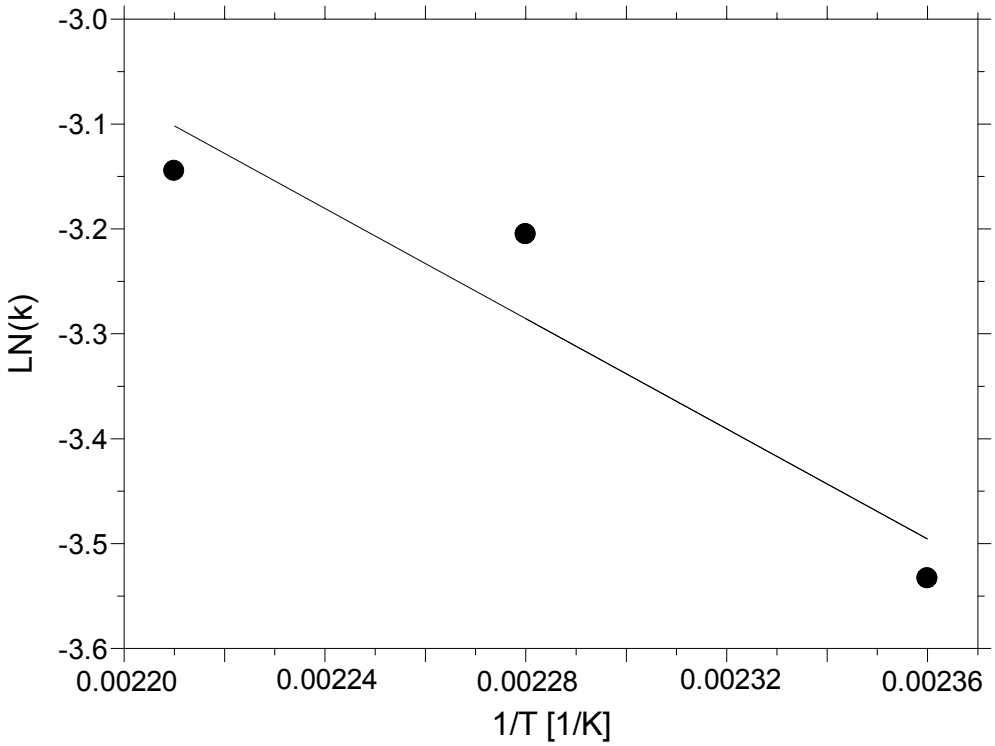


Figure 3-29. Effect of temperature on the rate constant *k* for acrylamide formation in potato chips fried under traditional frying (Eqn 3-13).

3.2.3.2. Kinetics of acrylamide formation in vacuum fried potato chips

As discussed by Chen and Ramaswamy (2002), the rate of change of a quality factor "C" may be represented as in Equation (3-16):

$$\frac{dC}{dt} = -k(C)^n \qquad [3\text{-}16]$$

where k is the rate constant (1/s), C is the property being monitored, and n is the order of the reaction. For the majority of foods, the time dependence relationships can be described by zero- or first-order reactions (Lenz and Lund, 1980). If Eqn (3-16) is integrated, the following equations result for zero (Eqn 3-17), first (Eqn 3-18) and fractional conversion (Eqn 3-19) kinetic models. Eqn (3-20) is the general solution for Eqn (3-16).

$$C(t) = C_o + kt \qquad [3\text{-}17]$$

$$C(t) = C_o \exp(-kt) \qquad [3\text{-}18]$$

$$\frac{C(t) - C_f}{C_o - C_f} = \exp(-kt) \qquad [3\text{-}19]$$

$$C^{1-n} - C_o^{\,1-n} = (n-1)k''t, \quad n \neq 1 \qquad [3\text{-}20]$$

where C is the property being studied at time t, n is the order of the reaction, and C_o and C_f represent the initial and equilibrium values, respectively.

The influence of time on acrylamide formation in vacuum fried chips (Figure 3-26) was best described by first order kinetics (Eqn 3-18), since it increased exponentially with time at all temperatures. Linearizing Eqn (3-18), the following relationship was obtained:

$$\ln C = \ln C_o - kt \quad [3\text{-}21]$$

By graphing ln *C* vs. frying time (Figure 3-30) the parameters in Eqn (3-21) were found (Table 3-12).

Table 3-12. Regression coefficients of the first-order kinetic model for acrylamide formation in potato chips fried under vacuum (10 Torr).

Temperature (^{o}C)	***C*$_o$ [ppb]**	***k* [1/s]**	**R^2**
118	5.860	0.00339	0.937
125	3.688	0.00514	0.958
140	5.671	0.00956	0.990

The reaction rate (*k*) temperature dependence was determined using the Arrhenius equation (Eqn 3-11). The following relationship was found by plotting ln(*k*) vs 1/T (Figure 3-31):

$$\ln k = 13.1333 - 7344.14\left(\frac{1}{T}\right),\ R^2 = 0.99 \quad [3\text{-}22]$$

From Eqn (3-22), *A*=505498 1/s and E_a=61059.2 J/mol. The dependence of *k* on temperature in traditionally fried chips is then given by:

$$k(T) = 505498(1/s)\exp\frac{-7344.14\ K}{T} \quad [3\text{-}23]$$

Finally, acrylamide formation during vacuum (10 Torr) frying can be described as:

$$C(t) = C_o \exp\left(505498\exp\frac{-7344.14}{T} * t\right) \quad [3\text{-}24]$$

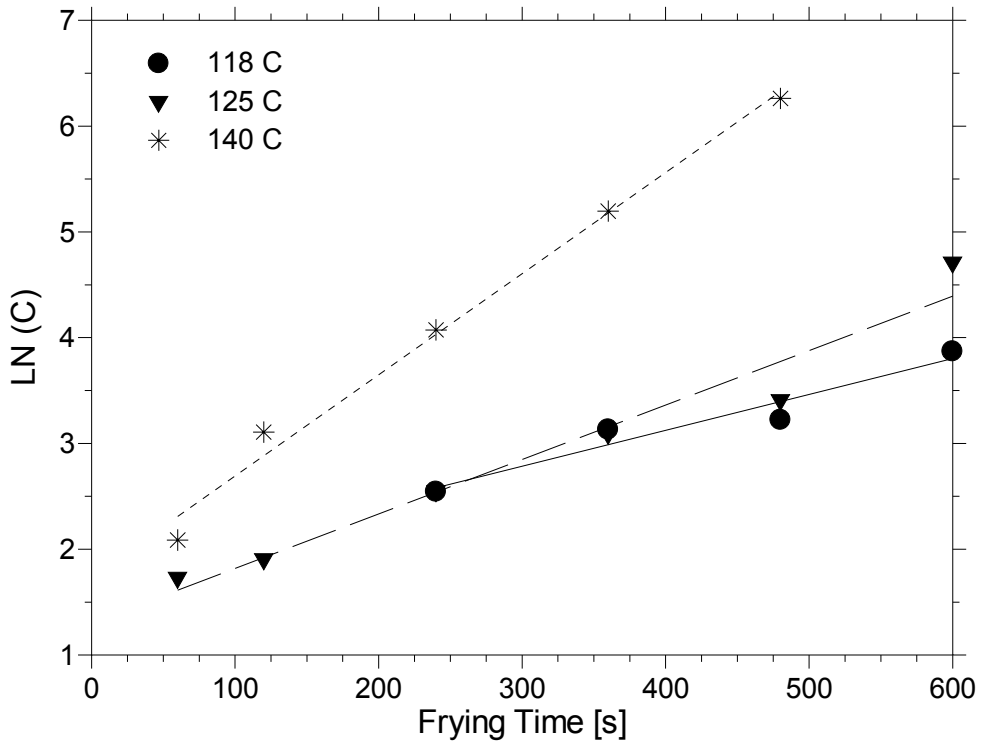


Figure 3-30. Relationship between acrylamide and frying time at different frying temperatures for potato chips fried under vacuum (10 Torr).

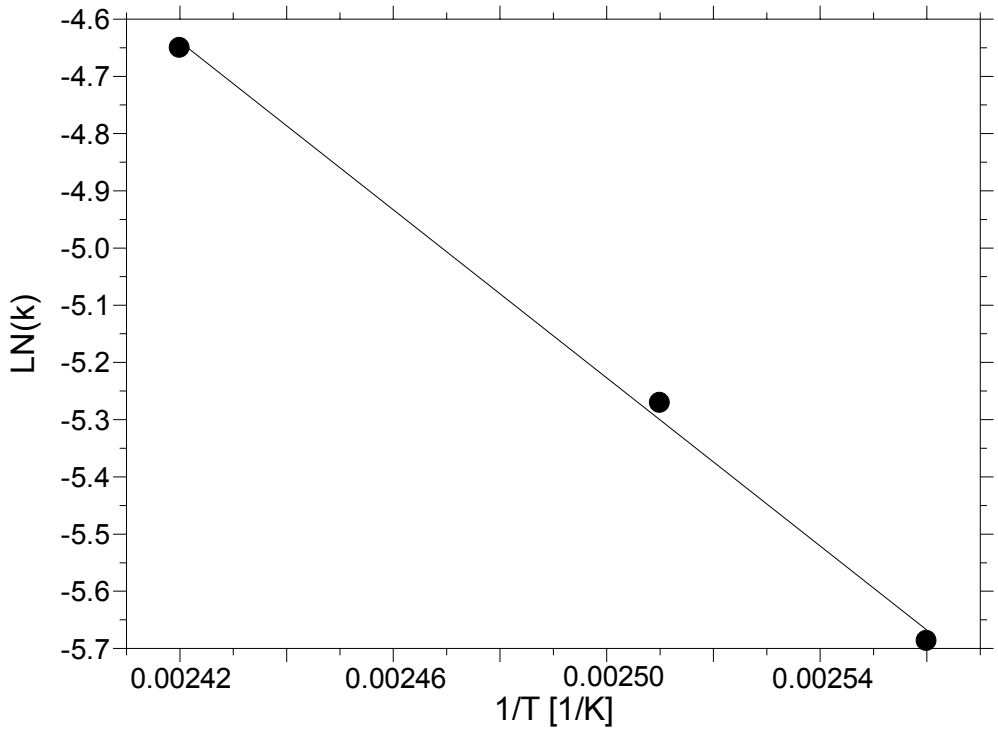


Figure 3-31. Effect of temperature on the rate constant *k* for acrylamide formation in potato chips fried under vacuum (10 Torr).

