

MICROSTRUCTURE AND SURFACE CHARACTERIZATION OF FRIED BATTER COATINGS

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ABSTRACT

Deep-fat frying of foods is a popular way to prepare tasty foods quickly. However, these foods are high in calories and fat, which over the last decades has led to the need for research on methods of reducing fat absorption during frying. Recent findings showed that fat absorption is largely a surface phenomenon and it is influenced by surface characteristics and microstructure. Therefore, study of surface and microstructure of fried foods, as affected by pre-treatment techniques, is of great interest to better understand the mechanisms of fat absorption. In this study the effects of batter formulation, pre-drying time and pre-heating temperature on surface characteristics and microstructure of fried batter using scanning electron microscopy and image processing techniques were investigated. The study also evaluated different fractions of inter-particle spaces of fried batter when occupied by air, moisture and fat. The effects of the parameters on porosity and mass transfer of fried batter were also studied.

Two different sets of batter systems were used. First, wheat flour batter was pre-heated at different temperatures namely 60, 70, and 80°C for 10 min. The batter samples were then deep fried at 180±2°C in canola oil. Batter pre-heated at 60°C showed higher moisture content, lower fat content and lower porosity than non-pre-heated batter and batters pre-heated at 70 and 80°C. Therefore 60°C pre-heating temperature was chosen to study the effect of different batter formulations (namely 100 g wheat flour to 0 g rice flour, 75 g to 25 g, 50 g to 50 g, 25 g to 75 g, and 0 g to 100 g) on moisture and fat content, and porosity. Batters formulated with higher amount of wheat flour showed higher moisture content, and lower fat content and porosity than batters formulated with higher amount of rice flour, which suggested that wheat flour is a proper ingredient compare to rice flour to reduce fat content of battered products.

Furthermore, the effect of batter formulations and pre-drying times on different fractions of fat in fried batter was studied. Different mathematical models for predicting moisture diffusivity and oil transfer during frying were evaluated. Batter with 100 g wheat flour was found to have higher surface fat and total fat contents, while its penetrated fat content was lower than that with other batters. Batter with no pre-drying showed higher surface, penetrated and total fat content than other systems. Moisture diffusivity ranged between 5.47×10^{-6} and 7.38×10^{-6} m²/min. Page model was found as the best fit model to evaluate the oil transfer rate constants.

The influence of batter formulation and pre-drying time on different fraction of inter-particle spaces in fried batter that are occupied by air, moisture, or fat was evaluated. Higher rice flour content in batter significantly increased the fraction of inter-particle spaces occupied by air (SOA), and decreased the fraction of spaces occupied by moisture (SOM) and fat (SOF). Longer pre-drying times showed greater fraction of spaces occupied by air, and smaller fraction of spaces occupied by moisture and fat. SOA, SOM, and SOF of the batter systems ranged from 2.20 to 46.03%, 2.55 to 47.07%, and 0.35 to 11.11%, respectively.

Surface characteristic of fried batters when prepared from different combination of wheat and rice flours was investigated. Variable pressure scanning electron microscopy (VP-SEM) was used to study the surface microstructure of batters. Parameters such as the total number, mean area, the biggest area, ratio and roundness of the generated ruptures on the coating surface after frying were used to characterize the microstructure of fried batters. Batters formulated only with wheat flour showed the highest number of ruptures whereas batters formulated only with rice flour showed the least. Roundness of the ruptures ranged from 0.19 to 0.35. The higher the amount of rice flour used in the formulation of batter, the higher the area of holes and cracks generated on surface. Fat content was directly correlated with the ratio of the observed holes and

cracks generated on the surface ($|r| = 0.72$), whereas moisture loss was correlated with ratio ($|r| = 0.67$) and mean area ($|r| = 0.77$).

Fractal dimension (FD) and lacunarity were applied to study the structure and irregularities of surface of batters with different formulations during frying. Using scanning electron microscopy, images of batter surfaces were captured, and analyzed via FracLac plug-in that was applied in ImageJ software. FD ranged between 2.79 and 2.82. Application of higher amount of wheat flour in batter preparation resulted in higher FD values compared to batters with higher amount of rice flour. FD significantly ($P < 0.05$) increased during frying. There was a high positive correlation between FD and fat uptake for all batter formulations ($|r| = 0.91-0.99$). Lacunarity, as a measure of degree of heterogeneity of batter ruptures, ranged between 1.19 and 1.25. The lacunarity results showed that the size and shape of ruptures, i.e. cracks and holes, generated on the surface of batters during frying was approximately uniform.

RÉSUMÉ

La friture des aliments est un moyen populaire de préparer rapidement des aliments savoureux. Toutefois, ces aliments sont riches en calories et en matière grasse, ce qui a conduit au cours des dernières décennies à des recherches sur des méthodes de réduction de l'absorption des graisses pendant la cuisson. Récemment, des résultats ont montré que l'absorption des graisses est un phénomène de surface et que la plupart de l'huile est absorbée lorsque la nourriture est retirée de l'huile de friture. Par conséquent, l'étude de la surface et de la microstructure des aliments frits soumis à des prétraitements représente une opportunité pour mieux comprendre les mécanismes de l'absorption des graisses. Dans la présente étude, les effets de la formulation de la pâte, du temps de pré-séchage et de la température de préchauffage sur les caractéristiques de surface et la microstructure de la pâte frite ont été étudiés en utilisant les techniques de microscopie électronique à balayage et de traitement de l'image. Les différentes fractions d'espace entre les particules de pâte frite, occupées par l'air, l'humidité et la graisse, ont également été évaluées.

Les effets de la température de préchauffage et de la formulation de la pâte sur le transfert de masse et la porosité de la pâte frite ont été étudiés. Deux ensembles différents de système de pâte à frire ont été utilisés. Tout d'abord, une pâte de farine de blé a été préchauffée pendant 10 min à trois températures différentes, soit 60, 70 et 80°C. Les échantillons de pâte ont ensuite été frits dans l'huile de canola à $180 \pm 2^\circ\text{C}$. La pâte préchauffée à 60°C a démontré une teneur plus élevée en humidité ainsi qu'une porosité et une teneur en matière grasse inférieure à la pâte non-préchauffée et à celles préchauffées à 70 et 80°C. Ainsi, une température de préchauffage de 60°C a été choisie pour étudier l'effet de différentes formulations de pâte (soit 100 g de farine de blé pour 0 g de farine de riz, 75 g pour 25 g, 50g pour 50 g, 25g pour 75 g, et 0 g pour 100 g) sur l'humidité et la teneur en matière grasse et la porosité. Les pâte formulées avec une quantité plus

élevée de la farine de blé ont montré une plus grande teneur en humidité ainsi qu'une porosité et teneur en matière grasse inférieure aux pâtes formulées avec une quantité plus élevée de farine de riz, ce qui suggère que la farine de blé est un meilleur ingrédient comparativement à la farine de riz pour réduire la teneur en matières grasses des aliments frits.

L'effet de la formulation de la pâte et du temps de pré-séchage sur la fraction de matière grasse dans la pâte frite a été étudié. Différents modèles mathématiques pour prédire la diffusion de l'humidité et le transfert de l'huile durant la friture ont été évalués. Il a été trouvé que la pâte contenant 100 g de farine de blé contenait plus de graisse de surface et un taux total de matière grasse plus élevé, tandis que la teneur en matières grasses pénétrées était inférieure à celle des autres pâtes. La pâte sans pré-séchage a démontré des teneurs en matières grasses de surface, pénétrées et totales plus élevées que les autres pâtes. La diffusivité de l'humidité se situait entre 5.47×10^{-6} et $7.38 \times 10^{-6} \text{ m}^2/\text{min}$. Le modèle de Page a été identifié en tant que modèle avec le meilleur ajustement pour évaluer les constantes de vitesse de transfert de l'huile.

L'influence de la formulation de la pâte et du temps de pré-séchage sur les différentes fractions des espaces entre les particules de pâte frite occupées par l'air, l'humidité ou la graisse a été évaluée. Une teneur élevée en farine de riz dans la pâte a augmenté de manière significative la fraction des espaces entre les particules occupées par de l'air (SOA) et diminué la fraction des espaces occupés par l'humidité (SOM) et la graisse (SOF). Un plus long temps de pré-séchage a augmenté la fraction des espaces occupés par l'air, et diminué la fraction des espaces occupés par l'humidité et la graisse. Le SOA, SOM et SOF des pâtes variaient de 2.20 à 46.03%, de 2.55 à 47.07% et de 0.35 à 11.11%, respectivement.

Les caractéristiques de la surface de la pâte frite préparée à partir de différentes combinaisons de farines de blé et de riz ont été étudiées. La microscopie électronique à balayage à pression variable (VP-SEM) a été utilisée pour étudier la microstructure de surface des pâtes. Des paramètres tels que le nombre total, la surface moyenne, le ratio et la rondeur des ruptures générées sur la surface du revêtement de la pâte après la friture ont été utilisés pour caractériser la microstructure de la pâte frites. Les pâtes formulées uniquement de farine de blé avaient le plus grand nombre de ruptures tandis que celles formulées uniquement de farine de riz avaient le plus petit nombre de ruptures. La rondeur des ruptures variait de 0,19 à 0,35. Plus la quantité de farine de riz utilisée dans la formulation de la pâte était élevée, plus l'aire des trous et des fissures générées sur la surface était élevée. La teneur en graisse était en corrélation directe avec le ratio des trous et des fissures observées sur la surface ($|r| = 0.72$), tandis que la perte d'humidité était en corrélation avec le ratio ($|r| = 0.67$) et la surface moyenne ($|r| = 0.77$).

La dimension fractale (FD) et la lacunarité ont été utilisées pour étudier la structure et les irrégularités de la surface des pâtes avec différentes formulations pendant la friture. En utilisant la microscopie électronique à balayage, les images des surfaces ont été prises et analysées par FracLac plug-in appliqué dans le logiciel ImageJ. La FD se situait entre 2.79 et 2.82. L'utilisation d'une la plus grande quantité de farine de blé dans la préparation de la pâte à frire a donné lieu à des valeurs de FD plus élevées qu'avec une quantité plus élevée de farine de riz. La FD a augmenté significativement ($P < 0,05$) au cours de la friture. Il y avait une forte corrélation positive entre la FD et l'absorption de graisse pour toutes les formulations de pâte à frire ($|r| =$ de 0.91 à 0.99). La lacunarité, en tant que mesure de degré d'hétérogénéité des ruptures de la pâte, se situait entre 1.19 et 1.25. Les résultats de la lacunarité ont démontré que la taille et la forme

des ruptures générés sur la surface de pâtes durant la friture, à savoir des fissures et des trous, était à peu près uniforme.

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NOMENCLATURE

ESEM	Environmental Scanning Electron Microscopy
VP-SEM	Variable Pressure Scanning Electron Microscopy
EM	Electron Microscopy
SEM	Scanning Electron Microscopy
TEM	Transmission Electron Microscopy
LM	Light Microscopy
CLSM	Confocal Laser Scanning Microscopy
CT	Computed Tomography
MR	Magnetic Resonance
AFM	Atomic Force Microscopy
STM	Scanning Tunneling Microscopy
SCM	Scanning Capacitance Microscope
MFM	Magnetic Force Microscopy
EFM	Electrostatic Force microscopy
SThM	Scanning Thermal Microscopy
PFM	Pulsed Force Microscopy
FMM	Force Modulation Microscopy
IUPAC	International Union of Pure and Applied Chemistry
V_{SP}	Sample Volume
V_C	Volume of Chamber Containing Sample

V_R	Volume of Second Empty Chamber
P_1	Pressure before Gas Expansion into the Second Chamber
P_2	Pressure after Gas Expansion into the Second Chamber
V_b	Bulk Volume
V_t	True Volume
V_p	Pore Volume
ρ_b	Bulk Density
ρ_s	Solid Density
ρ_a	Apparent Density
ϵ	Porosity
Z_i	Surface Height
\overline{Z}	Average Height Surface Profile
N	Total Number Data Points
ζ	Sampling Length
S_q	Root Mean Square Deviation Surface
S_{sk}	Surface Skewness
S_{ku}	Surface Kurtosis
S_{ds}	Density of the Summits of Surface
S_{tr}	Texture Aspect Ratio of Surface
S_{td}	Texture Direction of Surface
$S_{\Delta q}$	Root Mean Square Slope of Surface
S_{sc}	Arithmetic Mean Summit of Surface
S_{dr}	Developed Surface Area Ratio

$L(\varepsilon)$	Unit Measurement
F	Constant
FD	Fractal Dimension
FBM	Fractal Brownian Motion
DBC	Differential Box Counting Method
FDM	Frequency Domain
E	Average Absolute Intensity Difference
x	Image Coordinate
x'	Image Coordinate
y	Image Coordinate
y'	Image Coordinate
$A(\varepsilon)$	Surface Area
FFT	Fourier Transforms
$D_h(f)$	Average Horizontal Power Spectrum
$D_v(f)$	Average Vertical Power Spectrum
H_h	Horizontal Hurst Coefficients
H_v	Vertical Hurst Coefficients
CMC	Carboxymethyl Cellulose
W	Wheat
R	Rice
MRT	Multiple Range Test
ANOVA	Analysis of variance
MD	Minutes Drying

FC	Fat Content
SF	Surface Fat
PF	Penetrated Fat
TF	Total Fat
t	Time
MC	Moisture Content
MR	Moisture Content Ratio
M_0	Initial Moisture Content
M	Moisture Content at Time t
M_e	Moisture Content of Final Product
Deff	Effective Moisture Diffusion Coefficient
FR	Fat Content Ratio
F_0	Initial Fat Content
F	Penetrated Fat Content at Time t
F_e	Equilibrium Penetrated Fat Content
k	Constant Rate
db	Drying Base
RSME	Root Square Mean Error
R^2	Coefficient of Determination
SD	Standard Deviation
SOA	Space Occupied by Air
SOM	Space Occupied by Moisture

SOF	Space Occupied by Fat
WG	Wheat Gluten
SPI	Soy Protein Isolate
WBC	Water binding capacity
MA	Mean Area
BR	Biggest Rupture
Ra	Ratio
Ro	Roundness
NR	Number of Ruptures
λ	Lacunarity

CHAPTER 1

GENERAL INTRODUCTION

1.1. Background

Deep fat frying is a simultaneous heat and mass transfer process that causes physicochemical and microstructural changes inside the food during the process (Barutcu, Sahin et al. 2009). Although deep fat fried foods are widely consumed all over the world, awareness of the health impact of consuming high fat diet is becoming a concern for consumers and processors all around the world (Adedeji and Ngadi 2009). Studies have shown that oil uptake is directly related to the amount of water evaporation during frying. The more water is removed, the greater the amount of oil uptake is observed in fried products (Martelli, Carvalho et al. 2008, Suárez, Campañone et al. 2008, Achir, Vitrac et al. 2010, Dragich and Krochta 2010). The fast evaporation of moisture from the food during frying damages the surface and creates a porous structure, which plays a critical role in oil penetration into the batter crust (Barutcu, Sahin et al. 2009).

A number of potential methods for reduction of fat uptake in deep fat frying have been studied (Mellema 2003, Dana and Saguy 2006). These basically include 1) modification of frying techniques, 2) modification of frying medium, and 3) batter and breading coatings. Modification of frying techniques and mediums are promising methods for decreasing oil content, and they have been largely applied in the past few years. However, batter coating, as the technique of preparing a barrier against mass transfer, mainly water evaporation and oil absorption, still needs more attention.

Appropriate post-frying techniques such as removing the extra fat from the surface and proper frying temperature and time have been successfully applied in reducing the amount of fat content in final fried products.

The type of the oil that is used for frying has also been found to be important in reducing fat content. The use of triglycerides with polyunsaturated fatty acids is desired from a health point of view.

Batter and breading are complex systems prepared by mixing different ingredients including flour, starch, hydrocolloids, salt, bread crumbs, seasoning and water. The interaction of the ingredients determines the final quality of the products. Applying heat during frying makes a network (Adedeji, Liu et al. 2011), and creates the crust of fried products. This crust provides the brittle texture, and forms a barrier to prevent moisture loss and consequently oil uptake (Nasiri, Mohebbi et al. 2012).

Wheat flour, which is largely available in most regions, is the most common flour used in batter systems (Xue and Ngadi 2007). However to decrease oil uptake, interest has shifted to using substitutes such as corn, rice, soy, and other types of flour. Rice flour is often used in batter systems since it is a healthier alternative, and contains fewer calories (Barutcu, Sahin et al. 2009). Rice flour is also an alternative for gluten intolerance individuals. It has a relatively low amount of proteins, especially gluten, and shows a weak elasto-plastic structure compared to wheat proteins. As a result rice flour makes thin slurries with poor adhesion properties, which exhibit different textural and microstructural properties to batters prepared with wheat flour.

Porosity, pore size distribution, surface attributes, and other textural characteristics of fried foods influence the amount of oil uptake during frying (Moreno, Brown et al. 2010, Adedeji and

Ngadi 2011, Rahimi and Ngadi 2014). Porous structure has been defined as porosity, pore size distribution, specific surface area, specific conductivity and capillary breakthrough pressure (Kassama and Ngadi 2005). Mercury porosimetry and helium pycnometry are the two most common methods that provide some useful information about density, porosity, and pore size distribution of food materials (Kassama, Ngadi et al. 2003, Kassama and Ngadi 2005, Adedeji and Ngadi 2011). Pores occur in a variety of foods and significantly influence their quality. Mass diffusion coefficient, thermal diffusivity, and thermal conductivity of foods are also directly affected by the existence of pores (Ježek, Brnčić et al. 2009).

Food surface characteristics, specifically the geometrical irregularity, which is defined as roughness, have a pronounced importance in oil uptake phenomenon, and also sensorial and visual properties of fried foods (Moreno, Brown et al. 2010). Study on surface topography of chicken nuggets has demonstrated that the impact of this characteristic on the oil transport phenomena is dependent on several parameters including both chemical and physical properties of the products. The surface attributes of foods directly influence the amount of oil absorption during frying (Thanatuksorn, Pradistsuwana et al. 2005, Moreno, Brown et al. 2010). Most fried food manufacturers consider the surface topography as a determinant factor that influences the final oil content of fried foods and consequently the desirability of their products. Briefly, microstructural properties, such as porosity and surface characteristics, are very important attributes of foods that can considerably influence their quality during and after processing. Therefore, there is the need to study how frying may influence them.

The main advances in food engineering and technology of the 21st century come from investigations at the microscopic level. Imaging technologies have shown valuable applications in all major areas, particularly as the result of recent developments in camera technology and the

processing power of computer hardware (Chen, Zhang et al. 2013). A broad number of instruments are available to image food products for microstructural studies. The instruments are classified based on the scale of imaging. The range of the scale changes from millimeter to nanometer depending on the purpose of the imaging. Light microscopy, LM (Llorca, Hernando et al. 2007), confocal laser scanning microscopy, CLSM (Achir, Vitrac et al. 2010, Adedeji, Liu et al. 2011), transmission electron microscopy, TEM (Kaur, Maudens et al. 2014), scanning electron microscopy, SEM (Kalogianni and Papastergiadis 2014), and X-ray micro-CT (Adedeji and Ngadi 2009) are some examples of application of instruments used in imaging in food science and engineering. SEM and TEM techniques are capable of imaging at nanometer scales. X-ray and SEM present images from nano to micrometer scales. LM and CLSM are able to obtain structural information ranging from micrometers to very close to millimeter scales.

1.2. Hypothesis

Formation of rough surfaces and pores during frying is among the important determinants of physicochemical characteristics of fried foods. The hypothesis of this study is that coating formulation and pre-treatments, such as pre-heating and pre-drying, play critical roles in absorption of oil during and after frying, the formation of rough surfaces, the generation of the pores and, the manner in which oil is distributed across the surface of the fried foods as well in the surface topography of the fried products.

1.3. General objective

The objective of this study is to understand microstructural changes in batter systems during frying as influenced by components interactions and pre-treatments. Different formulations of batter, pre-drying, and pre-heating, scanning electron microscopy (SEM), and surface characteristics evaluation will be applied for studying these microstructural developments. The

results would be valuable for better designing of deep fat frying process for a wide range of food materials.

1.4. Specific objectives

Different batter formulations have been chosen for the study. The following specific objectives are set for the proposed project.

1. To study the effect of batter formulation and pre-heating temperatures on porosity and oil uptake of batter coating during deep fat frying.
2. To study the influence of batter formulation and pre-drying time on oil distribution fractions in fried batter.
3. To determine the influence of batter formulation and pre-drying time on fractions of inter-particle spaces in fried batter coating.
4. To characterize surface roughness of fried batter coatings via fractal dimensional analysis using SEM images and finding the relationship between the surface roughness, oil absorption, and batter formulation.
5. To study the effect of batter formulation on surface ruptures of fried batters using SEM imaging.

CHAPTER 2

LITERATURE REVIEW

2.1. Deep-fat frying

Deep-fat frying is one of the oldest processes of food preparation, which dates back as early as 1600 BC (Servet Gulum and Serpil 2008), and originates around the Mediterranean area (Moreira, Castell-Perez et al. 1999). The unique flavor-texture combination of fried foods has made these products the desired choice for consumers for decades. There are various fried products consumed globally and locally, which among them French fries is consumed throughout the world (Moreira, Castell-Perez et al. 1999). Some other fried foods are potato chips, doughnuts, extruded snacks, fried chicken products, and fish sticks.

Deep-fat frying is a complex unit operation involving simultaneous heat and mass transfer, which causes water removal and oil absorption through the surface of foods (Bouchon 2009). The temperature of oil in deep frying is above the boiling point of water, usually around 150 to 180°C (Farinu and Baik 2005). This high temperature forms water vapor during frying, which is transferred through the surface of the product due to pressure and concentration gradients (Servet Gulum and Serpil 2008). This water evaporation results in formation of a porous network known as crust. The porous structure of crust affects oil absorption by providing open channels and ways for entering oil (Mellema 2003). It was suggested that the oil absorption is highly dependent on the amount of moisture loss, frying temperature and time, surface treatments, pre-frying treatments, porosity, initial interfacial tension, oil quality, and the product characteristics being fried (Moreira, Castell-Perez et al. 1999, Lalam, Sandhu et al. 2013). Most food products

exhibit a heterogeneous and complex porous structure which makes the mechanism of oil uptake very complicated and not easy to understand.

2.2. Mechanisms of oil absorption

To reduce fat content of fried foods, it is essential to understand oil uptake mechanisms involved in the process of deep-fat frying, so that oil penetration into the structure of food can be effectively minimized. Generally, three major possible mechanisms of oil uptake have been suggested in various studies. These mechanisms are namely water replacement, cooling-phase effect and surface-active agents.

2.2.1. Water replacement or capillary mechanism

When food is immersed in hot oil and exposed to frying temperature, quick water evaporation creates a dried outer surface and forms a crust (Mellema 2003). Water vapor generates a positive pressure that develops cracks, defects, open capillaries, and channels in the structure of the fried products. As the process progresses, steam escapes through these cracks and channels, oil adheres to the food and enters the voids, product imperfections and crevices left by the changes in the structure (Saguy and Dana 2003). The existence of large voids and cracks limits the inner resistance due to positive vapor pressure (Dana and Saguy 2006), which results in oil uptake through these channels. This mechanism can explain the direct relationship reported between water loss and oil uptake. Oil that enters the voids may help maintain structural integrity by preventing shrinkage and collapse. Water replacement mechanism is basically related to large voids, cracks and crevices that normally characterize reformed and battered, as well as many starchy products. It should be noted that in non-reformed products, most of the capillaries that had been initially filled with water cannot be filled with oil as long as water continues to evaporate. If the capillary is closed on one side, oil cannot penetrate, because the inner steam

pressure prevents it. The oil is sucked inside the product only after its removal from the hot oil. The fact that oil penetration is limited to the immediate crust layer of non-reformed products indicates that capillaries are not playing a major role in oil uptake mechanism (Dana and Saguy 2006).