In summary, the kinetics of acrylamide content during traditional and vacuum frying are very different. During traditional frying the acrylamide content in potato chips increases with frying time and eventually levels off (Figure 3-25), while the acrylamide content in potato chips fried under vacuum (10 Torr) increases for all frying times (Figure 3-26). The difference in the behavior of the acrylamide content curves may be explained by the fact that temperatures were much lower during vacuum frying; therefore, it took longer at lower temperatures for acrylamide to start degrading; thus, acrylamide increases exponentially for longer times. However, the higher temperatures used during traditional frying will cause the acrylamide content of potato chips to remain constant over time, as explained in section 3.2.2.5.

The activation energies (E_a) for the traditional and vacuum frying processes were very different (21,830.9 J/mol vs. 61, 059.2 J/mol, respectively). A higher E_a means that temperature has a stronger influence on acrylamide content in potato chips fried under vacuum frying. Thus, when increasing the temperature from 165^{o}C to 180^{o}C (a 15^{o}C increase), the rate constant *k* increased only 6% during traditional frying. However, when increasing the temperature from 125^{o}C to 140^{o}C, the rate constant *k* for vacuum fried chips increased about 86%. This result indicates that at lower temperatures, temperature will have a greater impact on the increase of acrylamide content than at higher temperatures. This observation can be explained by the fact that at higher temperatures acrylamide starts degrading; therefore, the impact of temperature starts decreasing due to the formation and degradation of acrylamide (Taubert et al., 2004).

CHAPTER IV

EFFECT OF RAW POTATO COMPOSITION ON ACRYLAMIDE FORMATION IN POTATO CHIPS

4.1. Materials and Methods

4.1.1. Removal of reducing sugars and amino acids

Raw potato components (reducing sugars and amino acids) were removed to obtain a product with the same initial properties, since there is great variability between reducing sugar and amino acid content among tubers, as explained in section 2.3.1.3. Atlantic, provided by the Texas A & M University Potato Variety Development Program, was stored at 9±1°C and a relative humidity of 55%. The potatoes were reconditioned for 3 to 4 days at ambient temperature (23±2°C) before processing. The percentage of glucose in these tubers was 0.117 ± 0.005%, and the asparagine concentration was 98.7 ± 0.05 mg/100g after reconditioning, and were measured as explained in sections 4.1.2.2 and 4.1.2.3. The potatoes were washed, peeled, sliced (1.5 mm thickness) and placed in tap water at room temperature for about 5 min. They were then blotted dry with paper towels and about 100 g of slices were submerged in 500 ml of distilled water at 60°C while stirring frequently for 5 min. The slices were then removed and placed in 500 ml of a 50% ethanol solution at 40°C for 15 min., rinsed with 4 aliquots of 1 L cold distilled water, and stored in 1 L distilled water at 4°C overnight. A similar procedure was used by Martin and Ames (2001) to remove sugars and amino acids from potato tubers.

Glucose test paper strips were used to assure that glucose was successfully removed from the slices. Since glucose and amino acids have similar solubilities (Weast, 1976) it was assumed that fructose and amino acids were also removed.

4.1.2. Infusion of reducing sugars and amino acids.

Once reducing sugars and amino acids were leached from the slices, 100 g were infused in solutions with different concentrations of glucose and L-asparagine. All reagents were obtained from Sigma Chemical Co (St. Louis, MO). Glucose was the reducing sugar chosen because it is the most abundant in potatoes (Martin & Ames, 2001). L-asparagine was the amino acid selected for two reasons: it is the most abundant in potatoes (Martin & Ames, 2001) and it is believed to give origin to acrylamide through the Maillard reaction (Mottram et al., 2002).

Leached potato slices were rinsed twice with 1 L of distilled water at room temperature. They were blotted dry with paper towels, and placed in a beaker containing 200 ml of the infusing solution. A piece of parafilm was secured on the top of the beaker to avoid splashing during the infusion (Figure 4-1).

Figure 4-1. Beakers with potato slices placed in a vacuum oven prior to infusion.

Beakers containing the infusing solutions and the potato slices were allowed to equilibrate under vacuum (7.5 Torr) at room temperature. After one hour, the beakers were removed and the slices were blotted with paper towels.

Four different levels of asparagine and glucose were used as the infusing solutions (Table 4-1), for a total of 16 possible combinations (Table 4-2).

Table 4-1. Glucose and asparagines concentrations used to infuse leached potato slices.

Level	**Glucose (mg/ml)**	**Asparagine (mg/ml)**
Control	Non- infused	Non-Infused
Low	0.85	1
Medium	3.40	3
High	6.80	5

Table 4-2. Treatments with different glucose and asparagine (ASN) levels used to infuse leached potato slices.

Treatment	Glucose	ASN
1	None	None
2	Low	None
3	Med	None
4	High	None
5	None	Low
6	Low	Low
7	Med	Low
8	High	Low
9	None	Medium
10	Low	Medium
11	Med	Medium
12	High	Medium
13	None	High
14	Low	High
15	Med	High
16	High	High

4.1.2.1. Potato extracts

A sample of 20 g of infused potato slices was blended with 50 ml of distilled water for 1 min. This solution was then vacuum filtered using filter paper No. 4 (Whatman, Maidstone, England). The solid residue and the blender were washed several times with distilled water, and the eluate was added to the slurry. Two ml of α-aminobutyric acid (ABA, 10 mg/ml) were added as an internal standard. The slurry was transferred to a 200 ml volumetric balloon, and foam was removed by placing the balloons under vacuum for 5 min. The volume was then brought to 200 ml with distilled water.

4.1.2.2. Glucose determination

Dinitrosalycilic acid reagent (DNS) was used to determine the concentration of glucose in the potato extracts (Miller, 1959). The principle behind this method is explained in section 2.3.1.1.

Three ml of 1 % DNS reagent solution were added to 3 ml of potato extract in a test tube. The tubes were covered with parafilm to avoid evaporation. Two blanks were prepared using water with each batch. Test tubes were heated in water at 90^{o}C for 15 min to develop a red-brown color (Figure 4-2). They were removed from the hot water bath, and 1 ml of 40% potasium tartrate solution was added to stabilize the color. A cold water bath was used to cool the samples to room temperature for 20 min, and the absorbance was recorded with a spectrophotometer (Model UV-1601, Shimadzu Scientific Instruments, Japan) at 575 nm.

Figure 4-2. Test tubes being heated at 90^{o}C to develop the red-brown color for quantification of glucose.

A standard curve was prepared using different glucose concentrations (Figure 4-3). From the standard curve, x is the glucose concentration (mg/ml) and y is the absorbance, with y=0.03197+0.50865*x (R^2=0.997).

Samples were run in triplicates, and the concentration (g) of glucose in 100 g of slices was calculated as follows:

$$x = \left(\frac{y + 0.031669}{0.508653} \right) \frac{mg}{ml} * 200\ ml * \left(\frac{1\ g}{1000\ mg} \right) * 5 \qquad [4\text{-}1]$$

The limit of detection was determined to be 0.06 %.

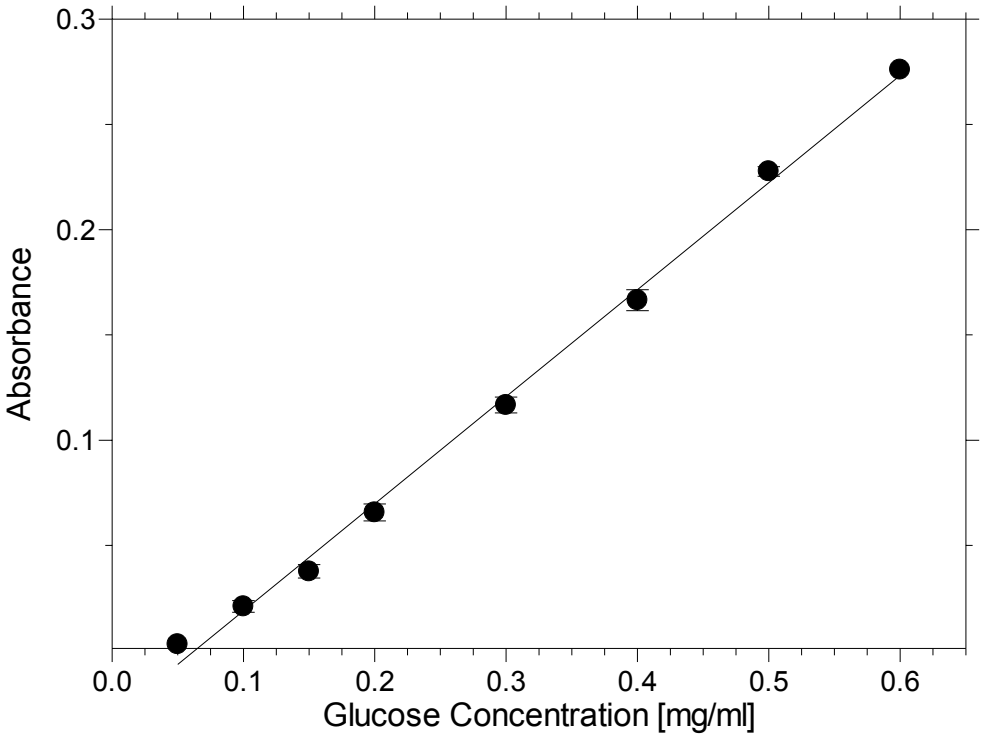


Figure 4-3. Glucose standard curve for the determination of reducing sugar concentration in the potato slices after infusion [y=-0.03197+0.50865*x, R^2=0.997].