2.2.2. Cooling phase effect

After completing the frying process and removing food from the hot oil medium, the product starts to cool, water vapor condenses and subsequently the internal pressure drops. Oil, which is adhered to the food surface, is sucked in due to the consequent ‘vacuum effect’ (Moreira, Castell-Perez et al. 1999). It has been proposed that oil uptake is a surface phenomenon involving equilibrium between adhesion and drainage of oil during post frying process known as cooling phase (Dana and Saguy 2006). Surface characteristics of foods and oil properties are the important factors controlling oil uptake during the cooling phase. For a product such as tortilla chips, only 20% of the oil is absorbed during the frying phase, while 80% remains at the surface of the product (Mellema 2003). About 64% of the surface oil is later absorbed into the interior part of the food during cooling phase. The condensation mechanism or cooling phase effect is probably the most important factor affecting oil absorption in short frying times, and large food samples with high moisture content. If longer frying times are used, the fat uptake is greater. Surface water evaporation stops when the crust is dry. Under these situations, oil might be absorbed before the food is taken from the frying fat. This can for example be the case for small food pieces like thin potato crisps, for which the heating is more intense, and drying is completed earlier.

2.2.3. Surface-active agents

During frying, and in presence of oxygen, moisture, and high temperature, oil degrades due to hydrolytic reactions and generates fatty acids and polar compounds (Saguy and Dana 2003, Ahmad Tarmizi, Niranjana et al. 2013, Crosa, Skerl et al. 2014). Hydrolytic reactions involve the cleavage of bonds between glycerol and fatty acids. The high frying temperature accelerates hydrolysis and subsequently, the formation of diglycerides, monoglycerides, free fatty acids and glycerol (Blumenthal 1991, Choe and Min 2007). The interfacial tension between the oil and the food is decreased due to the presence of some of the degradation compounds, which act as wetting agents and are also considered as surface active agents. Surfactant formation increases the contact between the food and the frying oil, leading to more oil uptake. Surfactant agents also impact heat transfer at the oil–food interface and reduce the surface tension between the two immiscible materials. As contact time between the food and the frying oil increases, more heat is transferred from the frying oil to the food, leading to higher dehydration at the surface and water migration from the core to the exterior of the fried food. Thus higher surfactant concentrations produce oil-soaked products with an overcooked exterior and undercooked interior.

2.3. Batter systems

Batters are the common component of coating systems. They are flour-water mixtures, basically used to coat food materials before deep fat frying and to add value to a product by modifying its flavor, texture, volume and weight (Mohamed, Hamid et al. 1998). In general, there are two types of batters namely: interface/adhesion batter and puff/tempura batter (Kulp and Loewe 1990). Although such terminology varies based on the way the batters are used, these two categories underline the general concepts.

Interface/adhesion batter is usually used with breading or bread crumb to improve the appearance, flavor, granulation, and crispness of coated fried products (Mallikarjunan, Ngadi et al. 2004). In interface/adhesion batter, the breading part is adhered to the food surface. The uniformity and thickness of the coating layer is very critical in the acceptability of the final product (Kulp and Loewe 1990). Viscosity of the batter is the dominant factor which affects batter pickup. A more viscous batter yields a higher batter pick up of breading in comparison to a thin and more watery suspension (Mukprasirt, Herald et al. 2000). In preparation of viscous batters, high percentage of wheat or corn starch, especially chemically or thermally modified starches, is applied to improve its adhesive properties (Kulp and Loewe 1990).

In puff/tempura system, the batter can be served as the outside coating of foods without breading or bread crumbs; and therefore needs structural and visual qualities that are more complex than the interface batter. Tempura batter contains leavening agents to generate gas and puff the product (Kulp and Loewe 1990). It has a high viscosity and provides a thicker coating layer than the interface batter system. Chicken nuggets (Albert, Perez-Munuera et al. 2009, Labropoulos, Varzakas et al. 2013), squid rings (Llorca, Hernando et al. 2005), and fish balls (Kilincceker and Hepsag 2011) are some examples of typical foods which are coated with puff/tempura batters.

In both cases, the technological role of the batter is to provide a uniform layer to coat the product, remain on the surface before, after and during the final frying process (Sumnu and Sahin 2010).

2.4. Batter formulation

As mentioned earlier, batter is a liquid dough system, basically including water and flour (Fizman and Salvador 2003). However, the list of ingredient is fundamentally much longer than just being flour and water. The main flour type, which is typically used for preparing batter systems, is wheat flour, which determines the batter fundamental characteristics. Gluten, the major protein of wheat flour, provides elastic properties in the batter (Champenois, Rao et al. 1998). This property makes batter to expand during frying and creates an appropriate spongy coating, which accelerates the transfer of oil and moisture during frying.

Some limitations in using wheat flour, such as being not available in all areas, price, and allergenic issues due to its gluten content forced manufacturer to think about other alternatives (Fizman and Salvador 2003). Some ingredients have also been used as substitutes to wheat flour to decrease oil absorption during frying. It was reported that substitution of a part of wheat flour in batter formulation with corn flour decreased the final oil content (Llorca, Hernando et al. 2003). Applying pre-gelatinized starch in formulation of batter significantly increased batter pick up and consequently decreased fat uptake during frying (Altunakar, Sahin et al. 2004). Wootton and Bamunuarachchi (1978) compared the chemical properties of native and pre-gelatinized wheat starches and found that moisture content of native starch is higher than pre-gelatinized one, 13.60 and 11.90%, respectively. However, water binding capacity of pre-gelatinized wheat starch was higher than that of native wheat starch, 33 and 42%, respectively. Higher water binding capacity in pre-gelatinized starch caused higher batter pick up. Altunakar et al. (2004) reported that pre-gelatinized tapioca starch was an effectual starch in reducing oil uptake. Batter pick up significantly increased when soy flour was used as an ingredient in preparing batter (Firdevs Dogan, Sahin et al. 2005). Higher protein content in soy flour compared to rice, corn, and wheat

flours results in higher water binding capacity (Firdevs Dogan, Sahin et al. 2005), and therefore less movement of particles is observed in dough prepared from this flour. Batter prepared from soy flour is then more viscous and shows higher batter pick up compared to batters prepared from rice, corn, and wheat flours. The results obtained in a study (Nasiri, Mohebbi et al. 2012) on the effects of soy and corn flour addition on quality of deep fat fried shrimp nuggets showed that batter formulation had a considerable effect on moisture and oil absorption and coating pickup. Batter containing soy flour provided the highest apparent viscosity, the highest moisture holding capacity, and lowest free moisture content during deep fat frying, while addition of corn flour decreased the apparent viscosity and moisture retention and increased the fat intake among all the formulations.

Rice flour has been introduced as a promising alternative to wheat flour in batter coating systems (Mukprasirt, Herald et al. 2000). Rice-flour-based batters can improve the value of batter systems because of being less allergenic than other grains such as wheat with gluten intolerance problem. It has relatively low amount of proteins, especially gluten, and shows a weak elasto-plastic structure compared to wheat proteins. As a result rice flour makes thin slurries with poor adhesion properties. Shih et al. (2004) and Firdevs Dogan et al. (2005) recommended rice flour as a substitute to reduce oil uptake. More recently, Adedeji and Ngadi (2011) reported contradictory findings that application of rice flour to batter system increased fat content. Therefore, questions have been raised about the functionality of rice flour in reducing fat uptake during frying of batter. Table 2-1 shows the details concerning the composition of rice and wheat flours (Adedeji and Ngadi 2011). As can be observed from the table, the protein content of wheat flour is approximately double the protein content of rice flour.

Table 2-1 Composition and particle size of wheat and rice flour.

	Wheat Flour	Rice Flour (g/g)
Protein (g/g)	0.133	0.072
Moisture Content (g/g)	0.116	0.110
Ash (g/g)	0.004	0.006
Fiber (g/g)	0.033	0.007
Crude Fat (g/g)	0.009	0.005
Particle Size (μm)	24	105

2.5. Pre-treatment

Appropriate pre-treatment of foods can significantly lower oil absorption during deep-fat frying. Some pretreatment techniques, such as pre-drying and pre-cooking, have been widely used to decrease oil content of fried products such as chicken nuggets, potato chips and French fries.

2.5.1. Pre-drying

Initial moisture content and moisture loss during deep fat frying determine the physico-chemical and sensory properties of fried products. Pre-drying reduces the initial moisture content of raw food materials. Longer time of pre-drying signifies less free moisture to evaporate during frying (Gupta, Shivhare et al. 2000). Drying of foods before frying has a substantial negative influence on the equilibrium oil content of fried foods. The initial moisture content of foods has been proposed as the most clearly known factor which impacts the oil gain during frying (Krokida, Oreopoulou et al. 2001). Gupta et al (2000) showed that pre-dried potatoes absorbed significantly less fat compared to non-pre-dried samples. The possible reasons for decreasing fat uptake during frying because of pre-drying can be the decrease of inner vacuum pressure, textural changes and formation of a compact material matrix (Debnath, Bhat et al. 2003).

When a food material fries, its moisture vaporizes, and an inner pressure is generated. After taking out the fried food from hot oil and during the cooling period, the water vapor condenses and generates a vacuum pressure inside the food, e. i. the pressure gradient switches. This inverse pressure gradient pulls oil droplets into the fried food (Moyano and Pedreschi 2006). By decreasing the initial moisture content by pre-drying, less moisture is available to evaporate during frying; and therefore, lower inner pressure is generated. As a result, the vacuum pressure in the cooling phase is lower and less oil is absorbed. Study of initial moisture content on moisture loss and fat uptake during deep fat frying of a model system showed that when the initial moisture content was low the moisture loss and oil gain during frying was considerably less than when high initial moisture content models were fried (Indira 1996).

Textural properties of dried foods are the result of many physico-chemical and structural changes that occur during drying. Structural properties, such as true and apparent densities, porosity, and specific volume, strongly depend on pre-drying time (Krokida, Oreopoulou et al. 2001). These authors reported that the initial true and apparent density and total porosity of French fries was increased by pre-drying, whereas specific volume decreased.

A number of techniques have been used to dry food materials before the final frying step. Immersion of potato strips in a sugar solution showed to be a method of lowering oil content (Mai Tran, Chen et al. 2007). Bunger, Moyano et al (2003) soaked potato strips in three different NaCl solutions prior to frying. NaCl soaking considerably reduced oil uptake, while there was no significant effect on color and sensory acceptability. Oven, microwave, and freeze drying methods were applied to reduce the initial moisture content of onion slices (Hansen 1998). Drying technique did not show any significant influence on fat content, whereas higher dry matter content decreased the amount of fat absorption.

2.5.2. Pre-cooking

Some pre-cooking procedures, such as microwave-heating and blanching have been proposed to reduce the amount of absorbed oil in fried products. Microwave heating has shown large applications in food processing sectors over a period of several decades. These applications include drying, pasteurization, sterilization, thawing, tempering, baking of food materials, and many other processes (Chandrasekaran, Ramanathan et al. 2013). Its popularity is due to its achieving high heating rates, shorter cooking times, more uniform heating, yielding of unique microstructures and properties, energy saving, and less change in flavor and nutritional value of foods compared to conventional heating processes (Amiryousefi, Mohebbi et al. 2012, Chandrasekaran, Ramanathan et al. 2013). Microwave pre-cooking has been reported to reduce fat absorption in breading coating during frying (Adedeji, Ngadi et al. 2009). It was observed that this technique significantly reduced the moisture content of breading coating before frying, and therefore less moisture was available to evaporate compared to uncooked samples.

Blanching prior to frying has been used in some potato processing industries to improve the color and texture of potato chips. After blanching, hot air is blown on the surface to remove extra water and partially dry the potato slices. Moyano and Pedreschi assumed that the blanching step can gelatinize surface starch and reduce oil uptake (2006). Based on this assumption, they studied the effect of blanching alone and blanching combined with air drying on oil content of fried potato slices. They unexpectedly observed that blanching without air drying showed higher amount of fat content than non-blanching fried potatoes, whereas pre-drying of blanched potatoes reduced oil uptake. It was concluded that high temperature quick blanching may increase the porosity of food materials and consequently resulted in higher amount of fat absorption (Alvarez, Morillo et al. 2000, Moyano and Pedreschi 2006). In contrast, using low temperatures and longer

times for blanching was shown to activate pectinesterase enzyme (PME) in potatoes, which decreased porosity, and resulted in reduction of fat content (Aguilar, Anzaldúa-Morales et al. 1997).

2.6. Microstructure studies

Quality of food products is directly related to their mechanical (textural) and sensorial (shape, size, appearance, color) properties (Aguilera, Stanley et al. 2000). These properties are considerably influenced by the structural arrangement of the food elements at all molecular, microscopic and macroscopic levels (Falcone, Baiano et al. 2006). Majority of these elements are below 100 μm range (Aguilera 2005). Microstructural characteristics, such as porosity, pore development, pore size distribution, number of the pores, cell walls, and surface morphology may influence texture, crust development, taste, consumer acceptability, fat distribution and many other specifications of fried products (Bouchon and Aguilera 2001, Pedreschi and Aguilera 2002, Adedeji, Ngadi et al. 2009, Adedeji and Ngadi 2010, Adedeji and Ngadi 2011).

2.6.1. Porosity of batter systems

Finding the relation between microstructure and mass (oil and moisture) transfer behavior of food materials is of great interest for many food scientists for predicting and modeling frying process. The porous structure of food products is an important factor that affects heat and mass transfer (Ngadi, Kassama et al. 2001). Some authors have explained that pore development is the result of the transport mechanisms of free water, bound water and water vapors during frying (Rahman 2001, Kassama and Ngadi 2004).

The most common parameter, which has been used to characterize pores, is porosity. Porosity is defined as the volume fraction of the pores or the ratio of empty spaces within a porous solid media to the total volume of the solid (Ngadi, Kassama et al. 2001, Rahman 2001).

Some other parameters such as number of pores, pore size distribution, pore shape, number of face or wall of pores and wall thickness of pores have been used for pore characterization (Rahman 2001, Adedeji and Ngadi 2011). Cumulative surface area, cumulative pore volume, percent pore volume distribution, and pore diameter have also been applied to explain porosity of different food materials (Ngadi, Kassama et al. 2001, Kassama, Ngadi et al. 2003, Kassama and Ngadi 2005).

International Union of Pure and Applied Chemistry (IUPAC) classified pores based on their sizes with the following groups (Everett 1972): (i) pores with widths exceeding about 0.05 μm or 50 nm (500 Å) are called macro-pores; (ii) pores with widths not exceeding about 2.0 nm (20 Å) are called micro-pores; and (iii) pores of intermediate size are called meso-pores. Kassama and Ngadi (2004) introduced three possible types of pores namely interconnected pores (accessible from many directions), isolated pores (inaccessible) and non-connected pores (accessible from one direction). Interconnected pores show a more important role in transport of fluid through the porous medium than the non-connected pores, while isolated pores have limited influence on transport phenomena. Interconnected pores and non-connected pores are the reservoirs of moisture and oil in fried products. Distribution of oil, moisture, and even air through these spaces changes during the frying process. The measurement of inter-particle spaces occupied by oil, moisture, or air is therefore necessary for studying the mechanism of oil uptake and moisture loss during frying.

There are several techniques that have been used to study pore characteristics of food materials. These include gas pycnometry, microscopy, X-ray computed tomography, magnetic resonance imaging, and mercury porosimetry (Adedeji and Ngadi 2010). Pycnometric methods have been used to measure true and apparent densities and porosity of foods using nitrogen and

helium gases as displacing fluids (Moreau and Rosenberg 1998). Variation in the rate of gas penetration through the porous media is related to its porosity. This approach applies the ideal gas law to determine the true volume of the food materials. In this technique, the closed pores are not determined and the volume of the closed pores is involved with the true volume of the solid (Datta, Sahin et al. 2007). A pycnometer usually contains two chambers. The porous material is placed into the sample chamber, and a pressure of about 137.9 kP (20 psig) is applied to that chamber. Then, a valve which attached the second chamber of defined volume to the first is released, and the gas flows into the second chamber. In this method, the change in pressure in the sample chamber, as the valve is released, is a key to measure the volume of the porous sample.

The volume of the porous sample is calculated as following (Micromeritics. 1992):

$$V_{SP} = V_C + V_R / (1 - P_1 / P_2) \quad \text{Equation (2-1)}$$

where V_{SP} is the volume of the sample, V_C is the volume of the chamber containing the sample, V_R is the volume of the second empty chamber, P_1 and P_2 are the pressures before and after gas expansion into the second chamber.

Apparent density is then calculated by dividing the mass of the sample by V_{SP} of the food (excluding open pores but including closed pores). Total volumetric porosity is obtained by dividing the difference between bulk and true volumes to bulk volume as follows:

$$\text{Porosity } (\varepsilon) = (V_b - V_t) / V_b \quad \text{Equation (2-2)}$$

where V_b and V_t are the bulk and true volumes, respectively. It can also be determined as a ratio of the volume of reachable pores inside the sample and the total volume of the food material that may or may not include open pores.

$$\varepsilon = V_p / V_{sp} = 1 - (\rho_b / \rho_s) \quad \text{Equation (2-3)}$$

$$\varepsilon = 1 - (\rho_b/\rho_a) \quad \text{Equation (2-4)}$$

where V_p , ρ_b , ρ_s , and ρ_a are the pore volume, bulk density, solid density and apparent density, respectively.

Helium pycnometry technique was used to study the porosity of batter coatings with different wheat to rice flour combinations during frying (Kassama and Ngadi 2005, Adedeji and Ngadi 2011). Porosity of batters significantly increased for the first minutes of frying, and beyond that it relatively remained constant. Heat and mass transfers during frying cause serious changes in structural configuration that may increase the porosity for a period of time during frying till equilibrium is reached when there is limited moisture left in the food (Adedeji and Ngadi 2011). These authors reported an increase in porosity of fried batters when higher amount of rice flour was used in batter preparation.

2.6.2. Food surface characterization

Surface topography is an essential physical attribute of solid foods impacting not only their sensorial and optical feature, but also their behavior during processing and storage (Quevedo, Carlos et al. 2002). Different operations on food products during their process cause and create new surfaces with new characteristics. To investigate the surface of food materials, it must be precisely “scanned” and evaluated in terms of measurable parameters such as roughness (Sheen, Bao et al. 2008). Roughness is entirely scale sensitive. A surface that regularly looks smooth to the naked eye, may be rough using advanced microscopic techniques and high magnification.

Surface geometry is by nature a three-dimensional feature. In theory, any evaluation of two-dimensional profiles or sections cannot give a complete description of the real surface topography features. However, in practice, two dimensional measurements are still generally

acceptable on the assumption that the surface is isotropic, that is, the surface has the same topography features across the X–Y plane (Chen, Moschakis et al. 2006).

In a digital image of a surface, data is stored as an array of pixels with different intensity or grey scales (Quevedo, Carlos et al. 2002). Pixels are basic components of images that contain brightness value and locations in the coordinates (Zheng, Sun et al. 2006). Brightness explains the color feature of an image, whereas the coordinates describe its geometry such as size. Size features are usually obtained using measurements of area, perimeter, length, and width (Zheng, Sun et al. 2006). Texture is an important image feature that corresponds to both brightness value and pixel location. Texture is actually the local deviation of brightness from one pixel to another or within a small area. The texture of images or texture feature is a useful method applied for pattern identification to characterize the arrangement of basic constituent of a material in a surface (Quevedo *et al.* 2002).

Image features have been effectively used in a large variety of foods such as fruits, vegetables, meat, processed foods, and fish for characterizing and segmentation of surface properties (Bennedsen, Peterson et al. 2005), studying the distribution of food components (Pedreschi and Aguilera 2002, Adedeji, Liu et al. 2011), and evaluation of sensory characteristics (Cernadas, Carrión et al. 2005).

An example of these image features is surface irregularities, such as surface geometry or roughness (Moreno, Brown et al. 2010), and surface ruptures, in fried foods (Nagao, Hatae et al. 1997). Surface irregularities may be characterized in qualitative or quantitative approaches (Pedreschi, Aguilera et al. 2000). It is sometimes studied qualitatively, as images or micrographs, showing surface features as visual contrast. Quantitative description of the surface roughness of

foods has been achieved either using common statistical parameters, such as the roughness factor, or by the application of the concept of fractal dimension (Quevedo and Aguilera 2004). Statistical parameters can be represented quantitatively as relative heights in an evaluation map. It means that z value of selected points on a surface is evaluated as a function of position (x,y). Numerical values are used to quantitatively describe surface characteristics. The numerical value would allow different surfaces to be suitably studied and assist the interpretation of surface texture in relation to physical and mechanical role of the surfaces (Chen 2007).

Table 2-2 lists the parameters that have been frequently used for surface roughness measurement (Stout and Blunt 1995). Practically, not all listed parameters are needed to study the surface roughness, and only a few of them is used. Root-mean-square roughness (RMS or R_q) and average roughness (R_a) are the most regularly applied parameters to explain topographical aspects. These parameters are defined as follows (Chen 2007):

$$R_q = \left[1/N \sum_{i=1}^N \left(Z_i - \bar{Z} \right)^2 \right]^{1/2} \quad \text{Equation (2-5)}$$

$$R_a = 1/N \sum_{i=1}^N \left| Z_i - \bar{Z} \right| \quad \text{Equation (2-6)}$$

where Z_i is the surface height at the data point i , \bar{Z} is the average height of the surface profile, and N is the total number of the data points (Figure 2-1).

$$\bar{Z} = 1/N \sum_{i=1}^N Z_i \quad \text{Equation (2-7)}$$

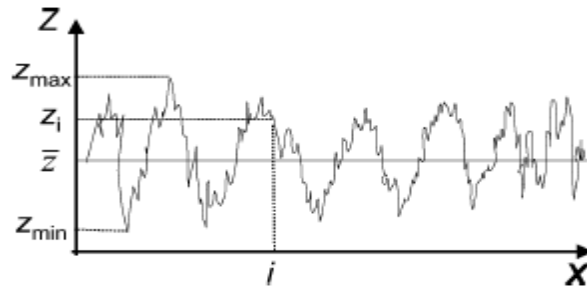


Figure 2-1 An example of a surface profile (Chen 2007).

The other valuable parameter is root-mean-square slope (Δq) of surface asperities:

$$\Delta q = \left[1/N \sum_{i=1}^N \left[(Z_{i+1} - Z_i)^2 / \zeta^2 \right] \right]^{1/2} \quad \text{Equation (2-8)}$$

where ζ is the sampling length (the space between two following points).

Table 2-2 Surface roughness parameters (Stout and Blunt 1995).

Amplitude parameters	
Sq	Root mean square deviation of the surface
Ssk	Skewness of the surface
Sku	Kurtosis of the surface
Spatial parameters	
Sds	Density of the summits of the surface
Str	Texture aspect ratio of the surface
Std	Texture direction of the surface
Hybrid parameters	
SΔq	Root mean square slope of the surface
Ssc	Arithmetic mean summit of the surface
Sdr	Developed surface area ratio

Another very important parameter for topographical measurements of various materials is fractal dimension, D (Chen 2007), which is used in several applications, such as, measurement of irregularities in an image, texture segmentation, surface roughness estimation and many other functions (Biswas, Ghose et al. 1998). It was for the first time used for characterization of coastlines (Mandelbrot 1982). Fractal dimension suggests that the surface of an object, as well as its profiles, does not necessarily follow the regular law of Euclidean geometry (Mandelbrot 1982); i.e. the surface does not simply show an integer (1, 2, or 3) dimension, and it is described with a fractional number.