4.1.2.3. Asparagine determination

Asparagine content was determined by precolumn derivatization with phenylisothiocyanate (PITC). The procedure was similar to the one followed by Hagen et al. (1992) and by Rodriguez-Saona & Wrolstad (1997).

Samples of potato slice extracts (25 µl) were placed in micro-centrifuge tubes with holes in the caps to allow evaporation during vacuum drying. The tube contents were then dried under vaccum (7.5 Torr) for 90 min. Then, 25 µl of a re-dry solution was added (LC grade methanol + 1 M LC grade sodium acetate trihydrate + triethylamine, 2+2+1, v/v/v) and the tubes and their contents were dried again under vacuum for 90 min. A derivatization solution (LC grade methanol + distilled water + triethylamine + PITC, 7+1+1+1, v/v/v/v) was added at this point and the tubes were kept at ambient temperature and pressure for 20 min. before drying under vacuum for 150 min. Derivatized samples were diluted with 1 mL distilled water, filtered through a .2 µm filter (Pall Corporation, Ann Arbor, MI), and injected into the HPLC. Four repetitions were performed for each treatment.

A High Performance Liquid Chromatograph (Dionex, Germering, Germany) equipped with a P680 pump, PDA-100 photodiode array detector, an ASI-100 automated sample injector, and Chromeleon 6.5 software (Dionex, Germering, Germany) was used in this study. The separations used a 25 x 0.46 cm Spherisorb ODS-2 column (Alltech Associates, Deerfield, IL) fitted with a 1 x 0.46 cm Spherisorb ODS-2 micro guard cartridge. The solvents were A: 0.14 M sodium acetates with 0.5 ml/l triethylamine (TEA) adjusted to pH 6 with glacial acetic acid, and B: 60 % acetonitrile in distilled

water. The elution program was similar to that used by Rodriguez-Saona & Wrolstad (1997): 10 min isocratic at 15 % B, 30 min linear gradient with 15-80 % B, and 5 min at 80 % B, using a flow rate of 0.20 µl/min. The effluent was monitored at 254 nm.

Standard curves were prepared for the internal standard (ABA) and for asparagine. Three concentrations were used for the ABA standard curve: 50 µg/ml, 100 µg/ml and 200 µg/ml (Figure 4-4).

The asparagine standard curve was developed from six different concentrations (10, 50, 100, 200, 400, and 600 µg/ml) plus the internal standard (100 µg/ml) (Figure 4-5).

Asparagine (ASN) concentration [mg/100 g] was calculated as follows:

$$ASN\left(\frac{mg}{100g}\right)=\frac{\left(A_1*\left(\frac{24.4799}{A_2}\right)\right)+0.674388}{0.209966}*\left(\frac{\mu g}{ml}\right)*(200\ ml)*\left(\frac{1\ mg}{1000\ \mu g}\right)*5 \qquad [4\text{-}2]$$

where A_1 (mAU*min) is the ASN area and A_2 (mAU*min) is the ABA area.

The limit of detection for asparagine was found to be 3.6 mg/100 g.

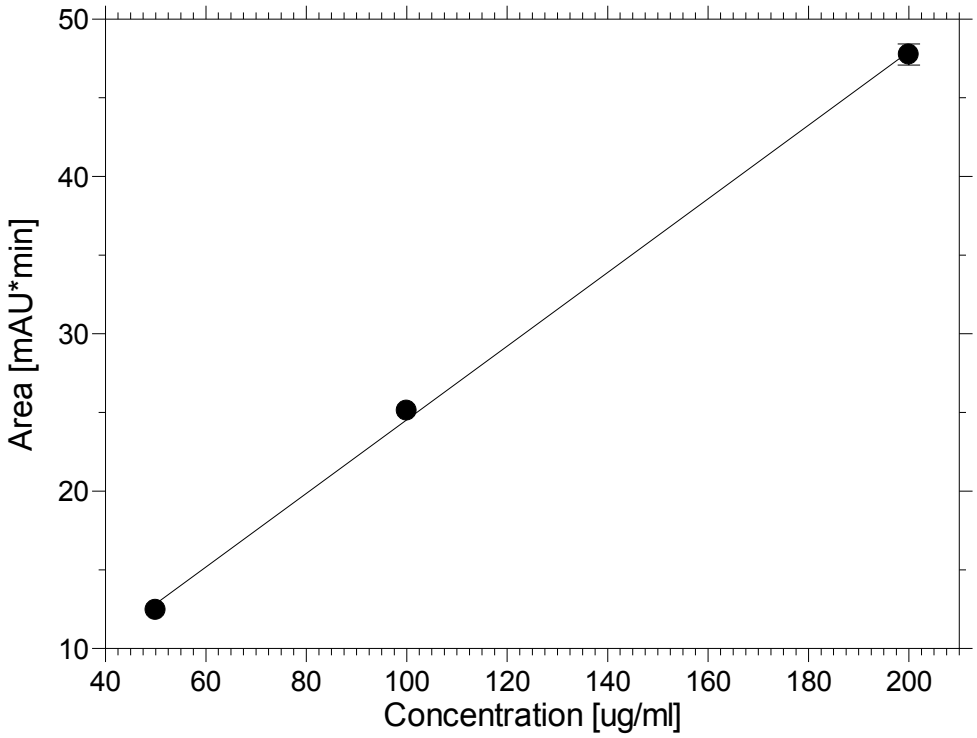


Figure 4-4. α–aminobutyric acid (ABA) standard curve [y=1.1305+0.234064*x, R^2=0.999].

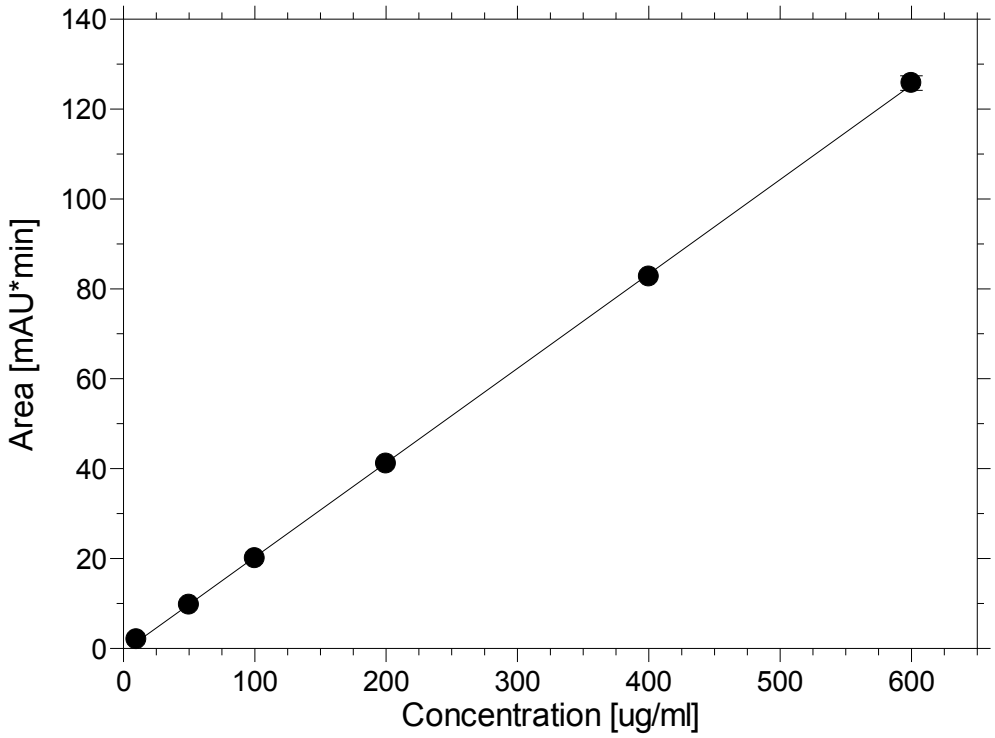


Figure 4-5. Asparagine (ASN) standard curve plus internal standard used to determine the concentration of asparagine in infused potato slices [y=-0.674388+0.209966*x, R^2=0.999].

4.1.3. Frying of infused slices

After the slices were infused with the desired amounts of glucose and asparagine, they were fried under atmospheric conditions at 165^{o}C for 4 min. The vegetable oil was changed after each treatment to ensure that glucose and asparagine from the previous runs did not affect the subsequent treatments. Fried slices were blotted dry with paper towels and allowed to cool at room temperature. The slices were then stored in low density polyethylene (LDPE) bags wrapped with aluminum foil to avoid degradation, and were placed in a dessicator until further analysis.