Like many other natural materials, foods have irregular shapes and conformations. It is not a simple task to describe the physical structure of highly tortuous foods such as agglomerated particulates (i.e. instant milk or coffee), flowering vegetables (i.e. broccoli and cauliflower), or porous products (i.e. bread, or puffed cereals and snack foods). These foods exhibit a complex geometry in which a large category of structural irregularities, including pores, protuberances, and replicating structures exist (Barrett and Peleg 1995). Fractal dimension is a tool that indicates the degree of derivation from smoothness and regularity. This technique provides the possibility to describe the intrinsic properties of surfaces with some fractal parameters (Stout 2000). It shows the relationship between the linear length of a pattern and the length of a measuring unit.

$$L(\varepsilon) = F\varepsilon^{1-D}$$

Equation (2-9)

or

$$\log L = (1-D)\log \varepsilon + \log F$$

Equation (2-10)

where $L(\varepsilon)$ is the unit measurement such as perimeter, and area, ε is the scale used, F is a constant, and FD is the fractal dimension. FD is measured by obtaining the value of $1-FD$, which is called the Hurst coefficient. This coefficient is calculated from the slope of least-square linear regression of the logarithmic plot of $L(\varepsilon)/F$ versus ε . Different measurements of unit $L(\varepsilon)$ result in various methods in obtaining fractal dimension; e.g. differential fractal Brownian motion (FBM) method, ε -blanket method, differential box counting (DBC) method, and frequency domain method (Biswas, Ghose et al. 1998, Quevedo, Carlos et al. 2002, Zheng, Sun et al. 2006).

Fractal Brownian motion (FBM) is based on the average absolute difference of pixel intensities. This method is an example of a statistical fractal that is described by Hurst coefficient (Gonzales-Barron and Butler 2008). However, as it is a non-stationary stochastic process, it is difficult to estimate this parameter directly from an observation of the FBM. Thus fractal dimension is estimated from the fractional Brownian noise, which is the difference of successive points in the FBM. The equation to calculate FBM is as follows (Zheng, Sun et al. 2006):

$$E = d^{3-FD} \quad \text{Equation (2-11)}$$

where E is the average absolute intensity difference determined from all possible pixel pairs in the distance d in four directions (horizontal, vertical, diagonal, and antidiagonal). E and d are calculated from the following equations (Gonzales-Barron and Butler 2008):

$$E = \frac{\sum_{x=0}^{M-1} \sum_{y=0}^{N-1} \sum_{u=0}^{M-1} \sum_{v=0}^{N-1} |I(u,v) - I(x,y)|}{P} \quad \text{Equation (2-12)}$$

$$d = \sqrt{(x - x')^2 + (y - y')^2} \quad \text{Equation (2-13)}$$

M and N are the dimensions of the region, and P is the number of pixel pairs in the region.

In ε -blanket method the fractal dimension of curves/surfaces is computed using the area/volume measured at different scales (Annadhasan 2012). In this technique, the image is

progressively covered by a “blanket”, the thickness of which at any point is proportional to the difference in height between that point and its preceding point (Barrett and Peleg 1995). Based on the pixel size (ε) used for the measurement, different estimates of the surface area ($A(\varepsilon)$) are obtained. The surface area $A(\varepsilon)$ is computed as the sum of the area of each pixel of size ε , plus the sum of the exposed area between each pixel and its neighboring pixels with different heights (Cabral and Rangayyan 2012). For an image of size $N \times N$ pixels, with each pixel having the same value and size $\varepsilon \times \varepsilon$ units of area, the surface area is equal to $(N \varepsilon)^2$. When adjacent pixels possess different values, more surface area of the blocks representing the pixels is exposed. The total surface area for the image is calculated by the following equation (Cabral and Rangayyan 2012):

$$A(\varepsilon) = \sum_{m=0}^{N-2} \sum_{n=0}^{N-2} \{ \varepsilon^2 + \varepsilon [|f\varepsilon(m, n) - f\varepsilon(m, n+1)| + |f\varepsilon(m, n) - f\varepsilon(m+1, n)|] \}$$

Equation (2-14)

where $f\varepsilon(m, n)$ is the 2D image presented as a function of the pixel size ε . A power-law relationship exists between $A(\varepsilon)$ and ε for images exhibiting fractal characteristics. The value of FD is calculated by

$$FD = 2 - \frac{\Delta \log[A(\varepsilon)]}{\Delta \log[\varepsilon]}$$

Equation (2-15)

The fractal model is appropriate if, over a range of pixel sizes, the regression between $\log[A(\varepsilon)]$ and $\log[\varepsilon]$ is linear.

Box counting method is the most frequently applied approach for computing fractal dimension (Cabral and Rangayyan 2012). Box counting method is based on the number of boxes, $N(\varepsilon)$, of size ε required to fill the entire curve. In this method the pattern or image space is partitioned into square boxes of equal sizes, and then the number of boxes covering the image is

counted (Medina, Skurtys et al. 2010). The process is repeated by partitioning the image space into smaller and smaller squares. The number of boxes, $N(\epsilon)$, varies when the size of ϵ changes. The smaller the size of ϵ , the greater the number of boxes, while the larger the size of ϵ (i.e. resolution), the smaller the number of boxes $N(\epsilon)$. Fractal dimension is calculated from the following equation (Medina, Skurtys et al. 2010):

$$FD = -\frac{\text{Log } N(\epsilon)}{\text{Log } (\epsilon)} \quad \text{Equation (2-16)}$$

In frequency domain method, fractal dimension is determined from the Fourier power spectrum of the image data (Quevedo, Carlos et al. 2002, Gonzales-Barron and Butler 2008). The fast Fourier transforms (FFT) are taken in the horizontal and vertical directions. The average horizontal $D_h(f)$ and vertical $D_v(f)$ power spectrum of the surface is a function of the frequency f and satisfies the following relationships:

$$D_h(f) \propto f^{2-\beta_h} \quad \text{Equation (2-17)}$$

$$D_v(f) \propto f^{2-\beta_v} \quad \text{Equation (2-18)}$$

where β_h and β_v are

$$\beta_h = 2H_h + 2 \quad \text{Equation (2-19)}$$

$$\beta_v = 2H_v + 2 \quad \text{Equation (2-20)}$$

where H_h and H_v are the horizontal and vertical Hurst coefficients, respectively. These coefficients are then computed from the slop of logarithmic plot of $D_h(f)$ versus f and $D_v(f)$ versus f , respectively. The fractal dimensions of the region in the horizontal and vertical directions, respectively, are (Quevedo, Carlos et al. 2002, Gonzales-Barron and Butler 2008):

$$FFT_h = 3 - H_h \quad \text{Equation (2-21)}$$

$$FFT_v = 3 - H_v \quad \text{Equation (2-22)}$$

The fractal dimension of the image is the average of FFT_h and FFT_v .

The concept of fractal dimension is broadly applied to study the three-dimension microstructure of food and engineering materials. Fractal function provides suitable information and model for explaining rough surfaces. Previous studies revealed that the surface attitudes of fried foods and especially the geometrical irregularity or roughness highly influence the oil uptake kinetic (Pedreschi, Aguilera et al. 2000, Quevedo, Carlos et al. 2002, Thanatuksorn, Pradistsuwana et al. 2005, Moreno, Brown et al. 2010). Using scanning laser microscopy, Moreno et al (2010) studied the relationship between surface roughness and oil uptake in fried formulated products. The surface topographic analysis showed that potato-flake-based products are considerably rougher than gluten-based products. It was also found that within each product category, there was a good agreement between oil uptake and surface roughness. Quevedo et al (2002) focused on fractal dimension to study the morphological changes in images acquired by microscopy in starch gelatinization during frying process and chocolate blooming during storage. Thanatuksorn et al. (2005) applied fractal dimension measurement to find the correlation between surface roughness and fat content of a wheat flour and water based model. All studies showed a strong correlation between fractal dimension (surface roughness) and fat absorption.

2.7. Image processing and analysis

Visualization is defined as any method for generating images, diagrams or animations to display a message (Miura 2013). The interest in quantifying the different structural characteristics of foods is growing up, and it includes capturing images of the structure and performing measurements on the images by image processing and analysis. Image-processing analysis mainly consists of five steps including image acquisition, pre-processing, segmentation, object measurement, and classification (Falcone, Baiano et al. 2006).

2.7.1. Image acquisition

Image acquisition means the capture of an image in a digital form and storing it as matrices of x columns by y rows containing thousands of pixels (Falcone, Baiano et al. 2006). The ability of human in visual perception and perceiving sufficiently well a three-dimensional impression might deceive us that interacting with the three dimensional world around us is a simple task, whereas it is not that simple (Jähne 2005). Generally, all imaging techniques project a three-dimensional space onto a two-dimensional image plane. Therefore, basically, image acquisition is regarded as a projection from 3-D into 2-D space. The loss of one coordinate constitutes a severe loss of information about the geometry of the observed scene. There are various imaging methods that can recover the depth coordinate which is lost by the projection of the object onto an image plane. These techniques may either retrieve only the depth of a surface in 3-D space or fully reconstruct the volumetric objects. The captured images might be simple macroscopic or microscopic light images, such as images obtained from confocal laser scanning microscopy, or might also be obtained from other advanced machines such as electron microscope (EM), atomic force microscope (AFM), magnetic resonance (MR) or computed tomography (CT) imaging techniques (Miura 2013). More details about imaging machines are described latter in this chapter.

2.7.2. Image pre-processing

The purpose of image pre-processing is the enhancement of the quality of an image to remove distortions and improve some image characteristics (Falcone, Baiano et al. 2006). These defects may originate from the specimen or the imaging procedure. For example, because of poor illumination conditions, the images might be very dark and of low contrast. Related techniques are applied to enhance the visibility or measurability of details in images. These techniques are

generally used either to improve the visibility of the important details in pictures for printing or to isolate the important details from the background to facilitate their measurement (Russ 2004). The quality of an image can be improved by using a more powerful light source or a better design of the illumination setup. However, if these modifications are not possible or the images are already captured and we have to use them, it is still possible to solve the problem. Image enhancement is actually the process of modifying digital images so that the results are more appropriate for display or further image analysis. Making the details of interest more visible is accomplished by making other component in the image less visible; therefore, it is very important to correctly decide about the details which will be extracted (Falcone, Baiano et al. 2006).

2.7.3. Image segmentation

Image segmentation is the preliminary and very important step of many video and computer vision applications. It is the process of dividing an image into clear regions sharing similar properties (Wang, Wang et al. 2011). In this step, an image is separated into features and background. The operation produces a binary image, where pixels belonging to the same group have similar intensity values (Falcone, Baiano et al. 2006). There are several ways to perform image segmentation. Pixel-based methods only analyze the gray values of the individual pixels. Region-based methods analyze homogenous regions by working on the gray values in larger areas. Finally, edge-based methods detect edges and then try to follow them till find discontinuity (Jähne 2005). Decision about using these techniques depends on the purpose of image analyzing.

2.7.4. Object measurement

The next step is measuring the features of interest for each of the discrete objects. Counting of the features in an image is a frequently performed task. It is normally a straightforward simple task requiring a computer program to recognize groups of pixels that share a side or corner with any of their neighbours (Russ 2004). Counting the objects of interest, measuring the size (area, perimeter, length, width), shape (circularity, eccentricity, compactness, extent), brightness and color, and texture (smoothness, coarseness, graininess) are the features that are generally measured (Falcone, Baiano et al. 2006).

Measuring the size of an object is strongly affected by shape, color and orientation of that object. Computer measurement can provide accurate numerical values about size of particles and also their size distribution. There are several parameters which might be considered for studying the size of objects such as diameter, maximum calliper dimension (which is distance between two points that are farthest a part), and diameter of the largest or smallest circle (Russ 2004).

2.7.5. Classification (Falcone, Baiano et al. 2006)

It allows assigning a new object to one of the individuated groups by comparing the measured attributes of the new object to those of recognized one. Numerous methods are explained in this technique including statistical, fuzzy, and neural network. The statistical classification applies models of classify objects. The fuzzy classification method categorizes objects into groups without defined boundaries so as to deliberate the level of similarity of the considered objects with respect to the others. The artificial neural network approaches emulate human intelligence with the power of statistics.

2.8. Instrumentations and equipment available for image acquisition

Scale of image acquisition and observation differs according to the purpose of study and the available instruments. To obtain images of food products, a large number of techniques such as light microscopy (LM), confocal laser scanning microscopy (CLSM), transmission electron microscopy (TEM), scanning electron microscopy (SEM), and other techniques have been used (Llorca, Hernando et al. 2007, Adedeji, Liu et al. 2011, Kalogianni and Papastergiadis 2014). Different application may require different illumination techniques. Figure 2-2 represents a general overview of the limits of spatial resolution that each technique can visualize. Scanning tunneling microscopy and transmission electron microscopy are the techniques capable of imaging to nanometer scales, with a vaster area of resolution for the former technique i.e. STM.

The X-ray imaging and scanning electron microscopies present images from nano to micrometer scales. The optical imaging and confocal laser scanning microscopies are able to obtain the structural information ranging from micrometers to very close to millimeter scales, and finally, some techniques such as nuclear magnetic resonance microscopy introduces the images with magnitude of millimeters. In the case of axial resolution (height), there is almost the same classification with the exception that CLSM technique allows to capture the heights with micrometer magnitudes, while the X-ray imaging works in the scale of millimeters (Clarke and Eberhardt 2002).

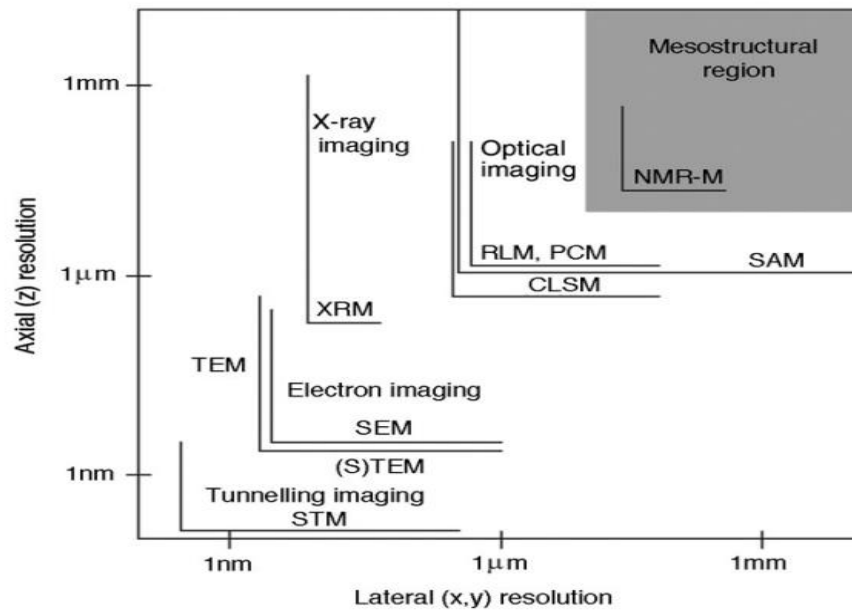


Figure 2-2 The current limits to spatial resolution for the different techniques of structural imaging (Clarke and Eberhardt 2002).

Based on the method of image acquisition, these instruments are classified into two groups which are known as light and electron microscopies.

2.8.1. Light microscopy techniques

Light microscopy (LM) is one of the most popular imaging techniques. It is regularly used to obtain fast, inexpensive quantitative and qualitative information in food analysis (Twyman 2005). These microscopes are composed of a beam of visible light (photons) that is the illumination probe source, a system to focus the source onto the sample (the condenser or condense glass lens), a place to locate the specimen, and the objective. There are different types of LM including bright field, polarizing, and fluorescence microscopy (Falcone, Baiano et al. 2006).

2.8.1.1. Bright field microscopy

The most common application of the LM is bright field illumination in which the light is continuously transmitted from an incandescent source and through the condenser, the specimen, an objective lens, and a second magnifying lens, before reaching the eye (Aguilera and Stanley

1999). To reflect light from an external source, the microscopes are either equipped with a mirror or an internal illuminator (Falcone, Baiano et al. 2006). Bright field microscopy is largely used in combination with a specific stain or dye, which is applied to foods, in order to improve contrast or differentiate tissues (Aguilera and Stanley 1999). It is appropriate to detect stained bacteria, thick tissue sections, thin sections with condensed chromosomes, large protists, living protists, algae, and other microscopic plant materials.

2.8.1.2. Polarizing microscopy

Polarizing technique of LM is used to study food components with crystalline structure. This type of foods exhibit birefringence properties. Birefringence is an optical phenomenon in which the sample exhibits different refractive indexes for light with plane polarization in two perpendicular directions (De Santis and Pantani 2013). Many food components are birefringent e.g., plant cell walls, muscle fibers, animal and plant fats, starch, and some flavor and seasoning components (Twyman 2005).

In this type of microscopy, two polarizers are inserted in the light path, which the first one is located between the light source and specimen and the second one between the objective and the eye. The generated light by the first polarizer vibrates in one of the planes perpendiculars to the direction of light transmit. Rotating of the second polarizer which is called analyzer allows distinguishing within a specimen the bright crystalline domains from the dark amorphous region (Falcone, Baiano et al. 2006). Common examples of the application of polarizing microscopy in the food microstructural studies are meat quality assessment via muscle fiber visualization and degree of muscle contraction measurement, identification of botanical origin of starch and following the phenomenon of starch gelatinization, and differentiating the fats and emulsions (Aguilera and Stanley 1999).

2.8.1.3. Fluorescence microscopy

Fluorescence microscopy is a system in which specimens either naturally show fluorescent properties or are induced to fluoresce; and then these emissions are studied microscopically (Aguilera and Stanley 1999). A wide range of plant and animals food components show natural fluorescence. For example, in plants these components include pigments, such as chlorophylls and carotenoids, phenolic compounds, such as lignin, and flavor compounds in herbs and spices. Examples for animal tissues include collagen, elastin, and some lipids. In addition, there are fluorescence dyes and components that fluoresce only in specific chemical environments (Twyman 2005). Nile red is a lipid-soluble dye that becomes fluorescent in hydrophobic mediums such as fats. In fluorescence microscopy, the high sensitivity of the technique and using the fluorescence capability of various compounds allow precise identification and localization of different components in foods.

2.8.1.4. Confocal laser scanning microscopy

This technique allows imaging through the depth of samples such that the features can be reconstructed in three dimensions (Tamime 2008). The instrument is built based on combination of old and new technologies and knowledge. Advanced computer imaging techniques, computer designed optics, and fluorescent probes developments have been integrally joined with advanced light microscopes. This combination has introduced an improved imaging instrument that is capable of capturing high resolution volumetric images of specimens (Aguilera, Stanley et al. 2000). It is a particular light microscopy technique that uses a point source of light focused on a fixed depth in the sample to obtain the image. The image is created line by line in the computer, as the beam scans the sample.

CLSM microscopy in conjunction with fluorescence probes provides the study of thin optical section in thick and intact samples (Falcone, Baiano et al. 2006). Labeling specific food components by dyes has produced the capacity for CLSM technique to provide useful information about distribution of these components through food products (Pedreschi, Cocio et al. 2008, Adedeji, Liu et al. 2011). This technique also allows the extraction of topographic data from a series of confocal images taken over a number of focal planes (Falcone, Baiano et al. 2006).

2.8.2. Electron microscopy techniques

Electron microscopy (EM) is a technique in contrast to LM, where its illumination source is electron focused with magnetic lenses rather than photons focused with glass lenses (Kaláb, Allan-Wojtas et al. 1995). This technique is used where a higher resolution than that obtained from LM techniques is required. Electron microscopy offers much greater detail than light microscopy (Falcone, Baiano et al. 2006). There are two principal EM modes (Kaláb, Allan-Wojtas et al. 1995) namely scanning electron microscopy (SEM) and transmission electron microscopy (TEM).

2.8.2.1. Scanning electron microscopy

Scanning electron microscopy provides the image of sample surfaces with a low energy beam (1-30KkeV) of electrons (Dudkiewicz, Tiede et al. 2011) and by emission or reflection of the electron beam (Falcone, Baiano et al. 2006). SEM investigation lets effective isolation and purification of food components, and helps to characterize the physical and textural properties of food materials (Lee and Rha 1979).

Conventional electron microscopy requires difficult and often destructive preparation techniques. It needs specific fixation, dehydration, and fat removal procedures, depending on the

physicochemical attributes and the required structural information. Recent improvements in electron microscopy has introduced a new technique, environmental scanning electron microscopy, which allows for imaging of moist samples, retaining the resolution whereas eliminating the difficult sample preparation steps (Timp and Matsudaira 2008).

Environmental scanning electron microscopy (ESEM) is a term frequently used for all SEM techniques able to operate at pressures high enough to allow imaging of samples with high moisture content without dehydration during image acquisition (James 2009). The operational difference to normal SEM is that the chamber, where the sample is placed in, is not subjected to high vacuum. Using an appropriate gas, typically water vapor, permits the sample chamber to work at 100-2500 Pa (Donald 2012). More generically, ESEM instruments that are capable of operating with specimen pressures up to approximately 2500 Pa are named variable pressure SEM (VP-SEM), whilst those instruments capable of operating with pressures up to approximately 600 Pa are known as low vacuum SEM (LV-SEM).

High pressure in the area around the sample brings some advantages such as the ability to image samples without applying a conductive coating, capturing images of high moisture content foods, and possibility of carrying out dynamic experiments (Timp and Matsudaira 2008, James 2009). VP-SEM has been largely applied to study the starch granules (James 2009). The specific role of starch and gluten in the structure of baked product has been investigated using this technique. It is possible to study the hydration and swelling of starch in ESEM when examining hydroscopic samples at high humidity while avoiding fully condensations and avoiding water film on the sample surface.

2.8.2.2. Transmission electron microscopy

Transmission electron microscopy is useful in determination of the internal structure of food materials (Falcone, Baiano et al. 2006). It is capable in studying the small scale structural elements in food products and the changes that occur in the matrix during the processing and storage. In TEM, a high energy electron beam (80-300 keV) is transmitted across a very thin layer of the food sample (Dudkiewicz, Tiede et al. 2011). A part of the beam is scattered from the sample and some of the electrons may be transmitted with little change in energy and direction. TEM imaging prepares information about the morphology of nano-sized object compared to SEM techniques, but preparation of these nano-sized samples is very challenging. The samples need to be sufficiently thin to let the electrons pass through.

2.8.3. Other techniques of microscopy

2.8.3.1. Magnetic resonance imaging

Magnetic resonance imaging is a nonionizing method with full three dimensional abilities, good soft tissue contrast, and high spatial resolution (Webb 2003). MRI technique is mostly used in the area of assessing brain problems and diseases, spinal disorders, cardiac function, and musculoskeletal damage (Falcone, Baiano et al. 2006). The application of this technique in food observations is a very new trend, where can be applied for food processing control and understanding the changes occurring in foods during processing. It is applied in large area of food controls from the detection of internal defects of foods, such as hollow heart in potatoes and bruises in apples, to complex analysis concerning grading the quality of some food products.

2.8.3.2. Scanning probe microscopy

Scanning probe microscopy operation is indeed mapping of particular surface parameters via analyzing the interaction between a microprobe and the sample surface (Clarke and Eberhardt

2002). There are a large number of microprobes that can be used for this technique, and based on these probes, different types of probe microscopy have been introduced namely atomic force microscopy (AFM), magnetic force microscopy (MFM), electrostatic force microscopy (EFM), scanning thermal microscopy (SThM), Pulsed force microscopy (PFM), force modulation microscopy (FMM), and scanning capacitance microscope (SCM).

AFM is the technique that is more often used in food microstructural assessments. This equipment gives the highest resolution that is accessible by direct imaging microscopy techniques, and can be used in time-lapse mode to study the changes during a process (Tamime 2008). AFM takes images at high magnification with high resolution in two or three dimensions with minimal task for sample preparation. AFM studies materials while scanning their surface. It takes images at high magnification with high resolution in two or three dimensions with minimal task for sample preparation (Liu and Cheng 2011).