4.1.4. Product quality attributes

The properties studied on the potato chips were color and acrylamide content. These analyses were performed as described in sections 3.1.5.3 and 3.1.5.5.

4.2. Results and Discussion

4.2.1. Glucose and asparagine content of slices after infusion

The level of asparagine in a potato cultivar used for chipping (Saturna) has been reported to be 0.094% (Martin & Ames, 2001). This agrees with the level of asparagine found in the chipping cultivar Atlantic used in this study (0.099%). Rodriguez-Saona et al. (1997) created a potato model system with 0.230% asparagine, and Martin & Ames (2001) achieved an uptake of 0.210% asparagine in leached potato slices. In the present study, the concentration of asparagine in the slices ranged from 0 to 0.271%. This lower

range was chosen since higher concentrations in the infusing solutions were very hard to dissolve. However, this range still allows study of the effect of different asparagine levels on the formation of acrylamide in potato chips.

Low reducing sugar content (below 0.25% and preferably below 0.10%) is desired for the production of potato chips (Moreira et al., 1999). Rodriguez-Saona et al. (1997) developed a potato model system with 0.1% glucose, while Martin and Ames (2001) obtained a concentration of 0.260% glucose in infused potato slices. The range of glucose concentration in this study ranged from 0% to 0.4%. The different concentrations obtained for four levels of glucose and asparagine after infusing with the solutions in Table 4-1 are shown in Table 4-3.

Table 4-3. Glucose and asparagine content in infused slices.

Level	**Glucose (%)**	**Asparagine (%)**
None	<0.06	<0.004
Low	0.099 $\pm$ 0.006	0.062 $\pm$ 0.0017
Medium	0.233 $\pm$ 0.006	0.167 $\pm$ 0.0020
High	0.425 $\pm$ 0.016	0.271 $\pm$ 0.0026

Table 4-4 and Figure 4-6 show the influence of glucose and asparagine concentration (%) on the formation of acrylamide (ppm). It can be seen that at increasing glucose and asparagine concentrations, acrylamide content increases. The graphics software package PlotIt (version 3.2, 1999) was used to perform multiple linear

regression to obtain an equation that predicts the acrylamide formation as a function of glucose and asparagine content (x=glucose (%), y=asparagine (%), and z=acrylamide (ppm)):

$$z=0.1891-18.96x+52.22y+130.4x^2-650.7y^2+288.3xy-194x^3-28.88x^2y-166.8xy^2+1706y^3 \quad [4\text{-}3]$$

Figure 4-7 shows that there was a good correlation (R^2=0.988) between experimental and predicted values of acrylamide formation with Eqn (4-3).

Table 4-4. Influence of glucose and asparagine concentration on acrylamide formation in potato chips fried at 165°C for 4 min.

Glucose (%)	**Asparagine (%)**	**Acrylamide[a] (ppm)**
0	0	0.167±0.094 a
0.099	0	0.181±0.100 a
0.233	0	0.210±0.033 a
0.425	0	0.227±0.031 a
0	0.062	0.214±0.018 a
0.099	0.062	2.137±0.114 b
0.233	0.062	5.923±0.120 c
0.425	0.062	9.770±0.256 d
0	0.167	0.125±0.013 a
0.099	0.167	2.557±0.048 e
0.233	0.167	7.235±0.379 f
0.425	0.167	17.750±0.337 g
0	0.271	0.208±0.031 a
0.099	0.271	5.584±0.363 h
0.233	0.271	18.076±0.393 i
0.425	0.271	28.750±0.624 j

[a]Samples with the same letter are not significantly different (P<0.05).

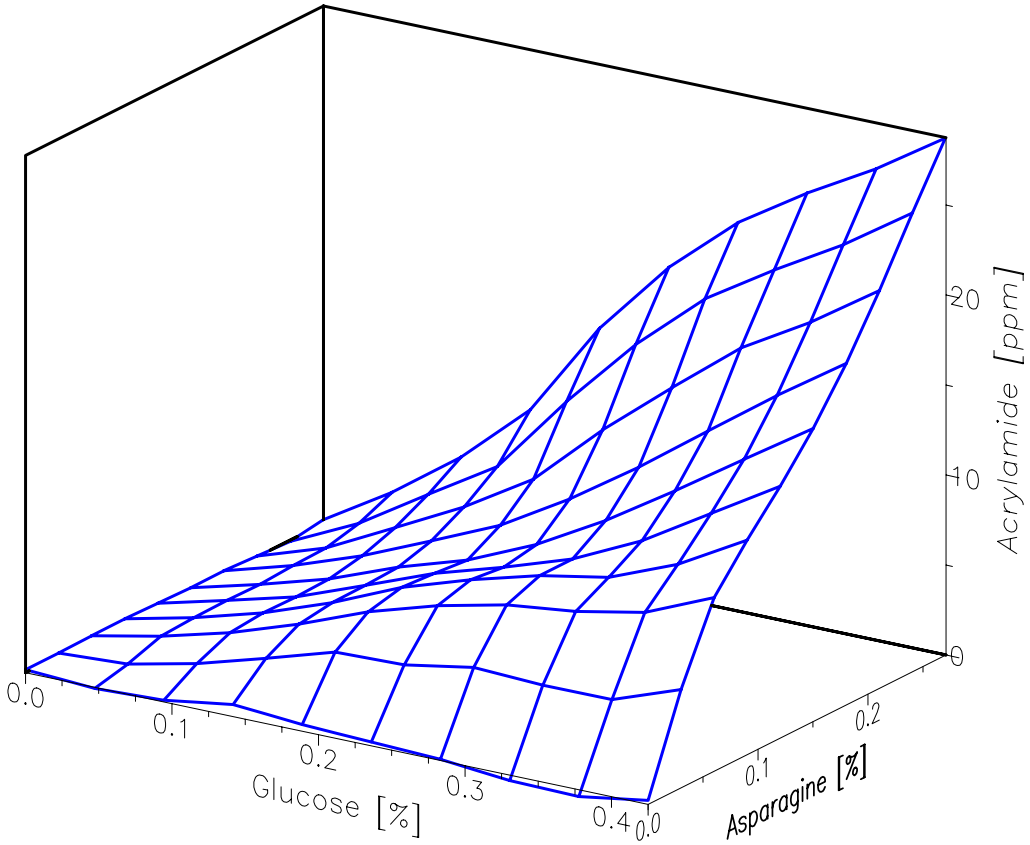


Figure 4-6. Influence of asparagine and glucose concentration on acrylamide formation in potato chips fried at 165^{o}C for 4 min.

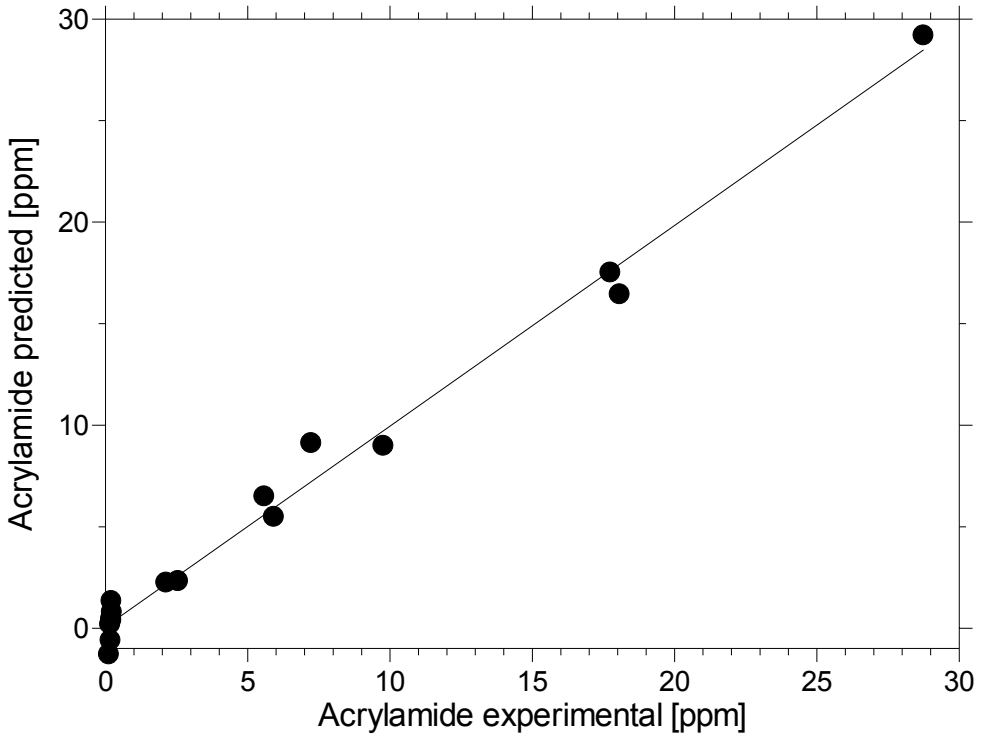


Figure 4-7. Correlation between experimental and regression values of acrylamide formation in potato chips fried at 165^{o}C for 4 min [y=0.0714154+0.988471*x, R^2=0.99].

Amrein et al. (2003) reported that the content of asparagine in 17 different cultivars varied between 0.201 to 0.425%, while the glucose content varied between 0.034 to 0.255%. They found that acrylamide formation in grated potatoes was proportional to the product of the concentrations of reducing sugars and asparagine. The maximum acrylamide content found by Amrein et al. (2003) was about 2 ppm at 120^{o}C.

Becalski et al. (2004) examined 66 potato samples to determine the influence of glucose and asparagine concentration on formation of acrylamide in French fries. The glucose concentration in the raw potatoes varied between 0.027% and 0.628%, and the asparagine concentration ranged from 0.149 to 1.137%. Acrylamide content varied from 0.050 to 1.8 ppm, reaching its maximum value at very high asparagine and glucose concentrations (0.862 and 0.5%, respectively).

The values of acrylamide obtained in this study were very high compared to reported acrylamide concentrations in potato products (Table 4-4). However, Zyzak et al. (2003) also obtained a high value of acrylamide content when using a model system. They varied the concentration of reducing sugars and amino acids in fabricated potato chips (made of potato flakes and water) to see which reactions resulted in acrylamide formation. The fabricated product had 1.20% asparagine and 0.67% glucose and it was fried at 206$\pm$2^{o}C for 30 s. The level of acrylamide obtained with this model system was 9.3 ppm. This value is still lower than the maximum value obtained in the present study

(about 28 ppm). The high temperature used by these authors (206±2°C versus 165°C) may explain the differences of magnitude in the formation of acrylamide, since very high temperatures may contribute to the degradation of this chemical (Taubert et al., 2004; Mottram et al., 2002)

Other authors have also found a high concentration of acrylamide when adding free asparagine to a food system. For instance, Amrein et al. (2004) found that adding asparagine (0.1%) to gingerbread dough increased acrylamide content from 0.2 ppm to 8 ppm when baking at 180°C for 3 min followed by 7 min at 190°C. This agrees with Surdyk et al. (2004) who found that at 0.7% fructose and 0.51% asparagine, acrylamide content in the crust of yeast-leavened wheat bread was 6 ppm when baking at 270°C for 15 min. Again, the higher temperatures used may explain the lower magnitude of acrylamide formation when adding asparagine in these products when compared to the results of the present study.

4.2.2. Influence of asparagine and glucose content in the color of infused potato slices

Figures 4-8 to 4-10 show the influence of glucose and asparagine levels on the *L*, *a*, and *b* color values. *L* is an indication of lightness, 0 being black and 100 white. It can be observed that the *L* values decrease with increasing glucose level, which means that the slices exhibit darker color. No obvious trend was recognized with increasing asparagine level on the *L* value of the potato chips.

The color-*a* increased as the glucose levels increased (Figure 4-9). A negative value is an indication of greenish color rather than red. A red color is usually an indication of overcooked potato chips: potato chips with higher glucose content looked more cooked than the ones with lower reducing sugar content. As with the *L* parameter, no specific trend could be seen with increasing asparagine levels.

The *b*-value indicates the blue-yellow chromaticity of the sample. The *b*-values were significantly higher as glucose levels increased (Figure 4-10). No significant difference was found in the *b*-values at different asparagines levels ($P<0.05$).

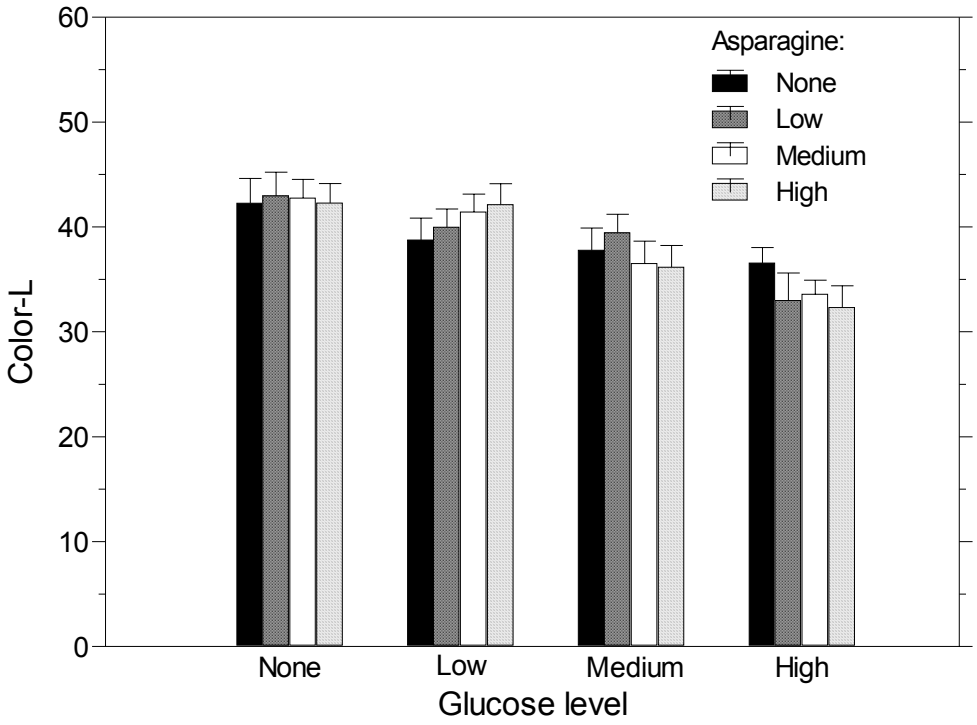


Figure 4-8. Influence of asparagine and glucose levels on color-*L* of potato chips fried under atmospheric conditions at 165^{o}C for 4 min.

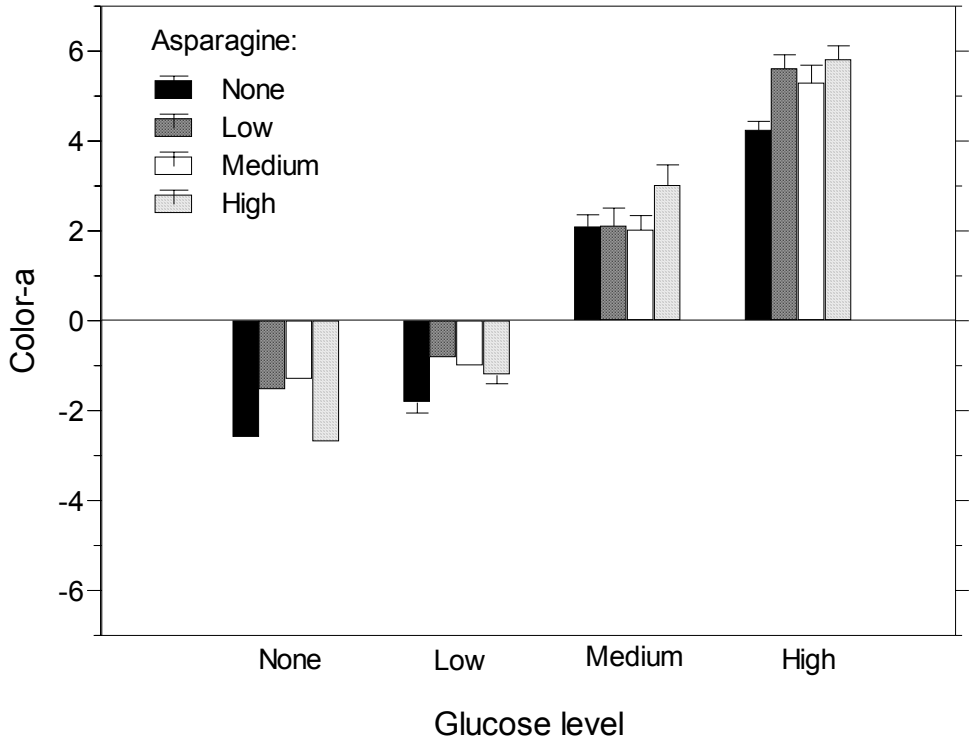


Figure 4-9. Influence of asparagine and glucose levels on color-*a* of potato chips fried under atmospheric conditions at 165^{o}C for 4 min.

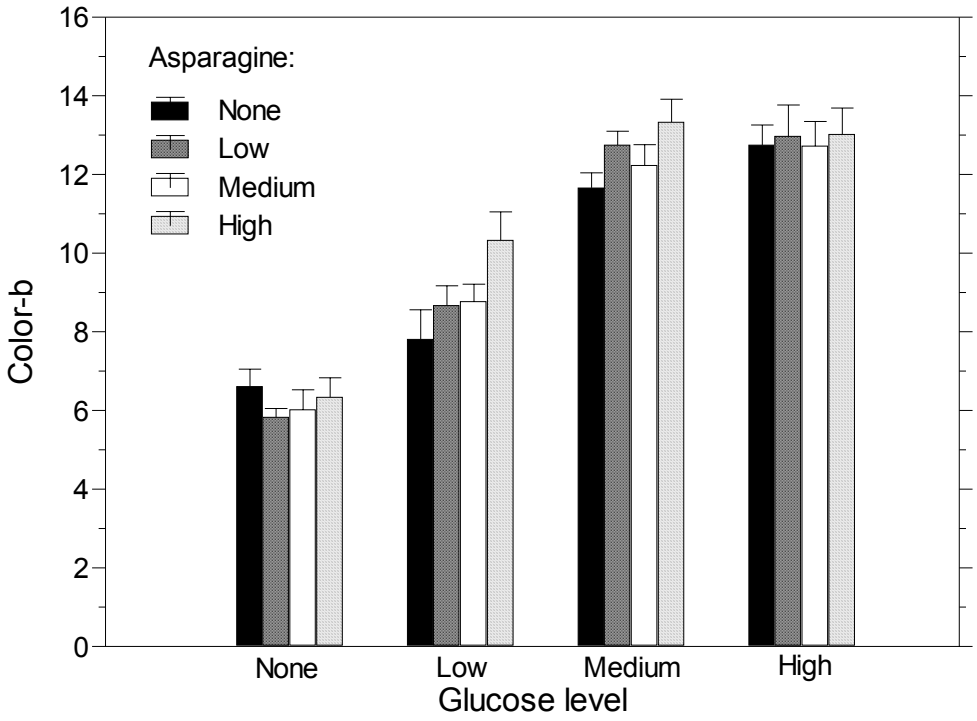


Figure 4-10. Influence of asparagine and glucose levels on color-*b* of potato chips fried under atmospheric conditions at 165^{o}C for 4 min.