X-ray computed tomography (CT) is a non-invasive, whole body scanning approach established initially for medicinal application (Jones, Lewis et al. 2004). However, this technique has been also used in other fields such as veterinary diagnostic in animals, analysis of porous cereal products, observation air bubbles in dairy products, and microstructural studies of food products (van Dalen, Blonk et al. 2003, Jones, Lewis et al. 2004, Adedeji and Ngadi 2009). This technique allows the analysis of the microstructure of food materials non-invasively up to a few millimeters across with a lateral and axial resolution down to a few nanometers. Its contrast is based on the difference in absorption of X-rays by the component, e.g. water and air of the samples (van Dalen, Blonk et al. 2003). It does not need primary sample preparation, and can be done under environmental conditions.

CONNECTING TEXT

Literature review shows that surface of fried products plays a very important role in fat absorption during frying. Some properties of surface of batter coating such as porosity and roughness greatly influence different fractions of fat in the final fried product. Pre-frying treatments have been recommended as techniques of fat reduction during frying of some foods. It was noted that scarce information is available about the effect of pre-heating temperature and batter formulation on moisture loss and fat absorption during batter frying. In chapter 3, different pre-heating temperatures were applied on batter formulated by only wheat flour to investigate how fat content changes. Then, the temperature that decreased fat content the most was chosen to study the effect of batter formulation, when different combinations of wheat and rice flour were used, on fat absorption during frying.

CHAPTER 3

EFFECTS OF PRE-HEATING TEMPERATURE AND FORMULATION ON POROSITY, MOISTURE CONTENT, AND FAT CONTENT OF FRIED BATTERS

3.1. Abstract

The effect of different pre-heating temperatures on moisture and fat contents, and porosity of wheat-flour-based batters during frying was studied. Batter pre-heated at 60°C and fried for 4 min showed higher moisture content, lower fat content and lower porosity than non-pre-heated batter and batters pre-heated at 70 and 80°C. Because of significant reduction in fat content, 60°C pre-heating temperature was chosen to study the effect of different batter formulations on moisture and fat content, and porosity. Five different wheat to rice flour ratios were prepared namely: 100 g wheat flour to 0 g rice flour , 75 g to 25 g, 50 g to 50 g, 25 g to 75 g, and 0 g to 100 g, and then each batter was pre-heated at 60°C. Batters formulated with higher amount of wheat flour showed higher moisture content, and lower fat content and porosity than batters formulated with higher amount of rice flour.

3.2. Introduction

Various techniques have been used to reduce fat uptake during frying. Application of coating is one of the most promising routes to reducing fat uptake (Mellema 2003). The use of batter and bread coating is a traditional method for preparing tasty and crispy fried products. Batter and breading consist of a variety of ingredients such as bread crumbs, corn meal, cracker meal, different types of flour, starch, hydrocolloids, salt, seasoning and water (Moreira, Castell-Perez et al. 1999, Fiszman and Salvador 2003). It is a complex system with very wide-range of ingredients; and the quality performance of the final products is highly dependent on interaction

between the ingredients. Adding water and heating the mixture result in a structural network; and create a tender and juicy, golden yellow color barrier crust, which prevents moisture loss and oil uptake during deep fat frying (Adedeji, Liu et al. 2011, Nasiri, Mohebbi et al. 2012).

Wheat flour is the most common flour used in batter systems (Xue and Ngadi 2007), while other flours such as corn, rice, and soy have also been largely used. Different flour types are usually added to improve product quality. Rice flour has been reported as an alternative for wheat flour for those with gluten intolerance. It has relatively low amount of proteins, especially gluten, and shows a weak elasto-plastic structure compared to wheat proteins. As a result rice flour makes thin slurries with poor adhesion properties. Shih, Boué et al (2004) and Firdevs Dogan, Sahin et al. (2005) recommended rice flour as a substitute to reduce oil uptake. More recently, Adedeji and Ngadi (2011) reported contradictory findings that application of rice flour to batter system increased fat content. Therefore, questions have been raised about the functionality of rice flour in reducing fat uptake during frying of batter.

Starch gelatinization and protein denaturation play a very important role in forming the barrier on the surface of coated foods and preventing oil uptake during frying. Starch gelatinization is the collapse of molecular arrangement in the starch granule resulting in granular swelling and loss of solubilisation (Yusop, Maskat et al. 2011). Gelatinization of starch during frying of a flour-based coating depends on the starch characteristics, such as its thermal properties, granule size, amylose and amylopectin content, and digestibility, starch/water ratio, oil temperature, heating rate, and process time. Batter composition, such as carbohydrates, proteins, lipids, salt, and other ingredients greatly influence water availability for starch gelatinization during frying. The composition results in different phase transition behavior and variation in thermal properties of starch (Xue and Ngadi 2007). Wheat gluten has shown a good

film-forming ability with the potential to prevent moisture loss and decrease oil absorption during frying (Day, Augustin et al. 2006). Moisture retention in formulated batter results in a lower density and, consequently, a more porous final batter texture (Salvador, Sanz et al. 2005).

As has been mentioned by many researches, oil uptake is basically a surface-related phenomenon resulting in competition between drainage and suction into the porous crust, when the fried product is removed from the hot oil and starts to cool down (Kochhar and Gertz 2004, Ahmad Tarmizi, Niranjana et al. 2013). It has been proposed that crust permeability is the main factor that influences the amount of fat uptake (Gazmuri and Bouchon 2009). In fact, most of the pre-frying treatments, such as pre-drying, pre-heating, and baking, have concentrated on surface structural changes and formation of crust with less permeability to oil absorption (Mellema 2003, Chen, Kuo et al. 2009, Soorgi, Mohebbi et al. 2012). Ngadi, Wang et al. (2009) and Adedeji, Ngadi et al. (2009) studied the effect of microwave cooking as a pre-treatment on mass transfer of batter coated chicken nuggets and reported a significant reduction in fat content of fried batter.

Most studies in the field of batter frying have only focused on the effect of batter formulation on fried product characteristics. However, not as much attention has been paid to the influence of pre-heating temperatures, as a pre-treatment technique, on the characteristics of fried batter coatings. The main objective of this study was to determine whether pre-heating temperature affects moisture content, fat content, and porosity of wheat-flour-based batter coatings; and the second objective was to determine how 60°C as a pre-heating temperature influences these parameters when batters with different formulations are prepared.

3.3. Materials and methods

3.3.1. Materials

Wheat flour (Five Rose All Purpose Flour, Les Cuisines Five Roses Kitchens, QC, Canada) was purchased from a local grocery store in Montreal, Canada. Long grain rice flour, RL-100, was prepared by Rivland Partnership (Riceland Foods, Arizona, USA). Carboxymethyl cellulose (CMC) was supplied by TIC Gums Inc., Maryland, USA. Canola oil (Les Essentiels De La Cuisine, Richmond, BC, Canada) was supplied by Food & Dining Service, McGill University.

3.3.2. Batter preparation

A solid mixture of wheat flour (96 g/100 g of total solid mixture), NaCl (2.5 g/100 g of total solid mixture), and CMC (1.5 g/100 g of total solid mixture) was prepared, and then was mixed thoroughly with distilled water in the ratio of 1:1.3. To study the influence of pre-heating temperature, the wheat base batter slurry was poured in rectangular aluminum cells (50×25×5 mm), covered with aluminum caps, and then pre-heated at three different temperatures, namely 60, 70 and 80°C for 10 min in a water bath. The non-preheated batter was considered as the control sample. The aluminum cells were then quickly cooled down in an ice bath. Finally, the aluminum caps were removed, and the cells with the batter inside were transferred for frying.

Further study on the influence of batter formulation on moisture content, fat content and porosity of fried batters was conducted at pre-heating temperature of 60°C. Five different ratios of wheat and rice flours were mixed to make batter namely: 100 g wheat flour to 0 g rice flour (100W0R), 75 g wheat flour to 25 g rice flour (75W25R), 50 g wheat flour to 50 g rice flour (50W50R), 25 g wheat flour to 75 g rice flour (25W75R), and 0 g wheat flour to 100 g rice flour (0W100R). Similar to the first batch of batters, 2.5 g NaCl and 1.5 g CMC were added to

96 g solid mixtures, and then mixed with distilled water in the ratio of 1:1.3. Finally the batter slurries were poured in the aluminum cells and transferred for frying.

3.3.3. Frying

A programmable deep fat fryer (Henny Penny Computron 7000 pressure Fryer, Model 500C; HP Corporation, Eaton, OH, USA) was used to fry the batter samples at $180 \pm 2^{\circ}\text{C}$ for 1, 2, 3, and 4 min. The fryer was filled with fresh canola oil and preheated and maintained at $180 \pm 2^{\circ}\text{C}$ for 2 hrs before frying. The oil was replaced each time after 2 hrs frying. Excess oil on the surface was mopped off using paper towel. Fried samples were then allowed to cool under ambient conditions.

3.3.4. Moisture content

The fried batter was freeze-dried in a freeze dryer (Modulyod-115; ThermoSavant, Holbrook, NY, USA) at -50°C and 100 mbar for 36 h and was allowed to equilibrate in a desiccator for 30 min. Weight of batter before and after freeze-drying was measured and used to calculate the moisture content.

3.3.5. Fat content

The freeze-dried batters were ground in a coffee grinder (Bodum 5678-57; C-Mill, Bodum Inc., New York, NY, USA), and then 3-5 g of samples was placed in thimbles in a VELP SER 148 (Velp Scientifica, Usmate, Italy) solvent extraction unit, and oil was extracted with petroleum ether. Oil content (dry basis) was computed by dividing the mass of extracted oil with the mass of freeze-dried sample.

3.3.6. Apparent density

To obtain apparent density, apparent volume of the samples was first measured. Fried batter samples were kept for 30 min at room temperature to cool down and their apparent volume

(volume of the sample including closed pores but excluding the open pores, ρ_a) was measured according to the method described by Adedeji and Ngadi (2011). Apparent volume of the weighed batters was measured in a helium pycnometer (Model 1305 Multivolume, Micromeritics Instrument Corporation, Norcross, GA) following the standard protocol (Micromeritics. 1992). The analysis was conducted at ambient temperature. All treatments were applied in triplicate and each sample was measured thrice. Apparent density was determined as the ratio of the mass of sample divided by the apparent volume.

3.3.7. Bulk density

The same set of samples used for apparent density measurements was weighted and then quickly dipped in a melted paraffin wax in order to cover the surface openings of the samples and then allowed to cool at room temperature. The difference between weight of batters before and after covering by paraffin wax was taken as the mass of paraffin wax. Knowing the mass and density of paraffin, the volume of the paraffin coating layer was measured. Samples were then dropped into a water displacement pycnometer and the displaced volume was recorded as the volume of the sample coated with paraffin. The difference between volume of paraffin wax and volume of the sample coated with paraffin wax was used as the bulk volume of batters. Bulk density was determined as the ratio of the mass of sample divided by the bulk volume.

3.3.8. Porosity

Porosity (ε) was calculated as the ratio of bulk density, ρ_b , to apparent density, ρ_a , subtract from one, i.e. a ratio of the void/space volume present in the sample after frying to its overall volume:

$$\varepsilon = 1 - \left(\frac{\rho_b}{\rho_a} \right)$$

Equation (3-1)

3.3.9. Statistical analysis

All treatments were applied in triplicates. Analysis of variance was performed using SAS system software (Version 9.3, SAS Institute, Inc., Cary, NC, USA) to determine the effect of main parameters, batter formulation and pre-heating temperature, on porosity, moisture and fat content of the samples. For the first set of experiment, Duncan multiple range test (MRT) was used, at 4 min of frying, for mean separation at $P < 0.05$ where treatment effect was significant to see how pre-heating temperature affects porosity, moisture and fat content of the wheat-flour-base batter samples. Similarly, for the second set of experiment, Duncan multiple range test (MRT) was used, at 4 min of frying, for mean separation at $P < 0.05$ where treatment effect was significant to see how batter formulation affects porosity, moisture and fat content of the 60°C pre-heated batter samples.

The “PROC Corr” of SAS software was used for finding the Pearson’s coefficient of correlation (r) between each pair of two dependent variables. The degree of correlation was described as follows (Agbisit, Alavi et al. 2007, Rahimi and Ngadi 2014): ($|r| < 0.20$, negligible; $|r| = 0.20\text{--}0.40$, low; $|r| = 0.40\text{--}0.60$, moderate; $|r| = 0.60\text{--}0.80$, marked; and $|r| > 0.80$, high).

3.4. Results and discussion

3.4.1. Pre-heating temperature

Figure 3-1 shows the moisture content profile during frying of wheat-flour-based batters pre-heated at different temperatures. Analysis of variance (ANOVA) showed that the interaction effect of pre-heating temperature and frying time on variation observed in moisture content at a confidence interval of 95% was significant. As was expected, moisture content significantly ($P < 0.05$) decreased during frying. Duncan multiple range test (MRT) at 4 min frying was performed to compare the effect of different pre-heating temperatures on moisture content. Non-preheated

batter and batter pre-heated at 60°C had significantly ($P < 0.05$) higher amount of moisture content than batters pre-heated at 70 and 80 °C, while no significant difference was observed between non-preheated batter and 60 °C pre-heated batter, and also between batters pre-heated at 70 and 80°C.

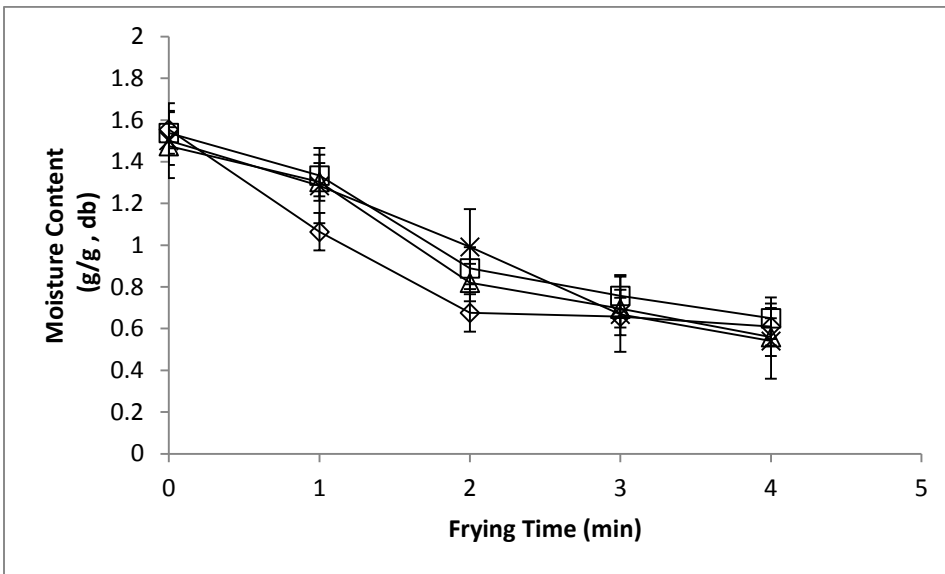


Figure 3-1 Moisture content vs. frying time of wheat flour based batters pre-heated at different temperatures. ◇: Non-pre-heated Batter, □: Batter pre-heated at 60°C, Δ: Batter pre-heated at 70°C, ×: Batter pre-heated at 80°C.

The major protein in wheat flour is gluten, which consists of generally about 80 to 85% of total wheat protein (Veraverbeke and Delcour 2002, Kovacs, Fu et al. 2004). Wheat gluten has the ability to form a viscoelastic film after hydration which reduces moisture loss (Cao, Wen et al. 2009). Although gluten is the main component responsible for viscoelastic properties of batters made from wheat flour, starch shows an important role in determining this behavior by interacting with gluten as well (Champenois, Rao et al. 1998). However, as was explained earlier, the batters were pre-heated in a close aluminum cell, where no moisture could evaporate during heating. Under this condition, high temperature heating, such as 70 and 80°C, caused over starch

swelling and protein denaturation, which destroys the structure of the film, and therefore higher moisture loss than non-pre-heated and 60°C pre-heated batters took place during frying.

Fat content profile during frying of wheat-flour-based batters with different pre-heating temperatures is illustrated in Figure 3-2. The interaction effect of pre-heating temperature and frying time on fat content at a confidence interval of 95% was significant. Fat content significantly ($P < 0.05$) increased during frying. Duncan multiple range test (MRT) at 4 min was performed to compare the effect of pre-heating temperature on fat content. Pre-heating significantly ($P < 0.05$) decreased fat content. Batter pre-heated at 60 °C showed lower amount of fat than batters pre-heated at 70 and 80 °C, while non-pre-heated batter showed the highest fat content. Kawas and Moreira (2001) steam-baked tortilla chips to gelatinized starch before frying and found that starch gelatinization inhibited oil penetration. Pre-gelatinized starch showed to lessen oil content during frying of batter coated chicken nuggets (Altunakar, Sahin et al. 2004). Gluten incorporation in formulation of batter coating presented relatively lower oil content in fried seafood (Salvador, Sanz et al. 2005). Starch gelatinization and gluten denaturation during pre-heating form a strong film that acts as a barrier to mass transfers and prevents oil absorption. According to the results reported by Addo, Xiong et al. (2001), when a high gluten flour is heated to 60 to 65°C , elastic networks form. Gluten, starch, and some minor component such as lipids and soluble proteins are involved in the formation of these elastic matrices. At temperatures higher than 65°C the gluten-starch networks appear to be disrupted possibly due to unfavorable protein-carbohydrate interaction or competition for water between gluten and starch. The disrupter of the network at temperatures higher than 65°C might be a possible reason for higher fat permeability of batters preheated at 70 and 80°C. Another probable reason is that starch molecules leach out at temperature beyond 65°C, and consequently the gluten network is

weakened due to the removal of substances which support to strengthen the matrix. Champenois, Rao et al. (1998) reported that at temperatures higher than 70°C disruption of starch granules happens, which makes it easier for the molecules to move because of rising their flexibility.

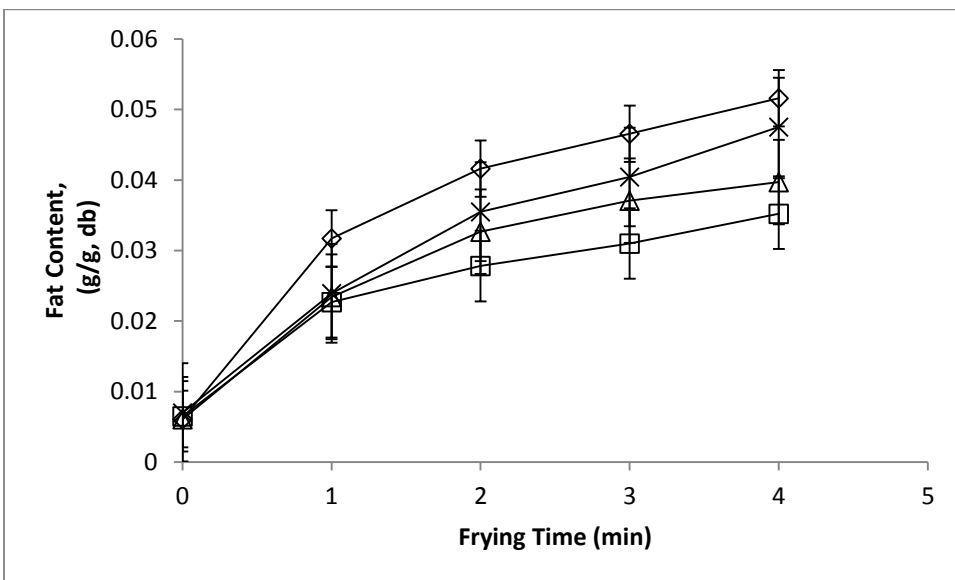


Figure 3-2 Fat content vs. frying time of wheat flour based batters pre-heated at different temperatures. \diamond : Non-pre-heated Batter, \square : Batter pre-heated at 60°C, Δ : Batter pre-heated at 70°C, \times : Batter pre-heated at 80°C.

Porosity during frying of batters with different pre-heating temperatures is shown in Figure 3-3. Porosity range for the fried batters was 5–45.30%, 6–21.40%, 6.60–13.14%, and 8.85–17.96%, for non-pre-heated, 60°C pre-heated, 70°C pre-heated, and 80°C pre-heated batters. Adedeji and Ngadi (2011) used the same pycnometry technique to study the porosity of same product and reported the porosity range of non-pre-heated deep-fat-fried coatings between 2.20–47.90%, which is very close to range obtained in our study for non-preheated samples. The interaction effect of pre-heating temperature and frying time on porosity at a confidence interval of 95% was significant. Porosity significantly ($P < 0.05$) increased with frying time. Duncan multiple range test (MRT) at 4 min was performed to compare the effect of pre-heating

temperature on porosity. Non-pre-heated batters showed significantly ($P < 0.05$) higher porosity than batters pre-heated at 60, 70, and 80°C. Porosities of all pre-heated batters were in the same range although the batter pre-heated at 60°C was significantly higher than those pre-heated at 70 and 80°C. No significant difference ($P < 0.05$) was observed between batters pre-heated at 70 and 80 °C. The significant higher porosity observed for non-pre-heated batter could be, as discussed earlier, connected to the film created because of starch gelatinization and gluten denaturation of wheat flour during pre-heating. The created film prevents moisture loss during frying, and therefore reduces pore generation.

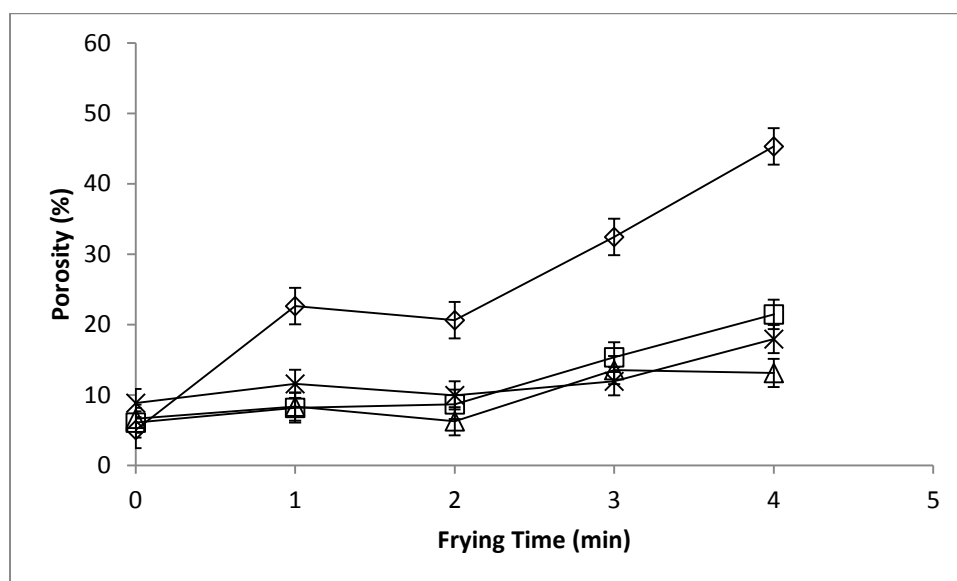


Figure 3-3 Porosity vs. frying time of wheat flour based batters pre-heated at different temperatures. ◇: Non-pre-heated Batter, □: Batter pre-heated at 60°C, Δ: Batter pre-heated at 70°C, ×: Batter pre-heated at 80°C.

The plot of porosity versus fat content of pre-heated batters at different temperatures is illustrated in Figure 3-4. Pearson's correlation showed that fat content had a marked to high positive correlation with porosity, where degree of correlation ranged from 0.69 to 0.91. The positive correlation between porosity and fat content confirms that both parameters increased

simultaneously during frying. These findings further support the statement by Adedeji, Ngadi et al. (2009) and Adedeji and Ngadi (2011) that higher porosity results in more oil uptake.