4.2.3. Color and acrylamide formation

Figure 4-11 shows the linear relationship between color (*a*-value) and acrylamide concentration in potato chips at constant asparagine levels (Table 4-5).

Table 4-5: Regression coefficients for the relationship between acrylamide formation and color-*a*.

Asparagine (mg/100g)	Slope	Intercept	R^2
<3.6	8.3047	192.15	0.9867
61.6	1290.4	2785.1	0.9859
167	2493.4	3806.2	0.9730
270.6	3283.4	9123.9	0.9972

This agrees with the findings of Pedreschi et al. (2005). As the asparagine level increases, the higher the rate of acrylamide formation (indicated by the higher slope).

Figure 4-12 shows an exponential relationship between color-*b* and acrylamide formation (Table 4-6):

$$Color - b = m * \exp(n * g) \qquad [4\text{-}4]$$

where *m* and *n* are the regression coefficients and *g* is the glucose concentration.

From Figures 4-9 and 4-10, the color values -*a* and -*b* depend mainly on glucose concentration, as discussed previously (Section 4.2.3). However, since acrylamide formation depends on both glucose and asparagine concentrations, it can be seen (Figures 4-11 and 4-12) that at constant *a* and *b* values, acrylamide concentration increases depending on asparagine content. Therefore, even though the color of two chips may look similar, the acrylamide concentration of one may be higher than the

other due to higher asparagine content. Surdyk et al. (2004) also found that when adding asparagine to wheat bread dough, crust color did not change significantly, while acrylamide increased dramatically. Taubert et al. (2004) found that at a level of browning 2 ("golden brown"), the acrylamide content of 15-mm potato slices varied only between 1 and 1.5 ppm, but in 3 mm slices, acrylamide content varied from 2.5 to 13 ppm; in grated potatoes, acrylamide content ranged from 4 to 18 ppm. Since color continues to develop during the Maillard reaction, and acrylamide may start degrading, browning alone should not be used as the sole predictor of acrylamide formation (Taubert et al., 2004). As observed in Figures 4-13 to 4-16, for the same frying conditions (165^{o}C, 4 min) and at the same asparagine level, potato chips that look over fried tend to be the ones with higher acrylamide content.

Table 4-6. Regression coefficients for the relationship between acrylamide formation and color-*b*.

Asparagine (mg/100g)	***m***	***n***	**R^2**
<3.6	0.1238	0.0466	0.9887
61.6	0.0190	0.4757	0.9315
167	0.0036	0.6604	0.9272
270.6	0.0034	0.6767	0.9696

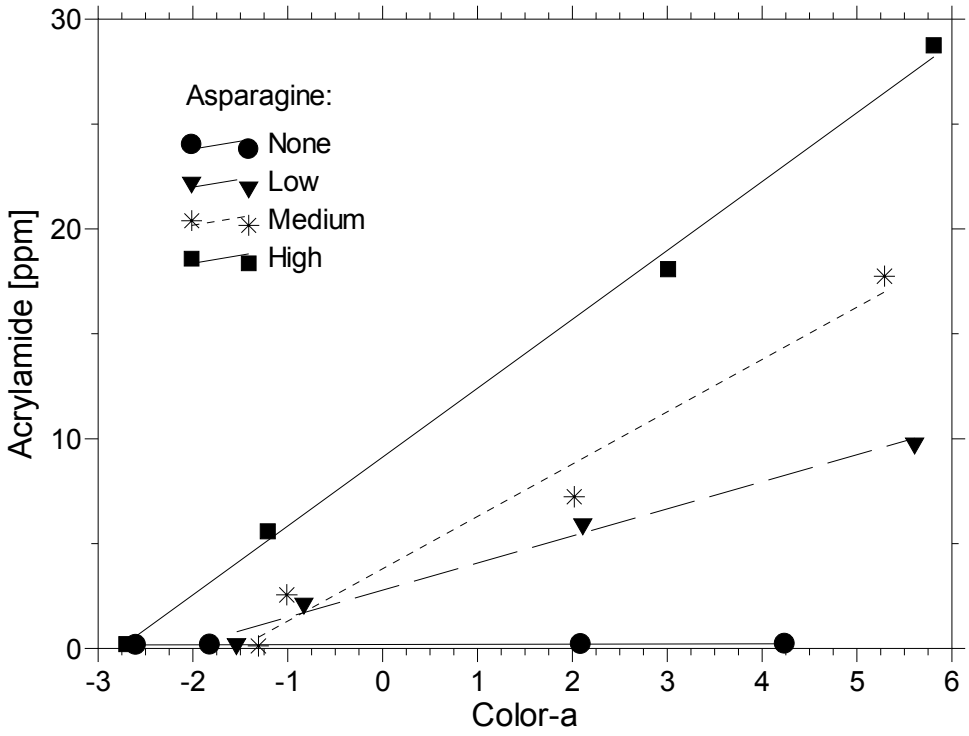


Figure 4-11. Acrylamide content vs. color-*a* for potato chips with different glucose and asparagine concentrations.

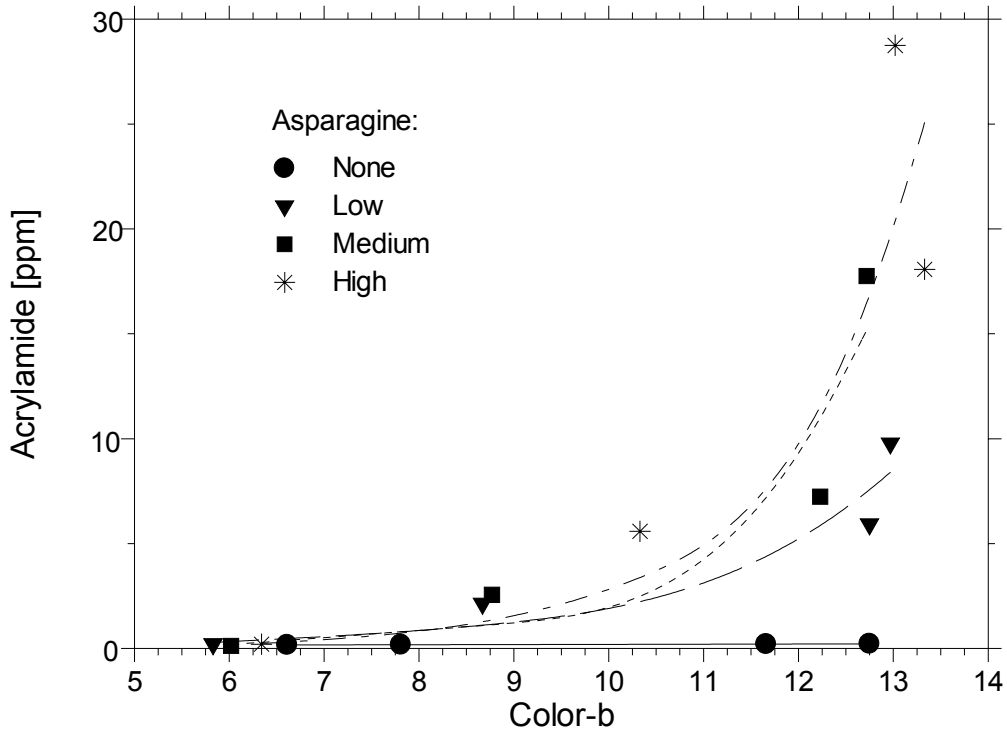
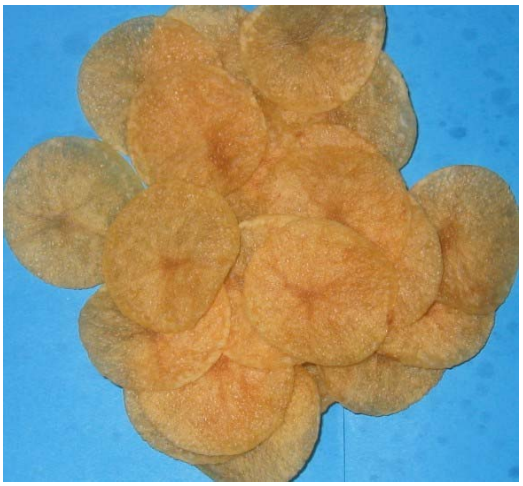


Figure 4-12. Acrylamide content vs. color-*b* for potato chips with different glucose and asparagine concentrations.

Glucose: none; asparagine: none; acrylamide: 167± 94 ppb.

Glucose: low; asparagine: none; acrylamide: 181± 100 ppb.

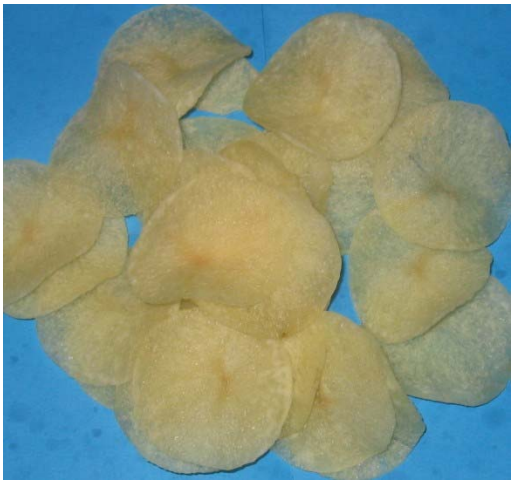
Glucose: medium; asparagine: none; acrylamide: 210± 33 ppb.



Glucose: high; asparagine: none; acrylamide: 227± 31 ppb.

Figure 4-13. Comparison of treatments with no asparagine (none level).

Glucose: none; asparagine: low; acrylamide: 214± 18 ppb.



Glucose: low; asparagine: low; acrylamide: 2137±114 ppb.

Glucose: medium; asparagine: low; acrylamide: 5923± 120 ppb.

Glucose: high; asparagine: low; acrylamide: 9770± 256 ppb.

Figure 4-14. Comparison of treatments with low asparagine level.

Glucose: none; asparagine: medium; acrylamide: 125± 13 ppb.

Glucose: low; asparagine: medium; acrylamide: 2257± 48 ppb.

Glucose: medium; asparagine: medium; acrylamide: 7235± 379 ppb.

Glucose: high; asparagine: medium; acrylamide: 17750± 337 ppb.

Figure 4-15. Comparison of treatments with medium asparagine level.

Glucose: none; asparagine: high;
acrylamide: 208± 31 ppb.

Glucose: low; asparagine: high;
acrylamide: 5584±363 ppb.

Glucose: medium; asparagine: high;
acrylamide: 18076± 393 ppb.

Glucose: high; asparagine: high;
acrylamide: 28750± 625 ppb.

Figure 4-16. Comparison of treatments with high asparagine level.

CHAPTER V

CONCLUSIONS

This study focused on determining how the variables of frying process, potato cultivar, and potato composition (asparagine and reducing sugars) affect the formation of acrylamide.

The first part of this research evaluated whether using different cultivars had an effect on acrylamide content. Seven potato cultivars were processed under two frying conditions: vacuum (10 Torr, 118^{o}C) and traditional (atmospheric pressure, 165^{o}C). The following conclusions were reached:

- White Rose produced chips with the highest acrylamide content under both processes (3899 ppb under traditional, 242 ppb under vacuum). This cultivar also had the highest reducing sugar content.
- Cultivars with lower reducing sugar content (NDTX4930-5W and ATX85404-8W) had the lowest acrylamide concentrations under both frying processes.
- The acrylamide content of all cultivars was significantly reduced when frying under vacuum conditions (10 Torr, 118^{o}C).
- For most cultivars, when the traditional method was used, the potato chips had a higher *L*-value, which indicates a lighter color.
- The *a*-value, which indicates the green-red chromaticity of the samples, was significantly lower for vacuum fried chips than for the traditionally fried potatoes, indicating that chips under traditional frying looked more over-fried (darker) than those fried under vacuum frying.

- For all cultivars, the traditionally fried chips had a higher *b*-value than vacuum fried chips, i.e., they were more yellowish than vacuum fried chips.
- There were no significant differences in texture when different frying processes were used.

The cultivar Atlantic was chosen to analyze the effect of frying processing parameters on the final product quality attributes (moisture content, oil content, color, texture, and acrylamide formation) because it is a popular chipping variety. The results are summarized as follows:

- The diffusion coefficient (D_e) for moisture loss of chips during frying increased as oil temperature increased for both frying methods.
- The effect of temperature on the diffusion coefficient (D_e) was modeled using an Arrhenius relationship.
- Final oil content was not significantly different for both frying methods.
- The oil absorption rate increased with increasing oil temperature.
- The final oil content was lower for chips fried under vacuum conditions (118^{o}C, 10 Torr) than under atmospheric conditions (165^{o}C).
- No significant differences were found in texture for potato chips fried using either method.
- Color-*L* decreased as time increased for potato chips fried under atmospheric conditions, i.e., the chips became darker as frying time increased.

- Color-*b* was higher for chips fried under atmospheric conditions than for chips fried under vacuum conditions, i.e., atmospheric fried chips looked more yellowish than vacuum fried chips.
- Acrylamide content increased with time for all temperatures, for both frying methods.
- The increase in acrylamide content during vacuum frying was less drastic than for traditional frying.
- There was a 94% decrease in acrylamide content when potatoes were fried to the same final moisture content (1.5%$\pm$0.3% w.b.) under vacuum compared to those fried under atmospheric conditions.
- Kinetics of acrylamide content during traditional frying was modeled by using a special case of the logistic kinetic model. Acrylamide content increased (up to about 300 s, 240 s, and 140 s at 150^{o}C, 165^{o}C and 180^{o}C, respectively) and eventually reached an equilibrium value (484, 847, and 1123 ppb at 150^{o}C, 165^{o}C and 180^{o}C, respectively).
- The influence of time on acrylamide accumulation in vacuum fried chips was best described by first order kinetics. Acrylamide content during vacuum frying kept increasing exponentially.

A model system was developed to study the effect of composition (reducing sugars and asparagine) on acrylamide content during frying in a traditional fryer. The following conclusions were drawn from this study:

- Acrylamide increased with increased asparagine and glucose content.

- No significant differences were found in color values [*L*, *a*, and *b*] with increasing asparagine concentration.
- The color-*L* values decreased with increasing glucose level, which means that the slices exhibited a darker color.
- The color-*a* values increased as the glucose concentration increased. Potato chips with higher glucose content appeared more over-fried than those with lower reducing sugar content.
- The color-*b* values increased as glucose concentration increased. Potato chips with higher glucose concentration looked more yellowish.
- A linear relationship was found between acrylamide and color-*a*, and an exponential relationship was found between acrylamide and color-*b* at constant asparagine concentrations. When asparagine concentration increased, the acrylamide content increased even when the slices exhibited very similar color values (*a* and *b*). Hence, color alone should not be used as a predictor for acrylamide concentration.

CHAPTER VI

RECOMMENDATIONS FOR FURTHER STUDY

Recommendations for future research on acrylamide formation and its potential reduction include:

- Pre-treat potato slices by drying before frying to reduce the frying time.
- Pre-treat potato slices by blanching and perform kinetic studies on acrylamide formation in blanched samples.
- Pre-treat potato slices by lowering the pH and study the interaction with atmospheric and vacuum frying.
- Prepare model potato systems with mixtures of other reducing sugars (such as fructose) and other amino acids (such as glutamine) to evaluate the interaction of food components.
- Perform kinetic studies using model systems with different asparagine and glucose combinations to ensure homogeneity of raw potatoes.
- Evaluate acrylamide formation in other food systems such as tortilla chips during atmospheric and vacuum frying.
- Determine the oil and product temperature profile during traditional and vacuum frying.
- Perform kinetics of acrylamide formation at different vacuum levels.
- Study acrylamide formation in different potato shapes (such as grated potatoes and French fries) during traditional and vacuum frying.
- Model acrylamide content in potato chips at higher temperatures.

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APPENDIX A

Table A1. Proximate analysis of potato (Lusas & Rooney, 2001).

Constituent	**Minimum (%)**	**Maximum (%)**
Water	63	87
Total Solids	13	37
Protein	0.7	4.6
Fat	0.02	0.96
Carbohydrates	13	30.5
Ash (minerals)	0.44	1.99

Table A2. Effect of frying time and oil temperature on moisture and oil content in potato chips fried under traditional frying.

T_{oil} (^{o}C)	Frying Time (s)	IMC (% w.b.)	FMC (% w.b.)	OC (% w.b.)
150	60	79.95±0.82	52.12±0.03	7.57±0.04
150	90	79.95±0.82	37.91±0.09	16.08±0.13
150	120	79.96±2.00	24.96±0.22	19.38±0.32
150	150	79.96±2.00	7.98±0.56	24.80±0.39
150	180	79.05±2.08	3.52±0.21	30.45±0.13
150	240	79.05±2.08	3.08±0.11	35.68±0.13
150	300	81.29±0.21	2.04±0.44	36.90±0.70
150	360	79.95±0.21	0.70±0.14	36.11±0.66
165	45	77.69±1.93	54.63±0.07	3.59±0.59
165	60	77.69±1.93	42.20±0.19	10.92±0.02
165	90	78.25±3.41	30.72±0.26	17.84±0.18
165	120	78.25±3.41	10.85±0.24	23.66±0.16
165	150	81.42±1.48	4.20±0.67	25.57±0.38
165	180	81.42±1.48	2.97±0.66	29.35±0.15
165	240	79.15±1.90	1.80±0.43	34.57±0.25
165	300	79.15±1.90	1.58±0.33	37.04±0.05
180	30	77.78±1.04	52.73±0.78	5.93±0.03
180	60	77.78±1.04	39.86±0.24	13.03±0.32
180	90	79.50±0.74	18.03±0.51	22.79±0.09
180	120	79.50±0.74	2.85±0.17	27.68±0.06
180	150	76.91±1.03	2.39±0.44	28.05±0.39
180	180	76.91±1.03	1.61±0.41	31.05±0.33
180	240	82.08±0.79	1.41±0.59	34.73±0.16
180	300	82.08±0.79	0.92±0.28	36.76±0.76

Tests were performed in triplicate. T_{oil}=oil temperature, IMC= initial moisture content, FMC= final moisture content, OC= oil content.

Table A3. Effect of frying time and oil temperature on moisture and final oil content of potato chips fried under vacuum (10 Torr).