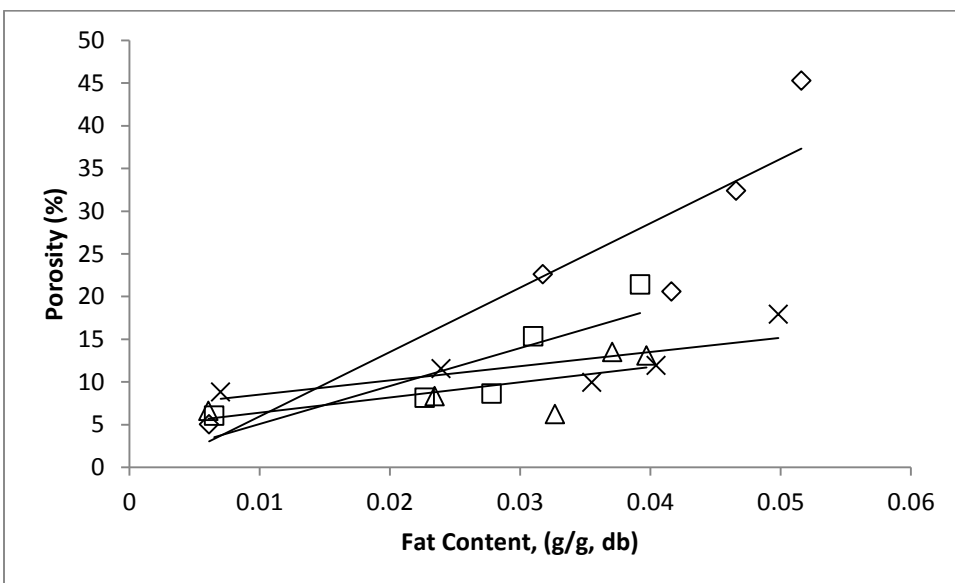


Figure 3-4 Porosity vs. fat content of wheat flour based batters pre-heated at different temperatures. ◇: Non-pre-heated Batter, □: Batter pre-heated at 60°C, Δ: Batter pre-heated at 70°C, ×: Batter pre-heated at 80°C.

3.4.2. Batter formulation

Figure 3-5 shows the moisture content profile during frying of batters with different formulations that were pre-heated at 60°C. Analysis of variance (ANOVA) showed that the interaction effect of batter formulation and frying time on variation observed in moisture content at a confidence interval of 95% was significant. Similar to the results of pre-heating temperatures, moisture content significantly ($P < 0.05$) decreased during frying. Duncan multiple range test (MRT) at 4 min was performed to compare the effect of batter formulations on moisture content. Increasing the amount of wheat flour in formulation of batter significantly ($P < 0.05$) increased the moisture content of batters after 4 min frying. Variation in protein, gluten, and starch contents (Hoseney, Finney et al. 1970, Veraverbeke and Delcour 2002, Torbica, Hadnadev et al. 2012) could be the possible reason for this different behavior. As considered earlier, the main protein in wheat flour

is gluten, while rice is known as a gluten free substitute (Torbica, Hadnadev et al. 2012). In contrast to wheat flour, which shows a high water holding ability because of gluten protein, rice proteins possess a low water binding capacity (Cao, Wen et al. 2009) and lacks the ability to form the necessary network for holding moisture.

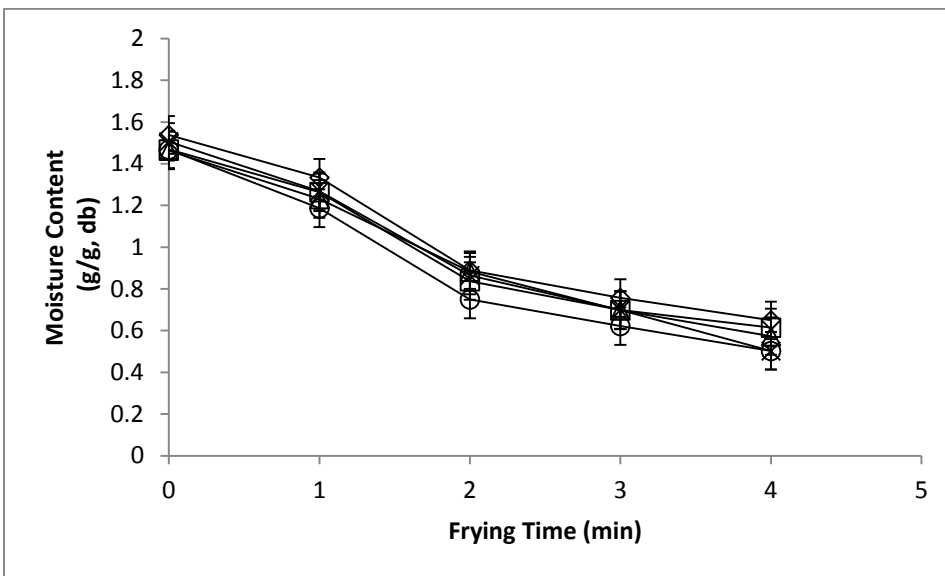


Figure 3-5 Moisture content vs. frying time of batters with different formulations pre-heated at 60°C. ◇: 100 g wheat flour to 0 g rice flour (100W0R), □: 75 g wheat flour to 25 g rice flour (75W25R), Δ: 50 g wheat flour to 50 g rice flour (50W50R), ×: 25 g wheat flour to 75 g rice flour (25W75R), and ○: 0 g wheat flour to 100 g rice flour (0W100R).

The effect of formulation on fat content during frying of batters pre-heated at 60°C is shown in Figure 3-6. The interaction effect of batter formulation and frying time on variation observed in fat content at a confidence interval of 95% was significant. Similar to the results found for pre-heating temperatures, fat content of batters with different formulation increased during frying time. Duncan multiple range test (MRT) was used to compare the effect of batter formulation on fat content after 4 min frying. More rice flour content in the formulation of batter significantly ($P < 0.05$) increased the amount of fat content observed in batters fried for 4 min. This agrees with results found by Adedeji and Ngadi (2011) and Ngadi, Wang et al. (2009). The

lower fat content in batters with higher amount of wheat flour could be because of film making ability of gluten protein in wheat flour as discussed earlier, and higher moisture retention during frying (Figure 3-5). However, our findings disagree with results published by Shih, Boué et al. (2004) and Firdevs Dogan, Sahin et al. (2005), where reduction of fat uptake with addition of rice flour to batter was observed. This difference between fat content results reported by different studies might be contributed to the post frying process, where in this study, and other studies with similar results, fried products were mopped off to remove the extra fat from the surface, while in studies by Shih, Boué et al. (2004) and Firdevs Dogan, Sahin et al. (2005), the extra oil after frying was left on the surface. A big fraction of oil is absorbed after frying and during cooling of the fried product (Durán, Pedreschi et al. 2007), therefore the post frying processes, such as removing the extra fat, highly influences the fat absorption behavior of the product.

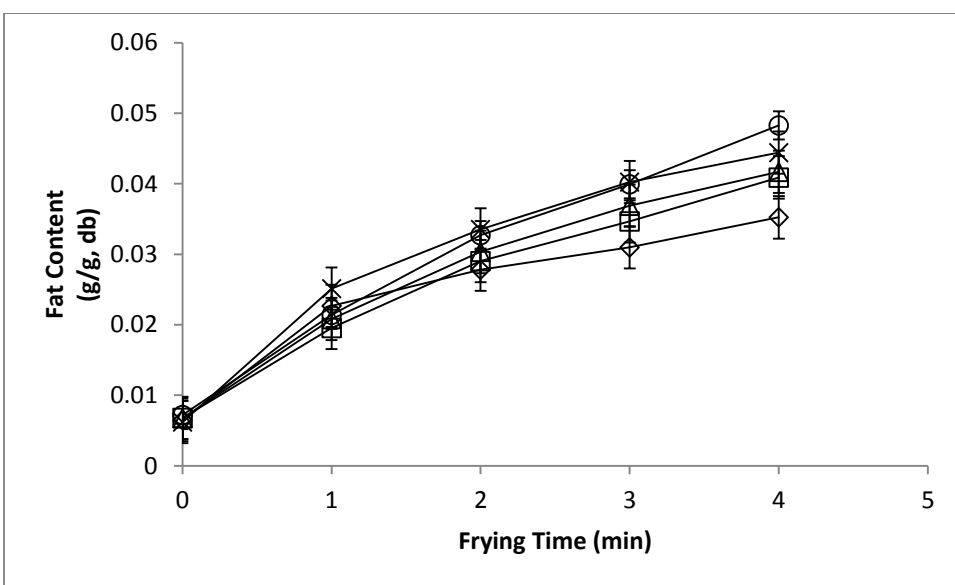


Figure 3-6 Fat content vs. frying time of batters with different formulations pre-heated at 60°C. ◇: 100 g wheat flour to 0 g rice flour (100W0R), □: 75 g wheat flour to 25 g rice flour (75W25R), Δ: 50 g wheat flour to 50 g rice flour (50W50R), ×: 25 g wheat flour to 75 g rice flour (25W75R), and ○: 0 g wheat flour to 100 g rice flour (0W100R).

Porosity during frying of batters with different formulations, which were pre-heated at 60°C, is shown in Figure 3-7. Porosity range for the fried batters was 6–21.40%, 3.93–22.51%, 5.58–25.52%, and 8.13–27.31%, and 5.56–30.34% for 100W0R, 75W25R, 50W50R, 25W75R, and 0W100R batters, respectively. The interaction effect of batter formulation and frying time on variation observed in porosity at a confidence interval of 95% was significant. Porosity significantly ($P < 0.05$) increased with frying time. Results were in agreement with those reported by Adedeji and Ngadi (2011), Taiwo and Baik (2007), and Krokida, Oreopoulou et al. (2000). Intense heating during deep fat frying causes water evaporation that creates some ruptures such as pores and channels. The longer the frying, the more pores are created. Duncan multiple range test (MRT) at minute 4 was performed to compare the effect of formulation on porosity of fried batters. As it is clear from Figure 3-7, batters prepared with higher amount of wheat flour showed significantly ($P < 0.05$) lower porosity than batters prepared with higher amount of rice flour. Lower porosity for batter formulated with more wheat flour could be linked to the fact that wheat has higher protein content than rice; makes a stronger gel, and therefore fewer pores are created.

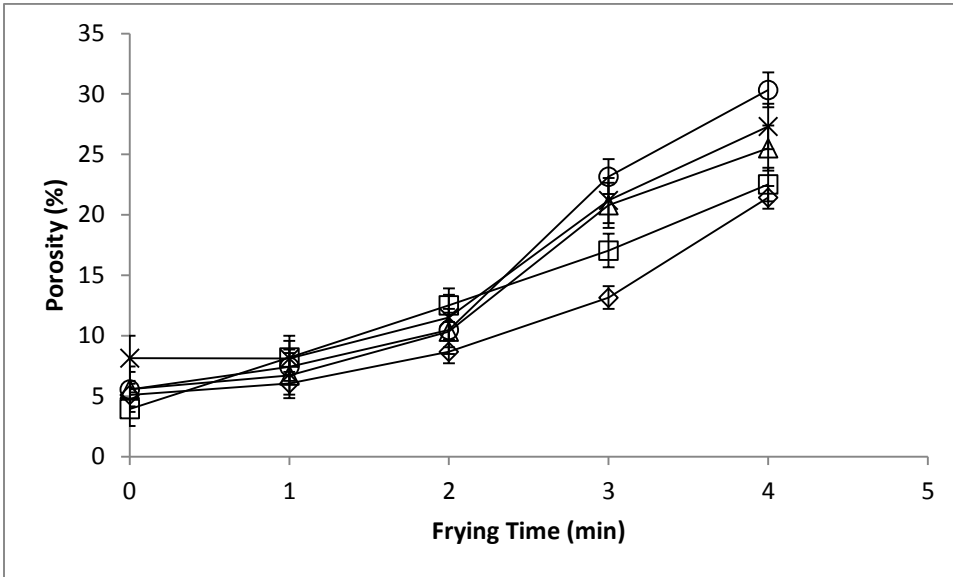


Figure 3-7 Porosity vs. frying time of batters with different formulations pre-heated at 60°C. ◇: 100 g wheat flour to 0 g rice flour (100W0R), □: 75 g wheat flour to 25 g rice flour (75W25R), △: 50 g wheat flour to 50 g rice flour (50W50R), ×: 25 g wheat flour to 75 g rice flour (25W75R), and ○: 0 g wheat flour to 100 g rice flour (0W100R).

3.5. Conclusions

Pre-heating temperature, batter formulation, and frying time, had significant ($P < 0.05$) effects on moisture loss, fat uptake and porosity of batter coating during frying. Pre-heating significantly decreased the amount of fat absorption during frying. Wheat-flour-based batters pre-heated at 60°C showed the lowest fat content and porosity compared to non-pre-heated batter and batters pre-heated at higher temperatures. Pre-heating at temperatures higher than 60°C weakened the preventing role of the generated film as a barrier against fat absorption. Therefore, 60°C was recommended as an ideal temperature for pre-heating of wheat-flour-based batters. Pre-heated batters formulated with higher amount of wheat flour showed less moisture loss, fat content, and porosity than pre-heated batters which were formulated with higher amount of rice flour.

CONNECTING TEXT

There have been recent reports that oil absorption is a surface phenomenon and a big part of the oil which remains on the surface of fried foods during frying is absorbed after frying when the food is cooling down. A technique was used in chapter 4, to measure different fractions of oil namely penetrated, surface and total fat in fried batter when pre-drying and different formulations were used. It was also decided not to remove the excess oil on the surface to compare the results for fat content in this work to the results obtained from chapter 3 to see whether post frying process is important in fat reduction.

CHAPTER 4

EFFECT OF BATTER FORMULATION AND PRE-DRYING TIME ON OIL DISTRIBUTION FRACTIONS IN FRIED BATTER

4.1. Abstract

Batter coating and pre-drying have recently been used as techniques for decreasing fat content of fried foods. The objectives of this study were to assess the effect of batter formulations and pre-drying time on surface, penetrated and total fat content of batter coating, and to evaluate different mathematical models for predicting moisture diffusivity and oil transfer during frying. Two different sets of batter systems were used. The first set consisted of batter with different ratios of wheat to rice flour whereas the second set was wheat-flour-based batter with different pre-drying times. The batter samples were deep fried at $180 \pm 2^{\circ}\text{C}$ in canola oil for different frying times between 0 to 4 min. Batter with wheat flour alone was found to have higher surface fat and total fat contents, while its penetrated fat content was lower than that with other batters. Batter with no pre-drying showed higher surface, penetrated and total fat content than other systems. Moisture diffusivity ranged between 2.01×10^{-6} and $2.85 \times 10^{-6} \text{ m}^2/\text{min}$. Page model was found as the best fit model to evaluate the oil transfer rate constants.

4.2. Introduction

Certain batter coatings have been shown to reduce fat uptake in deep-fat fried foods (Mellema 2003, Altunakar, Sahin et al. 2004, Akdeniz, Sahin et al. 2006, Adedeji and Ngadi 2011). It is a basically liquid dough containing flour and water, into which a product is dipped before it is fried (Kim, Lee et al. 2008, Visser, De Beukelaer et al. 2008, Albert, Perez-Munuera et al. 2009). The batter system acts as a barrier to mass transfer during frying thus reducing fat uptake and preventing moisture loss. It also improves food texture, flavor, aesthetic appeal, weight and

volume. Batter formulation is highly flexible, and apart from flour, it may also have other ingredients such as starch, seasoning, leavening agents, hydrocolloids, bread and crumbs. Wheat flour is the most common flour used in batter systems (Xue and Ngadi 2007), and it is easily available in most regions. However there is an increasing interest in using other substitutes such as corn, rice, soy and other types of flours. Rice flour has been used in batter systems since it is considered a healthier alternative providing fewer calories. Although rice flour has less thickening ability than wheat flour, it is reported to have a better functionality in reducing fat absorption (Barutcu, Sahin et al. 2009). The moisture and fat contents along with texture are among the physico-chemical properties of the fried products.

Pre-drying reduces the initial moisture content of the product; and longer pre-drying time results in less free moisture content during frying (Gupta, Shivhare et al. 2000). The initial moisture content of food is the most clearly known factor which impacts oil gain during frying (Krokida, Oreopoulou et al. 2001). It was reported (Gupta, Shivhare et al. 2000) that pre-dried potatoes absorbed less fat compared to non-pre-dried samples. Pre-fry drying apparently creates compact material matrix with less available moisture content. Less moisture content causes less inner vacuum pressure after taking out the food from fryer; and therefore less oil is sucked into the food (Debnath, Bhat et al. 2003).

Numerous studies have shown that a large fraction of fat remains on the surface region of the products during frying process, and it is immediately absorbed during the product's cooling phase, i.e. when food is removed from hot oil (Gamble, Rice et al. 1987, Moreira and Barrufet 1996, Ufheil and Escher 1996, Aguilera and Gloria-Hernandez 2000, Pedreschi and Moyano 2005, Durán, Pedreschi et al. 2007, Adedeji and Ngadi 2011, Ahmad Tarmizi, Niranjana et al. 2013). When fried products are removed from the fryer, high temperature difference develops

between the surface and the interior, which in turn generates high negative pressure in the pore space leading to more fat penetration into the product's microstructure during cooling. Generally, in terms of fat distributions in a fried product, 3 different fat fractions are defined: (i) structural fat, which is the fat absorbed during frying, (ii) penetrated surface fat, which represents the fat absorbed into the food during cooling after removal from the fryer, and (iii) surface fat, which represents the surface fat that remains on the surface and does not penetrate into the body of the food (Durán, Pedreschi et al. 2007, Pedreschi, Cocio et al. 2008). Durán, Pedreschi et al. (2007) studied fat partitions in pre-treated potato slices during frying and cooling phases. These authors reported that potato chips absorbed nearly 38 g/100 g of the total fat content during frying, and almost 62 g/100 g of the total fat content remained on the surface of the product without penetrating into the microstructure. However, during the cooling stage, 65 g/100 g of total fat content was absorbed into potato chips and only 35 g/100 g remained at the chip surface. In order to control fat absorption during frying of batter coated products, it is essential to understand the mechanisms involved during the process. There is a need to relate the total fat content, fat adhesion on the surface and fat transfer to the inner structure of a fried battered product. A number of studies have been published on the fat uptake phenomenon during frying in various battered products. However, there is no information about the different fractions of fat in fried batters as influenced by different formulations and pre-drying. The objectives of this study were to study distribution of fat fractions in batter coated systems and how batter formulation and pre-drying influence fractions of surface, penetrated and total fat contents of fried batter coatings, and also to evaluate different mathematical models for predicting moisture diffusivity and oil transfer during frying .

4.3. Materials and methods

4.3.1. Materials

Wheat flour (Five Rose All Purpose Flour, Les Cuisines Five Roses Kitchens, QC, Canada) was purchased from a local grocery store in Montreal, Canada. Long grain rice flour, RL-100, was prepared by Rivland Partnership (Riceland Foods, Arizona, USA). Carboxymethyl cellulose (CMC) was supplied by (TIC Gums Inc., Maryland, USA). Canola oil (Les Essentiels De La Cuisine, Richmond, BC, Canada) was supplied by Food & Dining Service, McGill University.

4.3.2. Batter preparation

Five different ratios of wheat and rice flours were prepared namely: 100 g wheat flour to 0 g rice flour (**100W0R**), 75 g wheat flour to 25 g rice flour (**75W25R**), 50 g wheat flour to 50 g rice flour (**50W50R**), 25 g wheat flour to 75 g rice flour (**25W75R**), and 0 g wheat flour to 100 g rice flour (**0W100R**). A fixed amount of salt (NaCl) and CMC were added at 2.5 and 1.5 g/100g, respectively. Batter slurry was prepared by adding distilled water to solid mix in the ratio of 1.3:1. Then, the batter system was thoroughly mixed for 1 min (Sumeet, CM/L – 0973467, Indai). To study the influence of pre-drying, in a different set, wheat flour alone was similarly mixed with NaCl, CMC, and distilled water. It was then partially pre-dried in an oven dryer (Isotemp 700, Fischer Scientific, Pittsburgh, PA) at 60° C for three pre-drying times namely 15, 30 and 60 min. Batter prepared from wheat flour alone and not pre-dried was considered the control batter. Batter samples in this case were namely: Non-dried batter (**0MD**), batter pre-dried for 15 min (**15MD**), batter pre-dried for 30 min (**30MD**), and batter pre-dried for 60 min (**60MD**).

4.3.3. Frying

A rectangular aluminum cell of dimension 50×25×5 mm was constructed and used to hold around 8 g batter. The advantages of using this device are: (1): the reproducibility of data

obtained is better than coating a real food directly with batter, (2): the amount of batter pick up, which is the batter applied to the surface of the product, is constant for all formulation, and (3): crust formation around the commercial products would be simulated. Visser, De Beukelaer et al. (2008) used a similar device to study the properties of crust resulted during deep fat frying of a deep fried model. They reported that the resulted crust was comparable to commercial products. A kitchen deep fat fryer (T-FAL, Model 6197, Scarborough, Ontario, Canada) was used. The fryer was filled with 1.5 L canola oil and temperature was measured using a K-type thermocouple (HHM16, OMEGA Engineering Inc., Stamford, CT, USA). The fresh oil was preheated and maintained at $180 \pm 2^{\circ}\text{C}$ for 2 h before frying. All samples were fried for 1, 2, 3, and 4 min, while the temperature of the oil was maintained at $180 \pm 2^{\circ}\text{C}$

. To minimize the variation of oil properties due to degradation during frying, each batch of oil was used for only 30 min before it was replaced with a fresh batch of oil. All treatments were prepared and fried thrice.

4.3.4. Moisture content

Moisture content of fried batters was measured by drying the samples in an oven at 105°C for 24 h. Weights of sample before and after drying were used to calculate moisture content.

4.3.5. Fat content

Three different fractions of fat content were studied namely: surface oil content (SF), penetrated oil content (PF), and total oil content (TF). SF was measured following the methodology implemented by Pedreschi, Cocio et al. (2008) with modifications. In 15 s after frying, fried batter samples were immersed in a 140 ml glass extraction vessel containing 100 ml petroleum ether for 3 s at 25°C . Then, petroleum ether from the glass vessel was removed by evaporation using a VELP SER 148 (VelpScientifica, Usmate, Italy) solvent extraction. SF content was

computed on dry weight basis by dividing the mass of oil in the glass vessel by the mass of dried fried batter before immersion in petroleum ether. SF is defined as the oil fraction which adhered to the product surface and did not penetrate the batter microstructure during frying. PF is defined as the oil fraction which penetrated the batter microstructure during frying. To measure PF, the fried sample, which was already immersed in petroleum ether for SF measurement, was dried in an oven (Isotemp 700, Fischer Scientific, Pittsburgh, PA) at 105°C for 24 h, then was grinded in a coffee grinder (Black & Decker, Concord, ON, Canada), and finally was placed in thimbles in a VELP SER 148 (Velp Scientifica, Usmate, Italy) solvent extraction unit. Oil was extracted by conventional method using petroleum ether. The penetrated oil content was measured on dry weight basis by dividing the mass of extracted oil from the ground sample after first extraction by the mass of dried sample (AOAC 1990). TF was defined as the sum of SF and PF.

4.3.6. Effective moisture diffusion and kinetics models

Effective diffusion coefficient in the fried batters can be modeled using the solution to the Fick's second law of diffusion equation (Ngadi, Dirani et al. 2006, Vasić, Grbavčić et al. 2014) as presented in Equation (4-1):

$$MR = \frac{M - M_e}{M_0 - M_e} = \frac{8}{\pi^2} \sum_{n=0}^{\infty} \frac{1}{(2n+1)^2} \exp\left(-\frac{(2n+1)^2}{4} \pi^2 \frac{D_{eff} t}{d^2}\right) \quad \text{Equation (4-1)}$$

where MR is moisture content ratio, M_0 , M , and M_e are respectively initial moisture content (dry basis), moisture content at time t (dry basis), and moisture content of the final product (dry basis), D_{eff} is the effective moisture diffusion coefficient (m^2/min), and d is the thickness (m) of the batter coating. As the thickness of the batters was much smaller than the other dimensions, it was assumed that the batter was an infinite slab. To apply this model, the following assumptions were also made: initial uniform distribution of moisture and temperature

in the batters, minimal shrinkage and gradients in temperature and moisture, and mass transfer from just one side of the batters (as it was covered from the other sides by the cell).

For $n > 0$, only the first term in Equation (4-1) is significant (Dak and Pareek 2014); and the sum of all other terms is assumed equal to the coefficient ε . Therefore, Equation (4-1) can be rewritten as

$$MR = \frac{M - M_e}{M_0 - M_e} = \frac{8}{\pi^2} \exp\left(-\frac{\pi^2 D_{eff} t}{4d^2}\right) + \varepsilon \quad \text{Equation (4-2)}$$

In the further calculations, ε was chosen equal to 0.189; so that when $t = 0$, $MR=1$. Since the equilibrium moisture content is negligible in the case of deep fat frying, Equation (4-2) reduces to:

$$MR = \frac{M}{M_0} = \frac{8}{\pi^2} \exp\left(-\frac{\pi^2 D_{eff} t}{4d^2}\right) + \varepsilon \quad \text{Equation (4-3)}$$

Different kinetic models namely Newton, Page, modified page, and Henderson & Pabis (Table 4-1) were used to describe the fat uptake rate (penetrated fat uptake) of batters during frying. Where k is frying rate constant (min^{-1}); n , and a are frying coefficients which their values depend on the applied model and frying curve; t is frying time (min) and FR is the fat content ratio that is calculated following Equation (4-4):

$$FR = \frac{F_e - F}{F_e - F_0} \quad \text{Equation (4-4)}$$

where F_0 , F and F_e are initial fat content (dry basis), penetrated fat content at time t (dry basis), and equilibrium penetrated fat content (dry basis), respectively. It was observed that penetrated fat content for all batter systems reached a constant amount after a few minutes of frying. Therefore, PF content after 4 min frying was taken as equilibrium PF content.

Table 4-1 Various mathematical models used for predicting frying kinetics of batter (Singh, Nair et al. 2013).

Model Name	Equation	Equation No
Newton	$FR = e^{-kt}$	Equation (4-5)
Page	$FR = e^{-kt^n}$	Equation (4-6)
Modified Page	$FR = e^{(-kt)^n}$	Equation (4-7)
Henderson & Pabis	$FR = a * e^{-kt}$	Equation (4-8)

FR: fat content ratio, k: kinetic of oil penetration, n and a: constant numbers.

In order to choose the better suitable model for fitting the frying kinetics, the coefficient of determination (R^2) and the root mean square error (RMSE) associated with the prediction models were taken into account. The MATLAB (Version 8.1.0.604 R2013a, The Math works, Inc., USA) package was used to obtain diffusion coefficient and the kinetic parameters.

4.3.7. Statistical Analysis

All treatments were applied in triplicates. Analysis of variance was performed using SAS system software (Version 9.2, SAS Institute, Inc., 1999, Cary, NC, USA) to determine the effect of formulation and pre-drying time on moisture and fat content of the samples, and also to statistically compare frying kinetics and moisture diffusions of different batters. Duncan multiple range test (MRT) was used for mean separation at $P < 0.05$ where treatment effect was significant.

4.4. Results and discussion

4.4.1. Batter formulation

Analysis of variance (ANOVA) on moisture content obtained from the experiments showed that the interaction effect of batter formulation and frying time on variation observed in moisture

content at a confidence interval of 95% was not significant. Duncan multiple range test (MRT) was performed to study the effect of the main parameters, i.e. batter formulation and frying time, on moisture content. The initial moisture content (db) for all batters was 141.63-147.47 g/100g of the sample, which decreased to approximately 67.66 – 80.13 g/100g after 4 min frying (equilibrium moisture content). There was a rapid moisture loss within the first 3 min of deep frying, and after that, moisture content slightly reached to an almost constant rate. Similar results involving a sharp increase in moisture loss at the beginning of frying, was observed in a mass transfer study of breaded chicken nuggets during deep fat frying (Ngadi, Dirani et al. 2006). As the temperature of the frying medium, the oil, is higher than boiling point of water, an intense evaporation of water during frying happened, and moisture content decreased. Higher rice flour proportion in the batter formulation resulted in significantly ($p < 0.05$) less moisture content. Gluten protein, the major protein in wheat flour has shown a good film-forming ability, which makes strong elasto-plastic wheat-flour-based dough with the potential to prevent moisture loss during frying (Day, Augustin et al. 2006). In contrast, rice flour lacks gluten protein and therefore possesses a lower water binding capacity (Cao, Wen et al. 2009).

Figure 4-1 shows the experimental and predicted values of moisture content ratio during frying of batters with different wheat to rice flour combinations. Analysis of the effective moisture diffusion coefficient (D_{eff}) showed that batter prepared from rice flour alone had a significant ($P < 0.05$) higher D_{eff} than other batters; whereas no significant difference was observed in batters using wheat flour in their formulation (Table 4-2). This can be attributed to the fact that gluten in wheat flour creates a strong film which binds water and reduces moisture evaporation during frying. Values of moisture diffusivity measured for batters ranged between 2.35×10^{-6} and $2.85 \times 10^{-6} \text{ m}^2/\text{min}$, where RSME and R^2 ranged from 0.06 to 0.08, and 0.89 to 0.95,

respectively. The low RSME and high R^2 values show that the model used was appropriate for predicting D_{eff} . The moisture diffusivity of shrimp nuggets, coated with batters that were prepared from mixture of wheat and soy flours was reported to be between 1.23×10^{-6} and $3.42 \times 10^{-6} \text{ m}^2/\text{min}$ (Nasiri, Mohebbi et al. 2012), which shows approximately similar results with our findings.

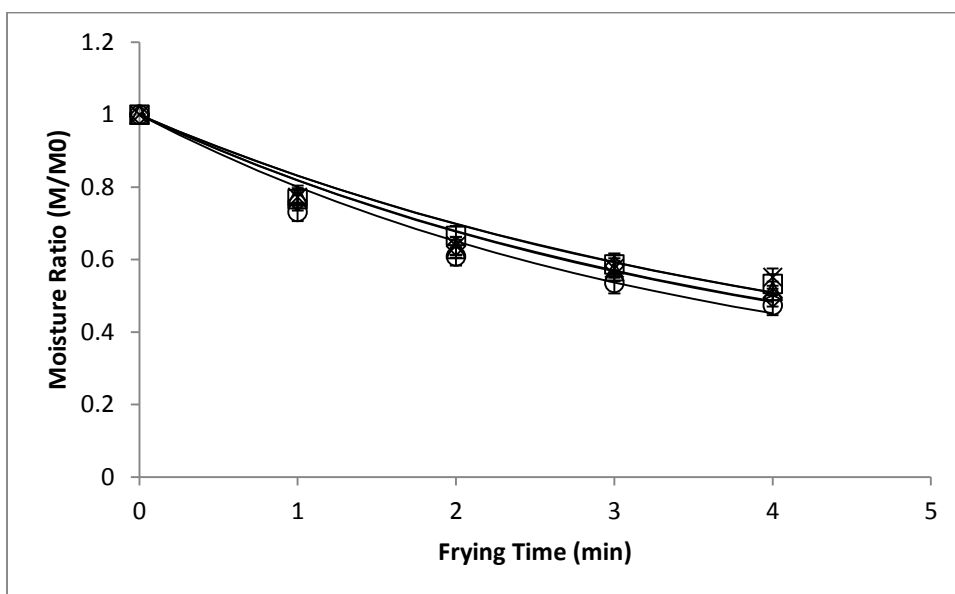


Figure 4-1 Moisture content during frying of batter coating with different formulations. ◇: experimental results for 100W0R (100 g wheat flour to 0 g rice flour), □: experimental results for 75W25R (75 g wheat flour to 25 g rice flour), △: experimental results for 50W50R (50 g wheat flour to 50 g rice flour), ×: experimental results for 25W75R (25 g wheat flour to 75 g rice flour), and ○: experimental results for 0W100R (0 g wheat flour to 100 g rice flour). Continuous line —: predicted values.

Fig. 4.2a,b,c show the graphs of surface, penetrated and total fat content, respectively, against frying time for batters with different formulations. In this study, the fried batters were immediately immersed in petroleum ether solvent; and therefore, oil penetration during cooling stage was intentionally stopped. The reason was to quantify the fractions of oil which is absorbed or not absorbed into the batter during frying. The surface temperature of the product generally reduces during the first 20 s to around 65°C (Mir-Bel, Oria et al. 2009), and then vacuum

pressure is created inside the batter; therefore to measure SF, it was necessary to conduct the oil extraction experiment in less than 20 s after taking the batters out from the fryer to have an accurate oil fraction measurement. Surface fat content ranged between 2.25 to 8.45 g/100g of the sample (this constituted 36.35 to 79.50 g/100g of the total fat content in the product) whereas penetrated fat content ranged between 1.20 to 4.26 g/100g of the sample (25.42 to 55.40 g/100g of the total fat content in the product), and total fat content ranged between 4.72 to 10.36 g/100g of the sample.

Table 4-2 Means \pm SD of effective moisture diffusion coefficient of batter coating with different formulations.

Formulation	Deff \times E-06 (m ² /min)	RMSE	R ²
100W0R	1.83 \pm 0.06 ^b	0.06	0.95
75W25R	1.69 \pm 0.08 ^b	0.06	0.94
50W50R	1.81 \pm 0.17 ^b	0.07	0.92
25W75R	1.68 \pm 0.07 ^b	0.08	0.89
0W100R	2.01 \pm 0.08 ^a	0.06	0.94

^{a,b} Means within the same column with different subscripts differ ($P < 0.05$). 100W0R: 100g wheat flour to 0g rice flour, 75W25R: 75g wheat flour to 25 g rice flour, 50W50R: 50 g wheat flour to 50 g rice flour, 25W75R: 25 g wheat flour to 75 g rice flour, and 0W100R: 0 g wheat flour to 100 g rice flour. RMSE: root mean square error. R²: the coefficient of determination.

Analysis of variance (ANOVA) showed that the interaction effect of batter formulation and frying time on variation observed in surface, penetrated, and total fat contents at a confidence interval of 95% was significant. Duncan multiple range test (MRT) at 4 min frying was performed to compare the effect of different batter formulations on fractions of fat content. Addition of rice in the batter influenced the distribution of oil fractions in the fried products.

Increasing the proportion of rice flour in batter formulation significantly ($P < 0.05$) decreased the surface and total fat contents, whereas increased the penetrated oil content. More proportion of wheat flour in the batter formulation resulted in more oil retention on the surface than in batters with less proportion of wheat flour. The result may be attributed to the gluten content of wheat flour which is highly hydrophobic with higher affinity for oil than rice flour and therefore more oil remained on the surface (Firdevs Dogan, Sahin et al. 2005).

Greater proportion of wheat flour in the batter formulation resulted in less oil penetration than batters with lower proportion of wheat flour. Wheat proteins have a strong ability in forming an elasto-plastic structure, while rice flour with relatively low protein content is typically weak in forming an elasto-plastic structure (Amboon, Tulyathan et al. 2012). The elasto-plastic property of wheat batter results in strong film with less permeability to oil absorption, apparently leading to lower oil penetration in wheat flour based batters.

It has been reported that rice-flour-based batter presents a microstructure with higher porosity compared to wheat-flour-based batter (Barutcu, Sahin et al. 2009). Porosity has a positive correlation with fat uptake (Adedeji and Ngadi 2011), and it can be another reason for the greater amount of penetrated oil for batters with higher proportion of rice flour. Indeed, the microstructure (mean pore size, connectedness, and permeability) of the crust, which is formed during frying, is a critical factor defining the amount of oil remaining on the surface or penetrating into the microstructure (Moreno, Brown et al. 2010).

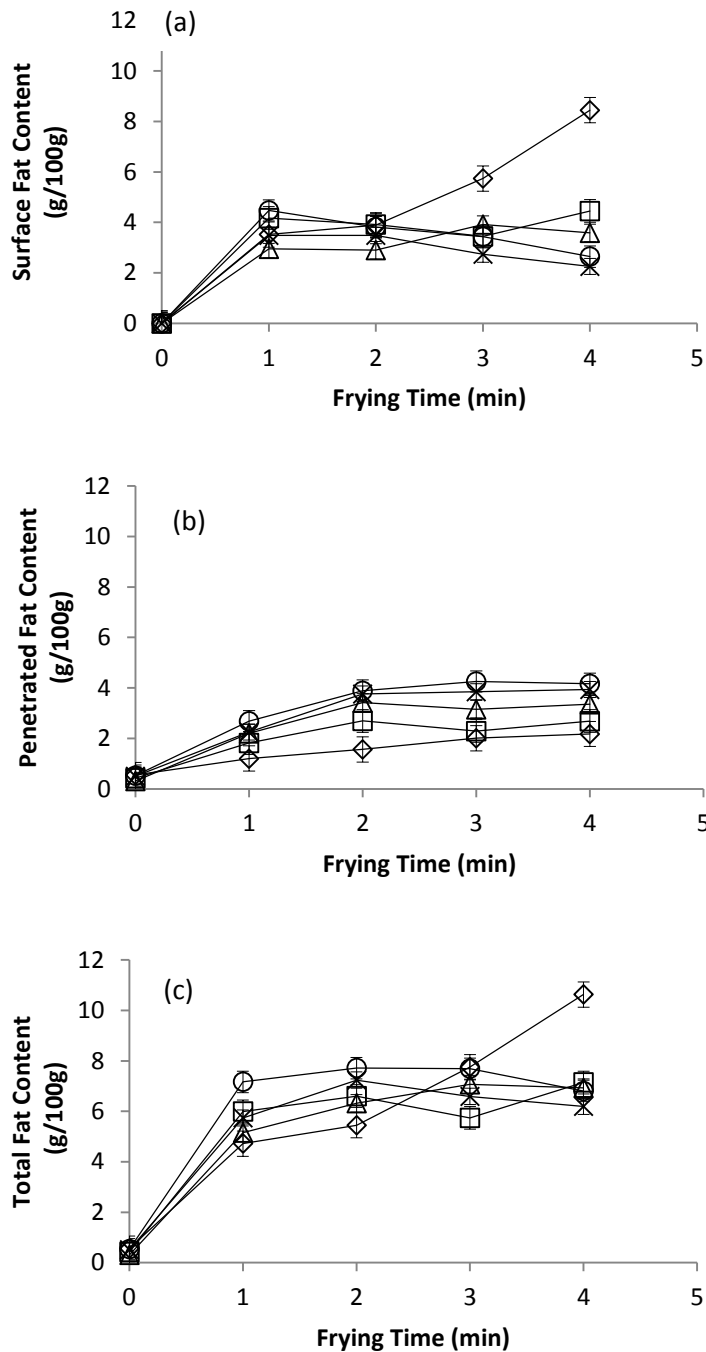


Figure 4-2 Fractions of fat during frying of batter coating with different formulations. (a): surface fat content, (b) penetrated fat content, and (c): total fat content, \diamond : 100W0R (100 g wheat flour to 0 g rice flour), \square : 75W25R (75 g wheat flour to 25g rice flour), Δ : 50W50R (50 g wheat flour to 50 g rice flour), \times : 25W75R (25 g wheat flour to 75 g rice flour), and \circ : 0W100R (0 g wheat flour to 100 g rice flour).

Similar to the results observed for surface fat content, total fat content of batters prepared with higher amount of wheat flour was significantly ($P < 0.05$) higher than batters prepared with

higher amount of rice flour. It confirms the fact that oil absorption is basically a surface-related phenomenon. Rice flour was reported to resist oil absorption more than wheat flour, and therefore showed less oil content during frying batter (Nakamura and Ohtsubo 2010). Most of the oil is absorbed during very short frying times (about 1 min). After that, the TF remained almost the same for all batters (Figure 4-2c) except for fully wheat-flour-based batter, which shows the same trend as SF (Figure 4-2a). In agreement with this result, Pedreschi, Cocio et al. (2008) found that the major part of oil was absorbed at the beginning of frying potato slices.

Mathematical models described earlier (Table 4-1) were fitted to obtain the rate of PF content of batters, as fat transfer rate, with different formulation, and statistical values were applied to define the accuracy of the models. Fat transfer rate constants were between 0.60 to 0.89 min⁻¹, 0.78 to 0.96 min⁻¹, 0.82 to 0.97 min⁻¹ and 0.66 to 0.90 min⁻¹ for Newton, Page, modified Page, and Hendeson & Pabis models, respectively (Table 4-3). Fat transfer kinetics of PF was not significantly affected by changing the amount of wheat and rice flours in the formulation of batters, whereas the moisture kinetic was affected. The reason could be attributed to the fact that PF constituted a small portion of total fat content and the variation between different formulations is not high. Mass transfer constant rates highly depends on major variables of frying process such as oil type and temperature, the product, pre-treatment applied, frying conditions, the ingredients of the batter, and constituents in the flour (Firdevs Dogan, Sahin et al. 2005, Adedeji, Ngadi et al. 2009, Troncoso and Pedreschi 2009, Nasiri, Mohebbi et al. 2012). Criteria for selection of the best model describing the kinetic rate of fat uptake for batters with different formulation were based on the highest R², and lowest RSME values. As it is presented in Table 4-3, the R² and RSME of the Page model in average were 0.96, and 0.06, respectively,

which showed that this model was the best fit to describe fat uptake change during frying of all batter formulations.

Table 4-3 Means \pm SD of kinetics of oil penetration of batter coating with different formulations.

Formulations	Models				
	Statistics	Newton	Page	Modified page	Henderson
		k(min ⁻¹)	k(min ⁻¹)/n	k(min ⁻¹)/n	a/k(min ⁻¹)
100W0R	Coefficient	0.63 \pm 0.16 ^a	0.78 \pm 0.01 ^a /1.07 \pm 0.04 ^a	0.90 \pm 0.04 ^a /0.85 \pm 0.01 ^a	0.75 \pm 0.04 ^a /1.03 \pm 0.03
	RMSE	0.08	0.05	0.09	0.09
	R ²	0.94	0.98	0.93	0.93
75W25R	Coefficient	0.70 \pm 0.02 ^a	0.82 \pm 0.08 ^a /0.90 \pm 0.04 ^a	0.82 \pm 0.01 ^a /0.76 \pm 0.03 ^a	0.72 \pm 0.01 ^a /0.98 \pm 0.01 ^a
	RMSE	0.09	0.10	0.10	0.10
	R ²	0.93	0.90	0.90	0.90
50W50R	Coefficient	0.77 \pm 0.15 ^a	0.97 \pm 0.13 ^a /0.89 \pm 0.05 ^a	0.91 \pm 0.01 ^a /0.89 \pm 0.05 ^a	0.84 \pm 0.09 ^a /1.01 \pm 0.01 ^a
	RMSE	0.08	0.06	0.09	0.09
	R ²	0.95	0.97	0.93	0.93
25W75R	Coefficient	0.60 \pm 0.04 ^a	0.96 \pm 0.01 ^a /1.21 \pm 0.01 ^a	0.85 \pm 0.01 ^a /0.70 \pm 0.05 ^a	0.66 \pm 0.01 ^a /1.01 \pm 0.01 ^a
	RMSE	0.07	0.07	0.08	0.08
	R ²	0.97	0.98	0.95	0.95
0W100R	Coefficient	0.89 \pm 0.04 ^a	0.83 \pm 0.10 ^a /1.20 \pm 0.20 ^a	0.97 \pm 0.04 ^a /0.91 \pm 0.01 ^a	0.90 \pm 0.03 ^a /1.01 \pm 0.01 ^a
	RMSE	0.03	0.03	0.04	0.04
	R ²	0.99	0.99	0.99	0.99

^{a,b} Means within the same column with different subscripts differ ($P < 0.05$). 100W0R: 100g wheat flour to 0g rice flour, 75W25R: 75g wheat flour to 25 g rice flour, 50W50R: 50 g wheat flour to 50 g rice flour, \times : 25 g wheat flour to 75 g rice flour, and 0W100R: 0 g wheat flour to 100 g rice flour. k: kinetic of oil penetration, n and a: constant numbers.

4.4.2. Pre-drying

Figure 4-3 shows the experimental and predicted values of moisture content ratio during frying of wheat-flour-based batters with different pre-drying times. Analysis of variance (ANOVA) showed that the interaction effect of pre-drying time and frying time on variations observed in moisture content at a confidence interval of 95% was not significant. Duncan multiple range test (MRT) was performed to study the effect of the main parameters, pre-drying time and frying time, on moisture content. The initial moisture content (db) for batters was 141.63, 133.83, 130.64, and 125.21 g/100g for 0MD, 15MD, 30MD, and 60MD batters, respectively, which decreased to approximately 70.10, 61.15, 56.01, and 54.98 g/100g after 4 min frying. The longer the pre-drying time, the lower the moisture content was observed.

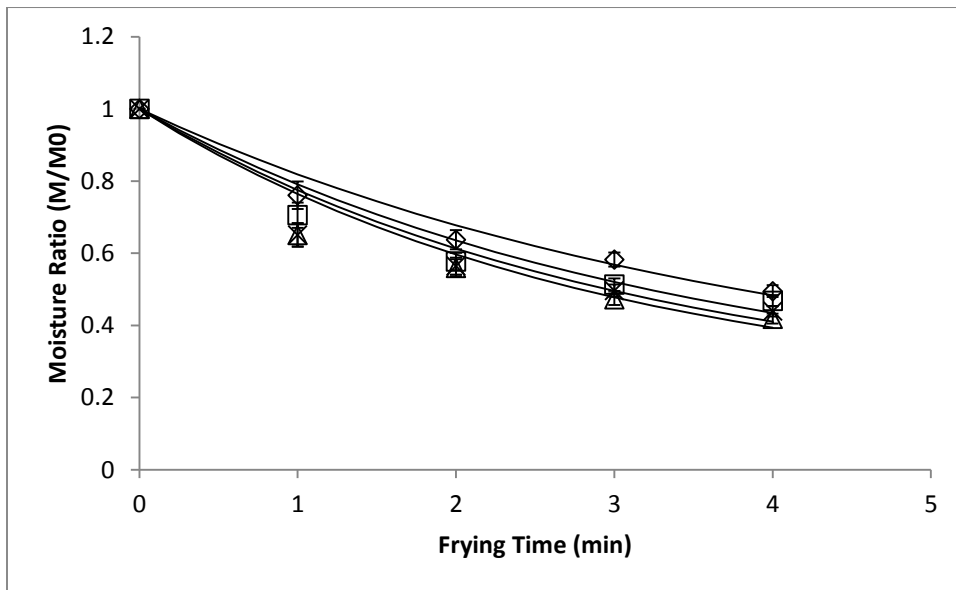


Figure 4-3 Moisture content during frying of batter coating with different pre-drying times. ◇: experimental results for 0MD (non-pre-dried batter), □: experimental results for 15MD (batter pre-dried for 15 min), Δ: experimental results for 30MD (batter pre-dried for 30 min), ×: experimental results for 60MD (batter pre-dried for 60 min). Continuous line —: predicted values.

Analysis of the effective moisture diffusion coefficient (D_{eff}) showed that pre-drying significantly ($P < 0.05$) decreased D_{eff} value of during frying (Table 4-4). Values of moisture

diffusivity measured for batters ranged between 2.01×10^{-6} and 2.57×10^{-6} m²/min, where RSME and R² and ranged from 0.06 to 0.07, and 0.93 to 0.95, respectively. The model shows a good fit as indicated by the low RSME and high R² values.

Table 4-4 Means \pm SD of effective moisture diffusion coefficient of batter coating with different pre-drying times.

Pre-drying time	Deff \times E-06 (m ² /s)	RMSE	R ²
0MD	1.83 \pm 0.06 ^a	0.06	0.95
15MD	1.77 \pm 0.05 ^{ab}	0.07	0.93
30MD	1.65 \pm 0.10 ^{ab}	0.06	0.95
60MD	1.49 \pm 0.08 ^c	0.07	0.93

^{a,b} Means within the same column with different subscripts differ ($P < 0.05$). 0MD: non-pre-dried batter, 15MD: batter pre-dried for 15 min, 30MD: batter pre-dried for 30 min, and 60MD: batter pre-dried for 60 min. R²: the coefficient of determination.