T_{oil} (^{o}C)	Frying Time (s)	IMC (% w.b.)	FMC (% w.b.)	OC (% w.b.)
118	90	78.94±2.47	53.32±0.08	2.68±0.78
118	120	81.72±0.87	40.78±0.44	6.01±0.25
118	150	81.72±0.87	29.18±0.27	14.66±0.03
118	180	78.19±1.80	18.65±0.36	18.66±0.12
118	240	78.19±1.80	6.32±0.87	27.70±0.04
118	300	80.09±1.42	4.06±0.19	26.81±0.09
118	360	80.09±1.42	2.69±0.25	29.15±0.20
118	480	77.94±0.96	1.91±0.41	28.34±0.08
118	600	77.94±0.96	1.68±0.22	29.68±0.24
125	60	78.72±1.52	60.17±0.25	3.74±0.28
125	90	78.72±1.52	48.22±0.16	7.62±0.24
125	120	80.04±2.85	34.92±0.73	10.72±0.43
125	150	80.04±2.85	18.52±0.26	13.84±0.51
125	180	80.00±2.44	8.10±0.28	15.12±2.02
125	240	80.00±2.44	2.54±0.13	21.05±0.21
125	300	80.70±2.23	1.50±0.09	29.23±0.52
125	360	80.70±2.23	1.60±0.27	29.26±0.19
125	480	79.75±1.43	1.27±0.26	30.91±0.30
125	600	79.75±1.43	1.01±0.51	27.47±0.53
140	30	80.57±1.18	62.16±0.20	3.19±0.22
140	60	80.57±1.18	41.15±0.76	14.80±0.38
140	90	79.05±0.83	21.60±0.44	25.19±0.58
140	120	79.05±0.83	10.48±0.49	33.98±0.39
140	150	82.36±0.70	4.55±0.76	34.54±0.13
140	180	78.82±2.76	2.48±0.37	30.46±0.52
140	240	79.28±1.54	1.18±0.15	34.19±0.12
140	300	79.28±1.54	1.68±0.47	31.80±0.11
140	360	79.65±0.66	1.37±0.28	28.54±0.48
140	420	79.65±0.66	1.11±0.14	28.78±0.18

Tests were performed at least in triplicate. T_{oil}=oil temperature, IMC= initial moisture content, FMC= final moisture content, OC=final oil content.

Table A4. Effect of frying time, oil temperature and pressure on acrylamide content of potato chips.

Pressure (Torr)	T_{oil} (^{o}C)	Frying Time (s)	Acrylamide [ppb]
760	150	60	36±4
760	150	120	32±6
760	150	180	37±1
760	150	240	193±31
760	150	300	370±34
760	150	360	484±30
760	165	60	6±8
760	165	120	54±9
760	165	180	511±88
760	165	240	753±106
760	165	360	847±80
760	180	30	14±2
760	180	60	28±0
760	180	150	912±47
760	180	180	983±94
760	180	240	1123±13
10	118	60	ND
10	118	120	ND
10	118	240	13±4
10	118	360	23±4
10	118	480	25±3
10	118	600	48±1
10	125	60	6±1
10	125	120	7±2
10	125	240	13±1
10	125	360	22±3
10	125	480	30±2
10	125	600	111±18
10	140	60	8±1
10	140	120	22±4
10	140	240	59±2
10	140	360	181±8
10	140	480	524±26
10	140	600	289±25

Four repetitions were performed. T_{oil}=oil temperature.

Table A5. Effect of frying time and temperature on color [*L*-, *a*- and *b*- values] in potato chips fried under traditional frying.

T_{oil} (^{o}C)	Frying Time (s)	Color *L*-value	Color *a*-value	Color *b*-value
150	60	51.66±1.12	-3.65±0.11	6.45±0.61
150	120	50.63±1.96	-3.19±0.08	8.61±0.43
150	180	51.92±3.02	-3.47±0.28	9.61±0.69
150	240	49.47±3.91	-2.96±0.81	12.07±1.01
150	300	46.35±1.95	-1.62±1.20	14.23±0.88
150	360	45.67±3.38	-2.84±1.07	14.22±0.93
165	60	47.94±0.89	-3.46±0.11	7.68±0.56
165	120	48.80±2.43	-3.18±0.11	9.62±0.49
165	180	47.51±2.24	-2.91±0.41	14.91±0.83
165	240	42.13±2.41	-2.02±0.21	16.31±1.37
165	300	42.93±1.79	-2.69±0.43	16.93±0.64
165	360	39.85±2.62	-1.31±0.25	17.41±0.90
180	30	47.90±0.67	-3.51±0.14	7.41±0.48
180	60	47.90±1.37	-3.42±0.15	8.11±0.49
180	120	46.75±3.15	-3.23±0.24	11.83±0.87
180	150	46.99±2.21	-3.18±0.36	15.56±0.77
180	180	45.37±2.74	-3.20±0.23	16.89±1.04
180	240	41.83±2.33	-1.96±0.14	18.49±0.99

Twenty repetitions were performed. T_{oil}=oil temperature.

Table A6. Effect of frying time and temperature on color [*L*-, *a*- and *b*- values] of potato chips fried under vacuum (10 Torr) frying.

T_{oil} (^{o}C)	Frying Time (s)	Color (*L*-value)	Color (*a*-value)	Color (*b*-value)
118	60	49.10±0.74	-4.01±0.27	8.41±0.71
118	120	46.18±0.92	-3.54±0.07	6.95±0.45
118	240	40.95±2.05	-3.58±0.17	10.32±0.39
118	360	46.01±3.01	-3.42±0.17	10.12±1.08
118	480	47.15±2.33	-3.79±0.17	11.20±0.68
118	600	47.25±2.37	-3.85±0.51	12.21±1.24
125	60	50.92±0.53	-3.80±0.17	7.24±0.53
125	120	40.79±2.07	-2.95±0.16	7.93±0.60
125	240	44.73±3.93	-3.44±0.18	10.13±0.82
125	360	43.75±2.39	-3.67±0.18	10.93±0.46
125	480	45.00±2.30	-3.64±0.17	11.15±0.61
125	600	42.52±2.03	-2.95±0.54	13.21±1.14
140	60	48.31±0.65	-3.78±0.14	7.06±0.70
140	120	43.27±2.99	-3.25±0.49	10.13±1.29
140	240	46.93±3.85	-3.74±0.11	12.00±1.02
140	360	43.98±3.28	-2.63±0.35	13.43±0.92
140	480	42.83±3.17	-2.45±1.36	14.07±1.20
140	600	44.18±2.18	-2.57±0.65	13.55±0.79

Twenty repetitions were performed. T_{oil}=oil temperature.

Table A7. Effect of time, oil temperature and pressure on hardness of potato chips.

Pressure (Torr)	T_{oil} (^{o}C)	Frying Time (s)	Hardness (N)
760	150	180	3.74±0.74
760	150	240	3.38±0.67
760	150	300	2.88±0.70
760	150	360	3.39±0.79
760	165	180	3.52±0.88
760	165	240	2.35±0.28
760	165	300	3.27±0.96
760	165	360	2.29±0.35
760	180	120	3.05±0.76
760	180	150	2.54±0.42
760	180	180	3.20±0.74
760	180	240	2.42±0.42
10	118	240	3.32±0.64
10	118	360	2.90±0.42
10	118	480	2.98±0.66
10	118	600	3.12±0.49
10	125	240	3.26±1.08
10	125	360	3.50±0.68
10	125	480	3.84±0.60
10	125	600	2.76±0.52
10	140	240	4.67±0.81
10	140	360	3.47±0.73
10	140	480	3.40±0.49
10	140	600	3.35±0.79

Tests were performed 10 times. T_{oil}=oil temperature.

Table A8. Effect of glucose and asparagine concentration on color [*L*-, *a*- and *b*-values] of potato chips fried under traditional frying (4 min, 165^{o}C).

Glucose (%)	Asparagine (mg/100 g)	Color (*L*-value)	Color (*a*-value)	Color (*b*-value)
0	0	42.27±2.35	-2.60±0.09	6.61±0.44
0.099±0.006	0	38.76±2.07	-1.82±0.23	7.81±0.75
0.233±0.006	0	37.78±2.12	2.09±0.27	11.66±0.38
0.425±0.016	0	36.55±1.49	4.24±0.20	12.75±0.51
0	61.6±1.7	42.97±2.25	-1.54±0.07	5.83±0.22
0.099±0.006	61.6±1.7	39.97±1.74	-0.83±0.07	8.67±0.50
0.233±0.006	61.6±1.7	39.46±1.74	2.11±0.40	12.75±0.35
0.425±0.016	61.6±1.7	33.00±2.61	5.61±0.31	12.97±0.80
0	167±2.0	42.76±1.77	-1.31±0.08	6.02±0.51
0.099±0.006	167±2.0	41.43±1.70	-1.01±0.07	8.77±0.44
0.233±0.006	167±2.0	36.52±2.13	2.02±0.32	12.23±0.53
0.425±0.016	167±2.0	33.59±1.34	5.29±0.40	12.72±0.63
0	270.6±2.6	42.28±1.85	-2.70±0.06	6.34±0.49
0.099±0.006	270.6±2.6	42.13±1.99	-1.21±0.19	10.33±0.72
0.233±0.006	270.6±2.6	36.18±2.06	3.01±0.46	13.33±0.58
0.425±0.016	270.6±2.6	32.32±2.07	5.81±0.31	13.02±0.67

Tests were performed 20 times.

VITA

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