Figure 4-4a,b,c show the graphs of surface, penetrated and total fat content, respectively, against frying time for batters with different pre-drying times. Surface fat content ranged between 2.66 to 8.45 g/100g of the batter sample (this constituted 51.55 to 72.47 g/100g of the total fat content in the product) whereas penetrated fat content ranged between 1.98 to 4.14 g/100g of the batter sample (35.51 to 38.38 g/100g of the total fat content in the product), and total fat content ranged between 5.16 to 11.66 g/100g. Pre-drying, resulting in different initial moisture contents, showed a significant ($P < 0.05$) effect on oil fraction contents. Analysis of variance (ANOVA) showed that the interaction effect of batter formulation and frying time on variation observed in surface and total fat contents at a confidence interval of 95% was significant, whereas it was not significant ($P < 0.05$) for penetrated fat content. Duncan multiple

range test (MRT) at 4 min was performed to compare the effect of different batter formulations on fractions of fat content.

Although dried batters showed a significant ($P < 0.05$) lower surface and total fat content than non-dried batter, no significant difference was observed between surface and total fat contents of pre-dried batters at different times. Penetrated fat content was not influenced by pre-drying. Durán, Pedreschi et al. (2007) studied oil partition in pre-treated potato slices, and in agreement with our result they reported that nearly 62 g/100g of the total oil content remained in the product surface, and about 38 g/100g of it was absorbed during frying. When fried samples are removed from the oil medium, a higher temperature difference develops between the surface and the interior, which in turn causes a higher negative pressure in the pore space leading to more oil penetration into their microstructure during cooling stage (Pedreschi, Cocio et al. 2008). Pre-drying causes shrinkage, which decreases the volume of open pores, creates a compact matrix, and increases the solid content (Debnath, Bhat et al. 2003, Pedreschi and Moyano 2005, Van Loon, Visser et al. 2007). Consequently, these changes in microstructure led to a reduction in total oil content. Consistent with these results, Debnath, Bhat et al. (2003) and Pedreschi and Moyano (2005) reported that moisture content of fried products decreased and oil content increased during frying.

Table 4-5 describes the fitted mathematical models and their accuracy in obtaining the rate of penetrated fat of batters with different pre-drying times. Fat transfer rate constants were between 0.63 to 0.99 min⁻¹, 0.78 to 0.96 min⁻¹, 0.85 to 1.01 min⁻¹ and 0.62 to 0.99 min⁻¹ for Newton, Page, modified Page, and Hendeson & Pabis models, respectively. Fat transfer kinetic obtained by all models was not significantly affected by pre-drying time. As it is presented in Table 4-5, the R^2

and RSME were on average 0.99, and 0.04, respectively, for Page model, which showed that this model was the best fit to describe fat uptake change during frying of all pre-drying times.

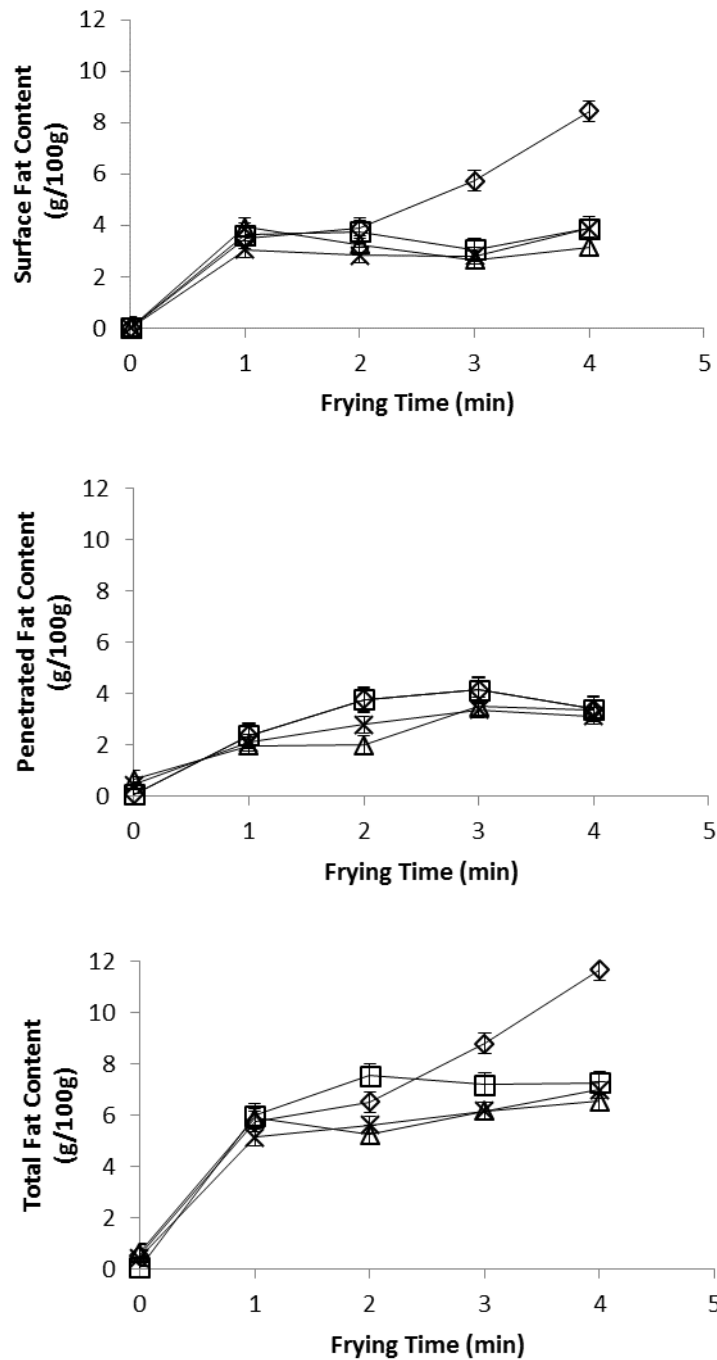


Figure 4-4 Fractions of fat during frying of batter coating with different pre-drying times. \diamond : 0MD (non-pre-dried batter), \square : 15MD (batter pre-dried for 15 min), Δ : 30MD (batter pre-dried for 30 min), \times : 60MD (batter pre-dried for 60 min).

Table 4-5 Means \pm SD of kinetics of oil penetration of batter coating with different pre-drying times.

Formulations	Models				
	Statistics	Newton	Page	Modified Page	Henderson
		$k(\text{min}^{-1})$	$k(\text{min}^{-1})/n$	$k(\text{min}^{-1})/n$	$a/k(\text{min}^{-1})$
0MD	Coefficient	0.63 ± 0.16^a	$0.78 \pm 0.01^a / 1.07 \pm 0.04^a$	$0.90 \pm 0.04^a / 0.85 \pm 0.01^a$	$0.75 \pm 0.04^a / 1.03 \pm 0.03$
	RMSE	0.08	0.05	0.09	0.09
	R ²	0.94	0.98	0.93	0.93
15MD	Coefficient	0.85 ± 0.02^a	$0.82 \pm 0.01^a / 1.06 \pm 0.02^a$	$0.91 \pm 0.01^a / 0.93 \pm 0.02^a$	$0.85 \pm 0.02^a / 1.00 \pm 0.01^a$
	RMSE	0.04	0.04	0.04	0.04
	R ²	0.99	0.99	0.99	0.99
30MD	Coefficient	0.74 ± 0.08^a	$0.86 \pm 0.01^a / 1.02 \pm 0.05^a$	$0.85 \pm 0.02^a / 0.70 \pm 0.09^a$	$0.62 \pm 0.15^a / 1.06 \pm 0.03^a$
	RMSE	0.11	0.03	0.13	0.12
	R ²	0.92	0.99	0.92	0.92
60MD	Coefficient	0.99 ± 0.09^a	$0.96 \pm 0.06^a / 1.20 \pm 0.01^a$	$1.01 \pm 0.10^a / 0.96 \pm 0.11^a$	$0.99 \pm 0.13^a / 1.01 \pm 0.02^a$
	RMSE	0.08	0.05	0.09	0.09
	R ²	0.94	0.99	0.93	0.93

^{a,b} Means within the same column with different subscripts differ ($P < 0.05$). 0MD: non-pre-dried batter, 15MD: batter pre-dried for 15 min, 30MD: batter pre-dried for 30 min, and 60MD: batter pre-dried for 60 min. k: kinetic of oil penetration, n and a: constant numbers.

4.5. Conclusion

A good knowledge of the mechanisms involved during frying process is essential to control oil absorption. In this study the total oil content, oil adhesion on the surface and oil transfer to the inner structure of fried batters with different wheat to rice flour ratios and pre-drying times were measured. Frying time and pre-drying had significant ($P < 0.05$) effect on moisture loss of batter

coating during frying. Frying time, batter formulation, and pre-drying had significant ($P < 0.05$) effect on oil uptake of batter coating. The higher the frying time, the higher the moisture loss and oil content was reported. Higher proportion of wheat flour showed higher surface and total oil content, and lower penetrated oil content. Batter without pre-drying had higher surface, penetrated and total oil content than batters that had undergone pre-drying. Moisture diffusivity and kinetics of oil penetration during frying were evaluated using different mathematical models. The Fick's diffusion equation, Page, and modified Page models adequately predicted these parameters in batters with different formulations and pre-dried batters during frying.

CONNECTING TEXT

As was seen, results of effect of batter formulation on fat content were contradicted in chapters 3 and 4. In chapter 3, higher amount of wheat flour in the formulation of batter decreased total fat content of fried batter, whereas in chapter 4, higher amount of wheat flour in the formulation of batter showed higher fat content. In the work reported in chapter 3, the surface of the fried batters was mopped off using clean tissue papers, while the surface of fried batters was not mopped off in the work reported in chapter 4. Therefore, study of microstructure and surface of fried batter were found to be very important in defining the mechanisms of oil absorption. In chapter 5, the inter-particle space fractions of fried batters, as important parameters influencing fat uptake, were measured.

CHAPTER 5

INTER-PARTICLE SPACE FRACTIONS IN FRIED BATTER COATINGS AS INFLUENCED BY BATTER FORMULATION AND PRE-DRYING TIME

5.1. Abstract

Evaluation the different fractions of spaces in a fried product that are occupied by air, moisture, or fat provides useful information for researchers and manufacturers to better understand the mechanisms of oil absorption during frying. The objective of this study was to determine the influence of batter formulations and pre-drying on the fraction of inter-particle spaces in batter coating. Pre-drying decreased the amount of fat to 2.97 g/100 g compared to non-pre-dried samples which showed 4.37 g/100 g fat content. Higher rice flour content in batter significantly increased the fraction of inter-particle spaces occupied by air (SOA), and decreased the fraction of spaces occupied by moisture (SOM) and fat (SOF). Longer pre-drying times showed greater fraction of spaces occupied by air, and smaller fraction of spaces occupied by moisture and fat. SOA, SOM, and SOF of the batter systems ranged from 2.20 to 46.03%, 2.55 to 47.07%, and 0.35 to 11.11%, respectively. Using higher amount of wheat flour in batter and longer pre-drying time showed to significantly decrease fat uptake during frying.

5.2. Introduction

The study of the microstructure of fried foods is very important in quality characterization, development, process modeling and optimization of the products. The structure of fried foods highly depends on their initial physico-chemical characteristics, oil properties, pre-treatment, and frying conditions such as temperature and time. During frying, some microstructural changes occur as a result of starch gelatinization and protein denaturation at the high frying temperatures.

The effect of microstructural changes on mass transfer during frying has been studied by several authors (Moreira, Castell-Perez et al. 1999, Saguy and Dana 2003, Dana and Saguy 2006), and it has generally been established that rapid water evaporation and consequently crust formation occur when food is exposed to high frying temperature. High internal pressure, which is generated due to steam, creates cracks, defects, open capillaries and channels in the cellular structures and membranes of the food material (Saguy and Dana 2003, Dana and Saguy 2006). After frying and during the cooling phase, water vapor condensation and subsequently pressure drop occur in some products. Consequently, oil which is adhered to the surface of the product is sucked into the large voids and crevices due to a generated vacuum effect (Moreira, Castell-Perez et al. 1999). It was reported that oil absorption during cooling phase is a pressure-driven flow and involves a balance between the capillary forces and oil drainage (Ziaiifar, Courtois et al. 2010). Thus, surface microstructure plays a critical role in oil uptake and also in the distribution of oil, water and air through the network of pores and voids during frying (Kassama and Ngadi 2004, Kassama and Ngadi 2005). Kassama and Ngadi (2004) reported three possible types of pores namely interconnected pores (accessible from many directions), isolated pores (inaccessible) and non-connected pores (accessible from one direction). Interconnected pores play a more important role in transport of fluid through the porous medium than the non-connected pores, while isolated pores have limited influence on transport phenomena. Interconnected pores and non-connected pores are the reservoirs of moisture and oil in fried products, and the distribution of oil, moisture, and even air through these spaces changes during the frying process.

The measurement of inter-particle spaces occupied by oil, moisture, or air is therefore necessary for studying the mechanisms of oil uptake and moisture loss during frying.

Different techniques such as decreasing the initial moisture content by pre-drying the product, surface coating and using different ingredients as the coating elements, and pre-cooking in oven or microwave have been used to reduce fat content of fried foods (Mellema 2003, Adedeji, Ngadi et al. 2009, De Grandi Castro Freitas, Berbari et al. 2009, Ali, Abdel-Razek et al. 2012, Lalam, Sandhu et al. 2013). Pre-drying and thus reduced initial moisture content of raw food materials results in less oil uptake during frying (Gupta, Shivhare et al. 2000). It was reported (Gupta, Shivhare et al. 2000) that pre-dried potatoes absorbed less oil compared to non-pre-dried samples. Moyano and Pedreschi (2006) similarly reported that pre-dried potato slices had significantly less oil content than control slices. Possible reasons for decreasing oil uptake during frying, because of pre-drying, include the creation of a compact material matrix, which increases in inner pressure, or increase in solid content of pre-dried material (Debnath, Bhat et al. 2003).

Batter and breading are complex systems prepared via mixing different ingredients such as flour, starch, hydrocolloids, salt, bread crumbs, seasoning and water. The final quality of products is determined by the interaction of these ingredients. Applying heat on batter and breading during frying creates a network structure (Adedeji, Liu et al. 2011) resulting as crust of the fried product. It provides a brittle texture, and makes a barrier to prevent moisture loss and consequently oil uptake (Nasiri, Mohebbi et al. 2012). Although wheat flour is easily available in most regions and is the most common flour used in batter systems (Xue & Ngadi, 2007), rice flour is used as a healthier alternative providing fewer calories (Barutcu, Sahin et al. 2009). There is lack of information in literature about the effects of initial moisture content and replacing different ratios of wheat flour by rice flour on microstructure of fried batter systems and consequently the oil uptake of batter coated fried foods. The objectives of this study were to determine the influence of batter formulations and initial moisture content on fat uptake, the

space occupied by air, moisture, and fat, and finally the fractions of inter-particle spaces in batter coating.

5.3. Materials and methods

5.3.1. Materials

Wheat flour (Five Rose All Purpose Flour, Les Cuisines Five Roses Kitchens, QC, Canada) was purchased from a local grocery store in Montreal, Canada. Long grain rice flour, RL-100, was prepared by Rivland Partnership (Riceland Foods, Arizona, USA). Soy protein isolated (SPI) was supplied from MP Biomedicals, LLC, Parc d'innovation, BP 50067, Illkirch, France. Wheat Gluten (WG) was received from Sigma-Aldrich Co. LLC, Ontario, Canada. Carboxymethyl cellulose (CMC) used was supplied by TIC Gums Inc., Maryland, USA. Canola oil (Les Essentiels De La Cuisine, Richmond, BC, Canada) was supplied by Food & Dining Service, McGill University.

5.3.2. Core part and coating layer preparation

In this study a meat analog was used as the core part of the product and then it was coated by batters with different formulations. The meat analog (as core part) was prepared by mixing soy protein isolate (22 g/100 g), wheat gluten (3 g/100 g), canola oil (6 g/100 g), and distilled water (69 g/100 g). Wheat gluten was first properly mixed with oil in a mixer (Sumeet, CM/L – 0973467, Indai). Then while mixing, distilled water was added to the mixture, and finally soy protein isolate was gradually added. The mixture was then filled in rectangular aluminum cells (50×25×5 mm³) and kept at 80°C for 10 min in a water bath (Lauda Dr. R. Wobser, GMBH&CO. Klasse. Konigshofen, Germany) to gelatinize. The gelatinized product was then covered with different batters of 5 mm thickness as the coating layer.

Five different ratios of wheat and rice flours were prepared namely: 100 g/100 g wheat flour to 0 g/100 g rice flour (**100W0R**), 75 g/100 g wheat flour to 25 g/100 g rice flour (**75W25R**), 50 g/100 g wheat flour to 50 g/100 g rice flour (**50W50R**), 25 g/100 g wheat flour to 75 g/100 g rice flour (**25W75R**), and 0 g/100 g wheat flour to 100 g/100 g rice flour (**0W100R**). A fixed amount of salt (NaCl) and CMC were added at 2.5 and 1.5 g/100 g, respectively. Batter slurry was prepared by adding distilled water to flour mix in the ratio of 1.3:1. Then, the batter system was thoroughly mixed.

To study the influence of pre-drying time, in another set of batter systems, four batches of 100 g/100 g of wheat flour were mixed with NaCl, CMC, and distilled water to obtain 65 g/100 g wb initial moisture content. The first batch which was fried without pre-drying was named as **0MD**. The three other batches, which were named **30MD**, **60MD**, and **90MD**, were pre-dried for 30 min, 60 min and 90 min, respectively, in an oven dryer (Isotemp 700, Fischer Scientific, Pittsburgh, PA) at 60°C.

5.3.3. Frying

A kitchen programmable deep fat fryer (De'Longhi, America Inc., Saddle Brooke, NJ 076663, China) was used to fry the samples at $180 \pm 2^\circ\text{C}$ for 1, 2, 3, and 4 min. The fryer was filled with 1.5 L fresh canola oil and preheated and maintained at $180 \pm 2^\circ\text{C}$ for 2 hrs before frying. To minimize the variation of oil properties due to degradation during frying, each batch of oil was used for only 30 min before it was replaced with a new batch of oil. All experiments were performed in triplicate.

5.3.4. Moisture content

The fried batter (coating layer) was carefully separated from the core part and freeze-dried in a freeze dryer (Modulyod-115; ThermoSavant, Holbrook, NY, USA) at -50°C and 100 mbar for 36

h and were allowed to equilibrate in a desiccator for 30 min. Weight of batter before and after freeze-drying were measured and used to calculate the moisture content on dry weight basis (db).

5.3.5. Fat Content

Each individual freeze-dried batter (weight: 3-5 g) was placed in thimbles in a VELP SER 148 (Velp Scientifica, Usmate, Italy) solvent extraction unit and oil was extracted with petroleum ether. Oil content was computed by dividing the mass of extracted oil with the mass of freeze-dried sample.

5.3.6. Apparent Density

To obtain inter-particle spaces occupied by air, moisture, and fat, it was first required to measure the apparent density of the samples at three different steps: 1) after frying 2) after de-watering 3) after de-fatting.

To measure apparent density after frying (ρ_{al}), fried batter samples were kept for 30 min at room temperature to cool down and their apparent density was measured according to the method described by Adedeji and Ngadi (2011). Apparent volume of the weighed batters was first measured in a helium pycnometer (Model 1305 Multivolume, Micromeritics Instrument Corporation, Norcross, GA).

Each sample was placed in the 35 cm³ sample chamber of the pycnometer and was subjected to cyclic action (purging) by pressurizing and depressurizing with helium gas prior to analysis in order to remove all the air and vapor trapped in the pores and crevices. The analysis was carried out at ambient temperature with pressure of up to 134.45 kpa. The system's valves were closed initially to allow equilibration to atmospheric pressure. Then the valve that leads to the sample chamber was opened to allow helium gas to enter up to 134.45 ± 1.38 kpa, and the

valve was closed for between 15 to 30 s to allow the gas to penetrate the sample thoroughly before pressure (P_1) reading was made. The second valve that leads to the expansion chamber from the sample chamber was then opened to allow trapped helium gas to flow in and pressure (P_2) reading was also taken after equilibration for 15 – 30 s. To determine the sample volume, the following equation was used.

$$V_{sp} = V_c - \frac{V_r}{[(\frac{P_1}{P_2}) - 1]} \quad \text{Equation (5-1)}$$

where V_{sp} is the volume of the sample; V_c is volume of the sample cell with the empty sample cup in place; V_r is the volume of the expansion chamber; P_1 is sample chamber initial pressure with the expansion chamber valve closed; and P_2 is the final chamber pressure with the expansion chamber valve open. Apparent density was computed by dividing the apparent volume (including closed pores inaccessible by helium gas) of the sample with the mass.

Detailed procedure is provided in the standard protocol manual of Micromeritics (Micromeritics, 1992). The analysis was conducted at ambient temperature. All treatments were applied in triplicate and each sample was measured thrice.

To measure apparent density after de-watering (ρ_{a2}), the same set of samples used at the first step were freeze-dried to remove their moisture and then the same procedure as described earlier was applied to measure apparent density.

In order to measure apparent density after-defatting (ρ_{a3}), the same set of samples used at the first and second steps were de-fatted using the method explained in fat content measurement section to remove their fat, and then the same procedure as described earlier was applied to measure apparent density.

5.3.7. Bulk density

The same set of samples used for apparent density measurements (samples after de-watering and de-fating) were weighted and then quickly dipped in a melted paraffin wax in order to cover the surface openings of the samples and then allowed to cool at room temperature. The difference between weight of batters before and after covering by paraffin wax was taken as the mass of paraffin wax. Knowing the mass and density of paraffin, the volume of the paraffin coating layer was measured. Samples were then dropped into a water displacement pycnometer and the displaced volume was recorded as the volume of the sample coated with paraffin. The difference between volume of paraffin wax and volume of the sample coated with paraffin wax was used as the bulk volume of batters. Bulk density was determined as the ratio of the mass of sample divided by the bulk volume.

5.3.8. Inter-particle space occupied by air (SOA)

Inter-particle space occupied by air (SOA) or porosity (ϵ) was calculated as the ratio of bulk density, ρ_b , to apparent density after frying, ρ_{a1} , subtract from one, i.e. a ratio of the void/space volume present in the sample after frying to its overall volume:

$$SOA = 1 - \left(\frac{\rho_b}{\rho_{a1}} \right) \quad \text{Equation (5-2)}$$

5.3.9. Inter-particle space occupied by moisture (SOM)

The ratio of bulk density of the sample, ρ_b , to apparent density after de-watering, ρ_{a2} , subtracted from one (i.e. a ratio of the void/space volume present in the sample after de-watering to its overall volume) is the inter-particle spaces occupied by air and moisture. Inter-particle space occupied by moisture (SOM) is calculated following Equation (5-3).

$$SOM = (1 - (\frac{\rho_b}{\rho_{a2}})) - SOA \quad \text{Equation (5-3)}$$

5.3.10. Inter-particle space occupied by fat (SOF)

The ratio of bulk density of the sample, ρ_b , to apparent density after de-fating, ρ_{a3} , subtracted from one (i.e. a ratio of the void/space volume present in the sample after de- fatting to its overall volume) is the inter-particle spaces occupied by air, moisture, and fat. Inter-particle space occupied by fat (SOF) is calculated following Equation (5-4).

$$SOF = (1 - (\frac{\rho_b}{\rho_{a3}})) - SOA - SOM \quad \text{Equation (5-4)}$$

5.3.11. Statistical Analysis

All treatments were applied in triplicates. Analysis of variance was performed using SAS system software (Version 9.3, SAS Institute, Inc., Cary, NC, USA) to determine the effect of formulation and initial moisture content on moisture and fat content of the samples. Duncan multiple range test was used for mean separation at $P < 0.05$ where treatment effect was significant. The “PROC Corr” of SAS software was used for finding the Pearson’s coefficient of correlation (r) between two dependent variables. The degree of correlation was described as follows (Agbisit, Alavi et al. 2007): ($|r| < 0.20$, negligible; $|r| = 0.20-0.40$, low; $|r| = 0.40-0.60$, moderate; $|r| = 0.60-0.80$, marked; and $|r| > 0.80$, high).

5.4. Results and Discussion

5.4.1. Moisture loss and fat uptake

Figures 5-1a and 5-2a illustrate the moisture content profiles during frying of batter coatings of different formulations and different pre-drying times, respectively. The moisture content profiles during frying showed typical characteristic trends as were reported previously for deep fat fried products (Gupta, Shivhare et al. 2000, Debnath, Bhat et al. 2003, Ngadi, Dirani et al. 2006,

Adedeji, Ngadi et al. 2009). The main effects of frying time, pre-drying time, and formulation were significant ($P < 0.05$) on moisture content. As expected, the moisture content decreased during frying for all pre-drying time and formulation treatments. Higher wheat flour proportion in the formulation of batter coating showed higher moisture content. Variation in protein, gluten, and starch contents with varying wheat and rice flours (Veraverbeke and Delcour 2002, Torbica, Hadnadev et al. 2012) could be the possible reason for this different behavior. The main protein in wheat flour is gluten, while rice is known as a gluten free substitute (Veraverbeke and Delcour 2002). In contrast to wheat flour, which shows a high water holding ability because of gluten protein, rice protein possesses a low water binding capacity (Cao, Wen et al. 2009) and lacks the ability to form the necessary network for holding moisture. Figure 5-2a shows that longer pre-drying time resulted in lower moisture content before frying started; and therefore lower amount of free moisture is available for removal during frying. Similar to results reported by Ngadi, Dirani et al. (2006), there was a rapid decrease in moisture content for the first 2 min of frying followed by constant rate moisture loss, which indicates rapid moisture evaporation at the beginning of frying.

Figures 5-1b and 5-2b show oil content profiles during frying of batter coating at different formulations and different pre-drying times, respectively. ANOVA test showed that the main factors of pre-drying time, formulation, and frying time significantly ($P < 0.05$) influenced the variation observed in fat content. In agreement with Adedeji and Ngadi (2011), batter formulated with 100 g/100 g wheat flour had the lowest fat content. Gluten protein in wheat flour forms a film during heating that acts as a strong barrier against fat absorption (Yuno-Ohta, Yamada et al. 2009).

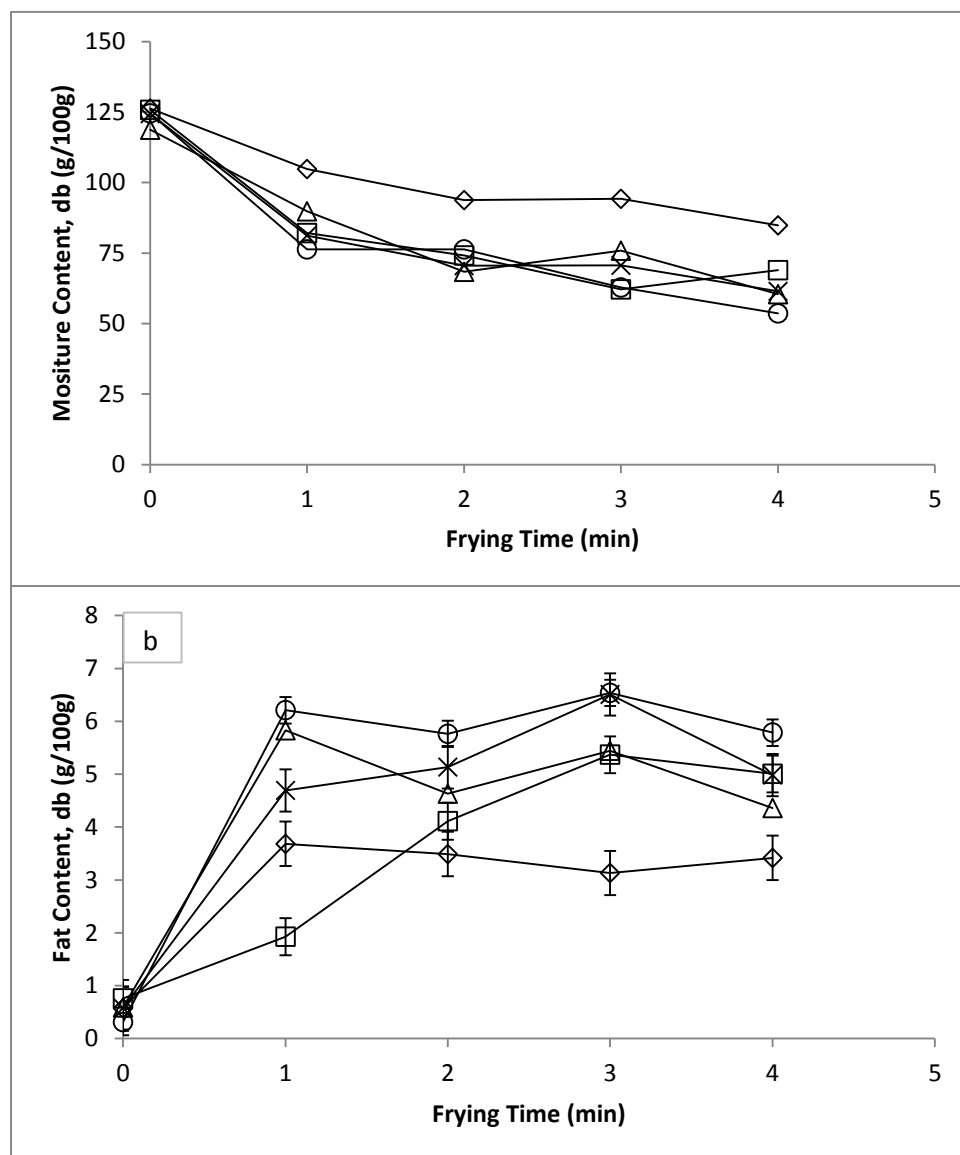


Figure 5-1 Moisture content (a) and fat content (b) during frying of batter coating with different formulations. ◇: 100W0R (100 g wheat flour to 0 g rice flour), □: 75W25R (75 g wheat flour to 25 g rice flour), △: 50W50R (50 g wheat flour to 50 g rice flour), ×: 25W75R (25 g wheat flour to 75 g rice flour), and ○: 0W100R (0 g wheat flour to 100 g rice flour).

The fat content in batters pre-dried for 90 min was significantly ($P < 0.05$) lower than other batters, while there was no significant difference between fat content of 0 min pre-dried batters and batters pre-dried for 30 and 60 min. Non-pre-dried batters, 30 min and 60 min pre-dried batters showed fat contents of 4.37, 4.29, and 4.06 g/100 g, respectively, while it was 2.97 g/100 g for 90 min pre-dried samples.

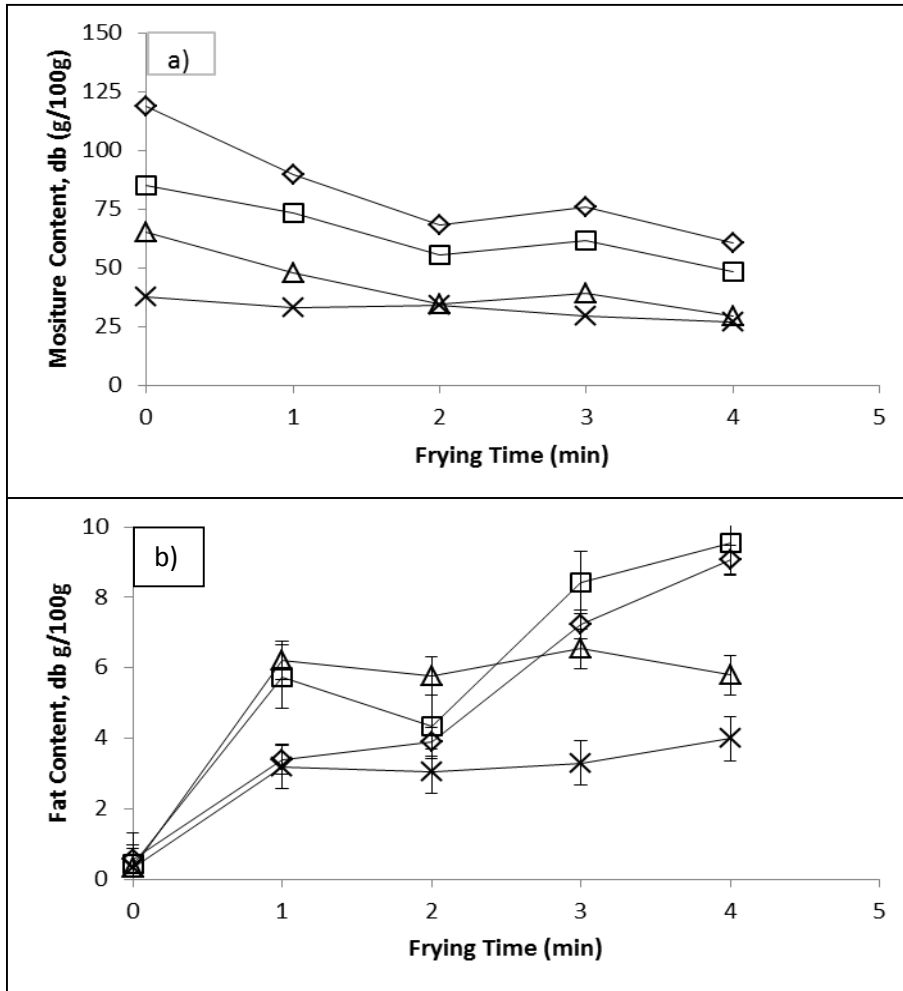


Figure 5-2 Moisture content (a) and fat content (b) during frying of 100W0R batter coating with different pre-drying times. ◇: 0MD (0 min pre-dried batter), □: 30MD (30 min pre-dried batter), Δ: 60MD (60 min pre-dried batter), and ×: 90MD (90 min pre-dried batter).

5.4.2. Inter-particle space occupied by air (SOA)

Inter-particle space occupied by air (SOA), which is also considered as porosity, of foods depends on batter composition, and the amount of water evaporated during frying. Analysis of variance (ANOVA) showed that porosity of fried batter was significantly ($P < 0.05$) influenced by the main parameters pre-drying time, batter formulation and frying time. There was no interaction effect of drying time and formulation and also frying time and formulation on variation observed in porosity at a confidence interval of 95%; however, interaction effect of drying time and frying time at the same confidence interval ($P < 0.05$) was significant, which

means the effect of batter formulation on porosity is less critical than the other two main parameters, i.e. drying time and frying time.

The porosity, SOA, of batters with different formulation at the function of frying time is shown in Figure 5-3a. Porosity ranges for 100W0R, 75W25R, 50W50R, 25W75R, and 0W100R were 16.22 - 34.80%, 19.95 - 32.56%, 21.97 - 36.55%, 18.70 - 42.54, and 20.13 -46.03 %, respectively. Increasing the amount of wheat flour in formulation of batter significantly ($P < 0.05$) decreased porosity. Lower porosity for batter formulated with more wheat flour could be attributed to its higher protein content than rice flour. Protein in wheat flour creates a strong gel with lower porosity.

Porosity, SOA, versus moisture content of batter coatings with different formulations is plotted in Figure 5-3b. Pearson's correlation showed that moisture content had a high negative correlation, -0.84, with porosity. This result shows that during frying, when moisture evaporates, some structural changes undergo in the batter and porosity increases. Kassama and Ngadi (2005) and Adediji and Ngadi (2011) similarly reported a negative correlation between moisture content and porosity using a simple linear model.

The plot of porosity versus fat content of batter coatings with different formulations is illustrated in Figure 5-3c. Pearson's correlation showed that fat content had a positive correlation with porosity. Kassama and Ngadi (2005), Adediji, Ngadi et al. (2009), and Adediji and Ngadi (2011) similarly reported a positive correlation between fat content and porosity. The positive correlation between fat content and porosity implies that higher porosity can cause more oil uptake.

Figure 5-4 shows the porosity versus frying time of batter coatings with different pre-drying times. Porosity range for the 0 min pre-dried fried batter was 2.31- 46.43%, which is very close to the range reported by Adedeji and Ngadi (2011), 2.20 - 47.90%. However, the porosity range for samples pre-dried for 30 min was between 14.43 and 49.46%. For samples pre-dried for 60 and 90 min, those ranges were 21.25 – 43.70% and 20 - 59.60%, respectively. The longer pre-drying time, and therefore more water removal during drying, results in evolution of significantly ($P<0.05$) more inter-particle spaces occupied by air. During drying of foods, the liquid water is evaporated and leaves the surface of the product as vapor. This vapor can distort the structure of the product and create cracks and holes. The process of pore formation during drying is very complex, and in addition to water content, it also depends on the processes of shrinkage and collapse during drying, surface tension, and environment pressure (Hussain, Shafiur Rahman et al. 2002). The degrees of correlation for 0MD, 30MD, 60MD, and 90MD were 0.83, 0.71, 0.62, and 0.59, respectively, which varies from high to moderate.

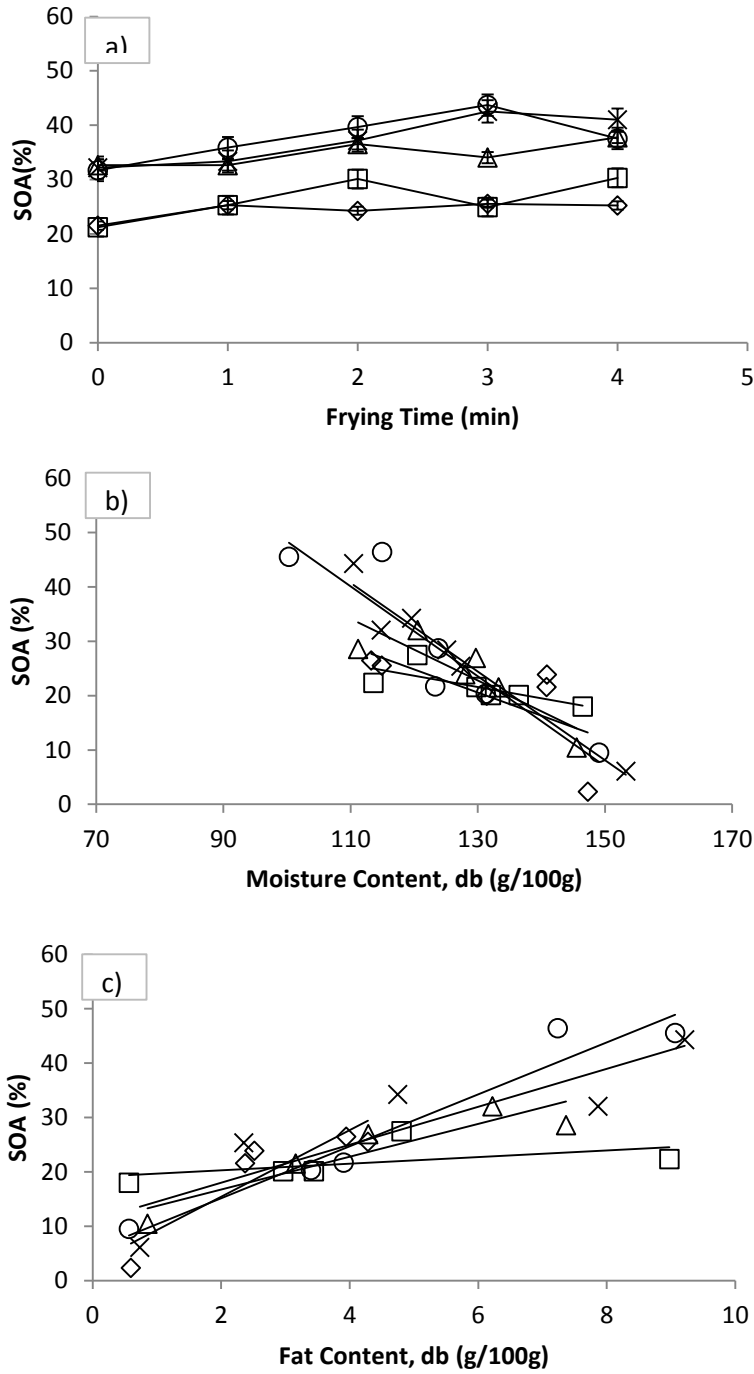


Figure 5-3 Inter-particle space occupied by air (SOA) during frying of batter coating with different formulations (a), SOA vs. moisture content (b), and SOA vs. fat content (c). ◇: 100W0R (100 g wheat flour to 0 g rice flour), □: 75W25R (75 g wheat flour to 25 g rice flour), △: 50W50R (50 g wheat flour to 50 g rice flour), ×: 25W75R (25 g wheat flour to 75 g rice flour), and ○: 0W100R (0 g wheat flour to 100 g rice flour).

There was a general increase in porosity with frying time for every pre-drying time and batter formulation. Porosity gradually increased for the first 3 min of frying, and then remained relatively constant. Adedeji and Ngadi (2011) similarly reported that porosity of fried batter increased for 60 – 120 s frying, and beyond this period it remained constant. Results were also in agreement with those reported by Taiwo and Baik (2007) and Krokida, Oreopoulou et al. (2000). This can be attributed to more oil absorption for longer frying times which fills the created air pores (Firdevs Dogan, Sahin et al. 2005). Kawas and Moreira (2001) reported an increase in porosity of tortilla chips during frying. During frying, water evaporates and leaves the surface of fried foods in the form of vapor, however due to restrictive intercellular diffusion; a part of the vapor may remain trapped within the pores. The vapor in the closed space can expand and become superheated, which may destroy the pore walls and contribute to total porosity (Taiwo and Baik 2007).

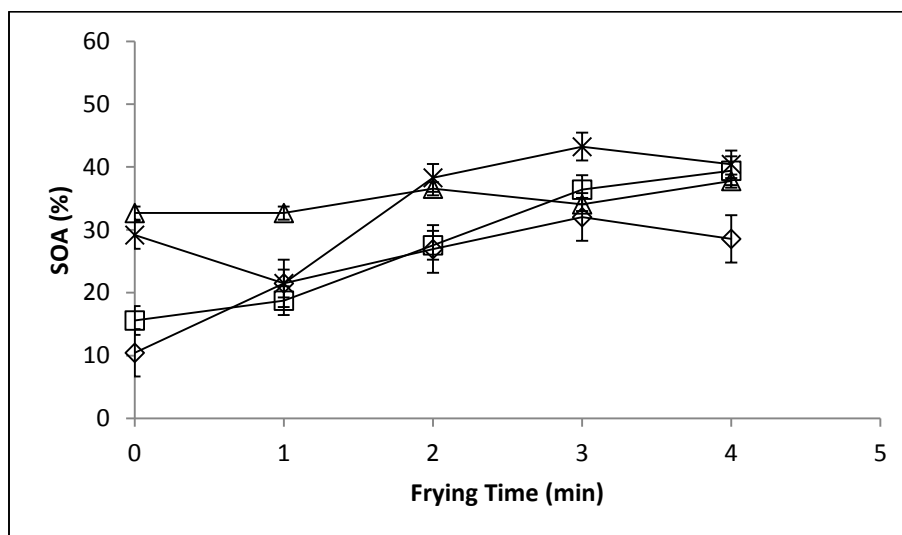


Figure 5-4 Inter-particle space occupied by air (SOA) during frying of 100W0R batter coating with different pre-drying times. \diamond : 0MD (0 min pre-dried batter), \square : 30MD (30 min pre-dried batter), Δ : 60MD (60 min pre-dried batter), and \times : 90MD (90 min pre-dried batter).

5.4.3. Inter-particle space occupied by moisture (SOM)

As stated earlier, water removal during frying causes texture disruption and generation of spaces. Some of these spaces are occupied by air while some remain filled with moisture, and some are filled with oil. The plots of SOM at a function of frying time at different batter formulations and pre-drying times are shown in Figures 5-5a and 5-6. Analysis of variance (ANOVA) showed that SOM of fried batter was significantly ($P < 0.05$) influenced by the main parameters pre-drying time, batter formulation and frying time. There was no interaction effect of drying time and formulation at a confidence interval of 95%. However, the interaction effect between drying time and frying time, and also drying time and formulation at the confidence interval of 95% was significant.

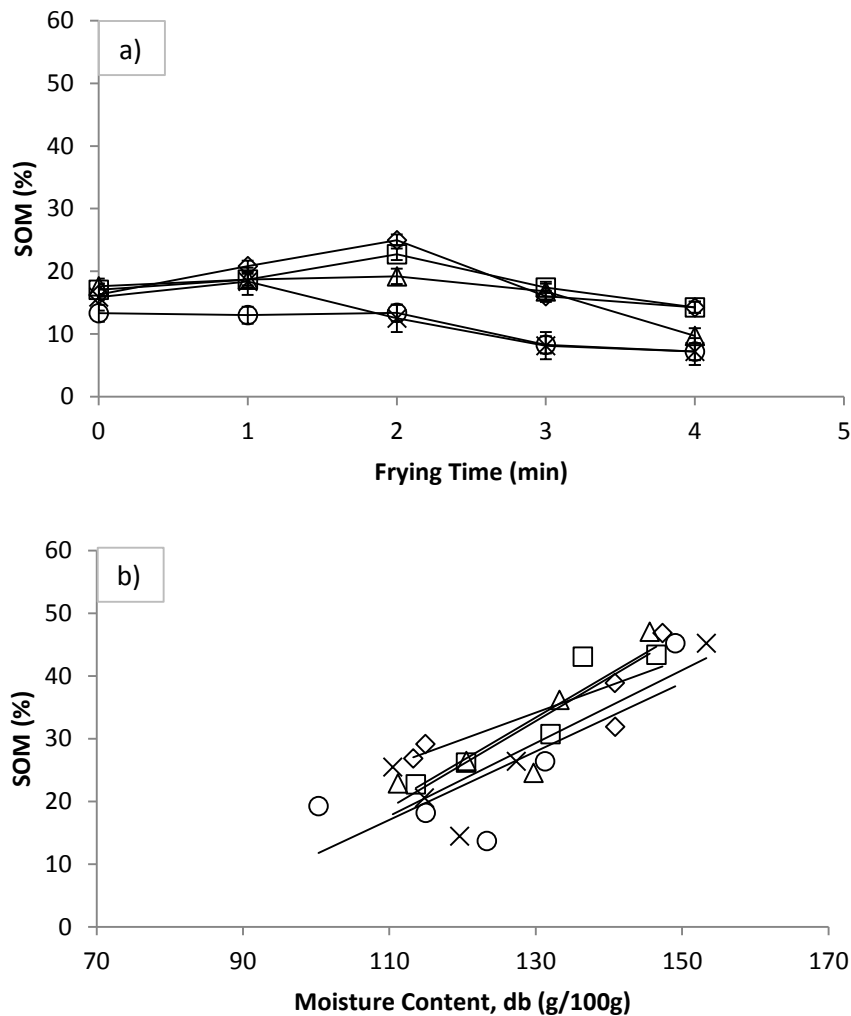


Figure 5-5 Inter-particle space occupied by moisture (SOM) during frying batter coating with different formulations (a), SOM vs. moisture content (b). ◇: 100W0R (100 g wheat flour to 0 g rice flour), □: 75W25R (75g wheat flour to 25 g rice flour), △: 50W50R (50 g wheat flour to 50 g rice flour), ×: 25W75R (25 g wheat flour to 75 g rice flour), and ○: 0W100R (0 g wheat flour to 100 g rice flour).

Increasing the amount of wheat flour in formulation of batter showed significantly ($P < 0.05$) higher SOM compared to batter formulated with less wheat flour.

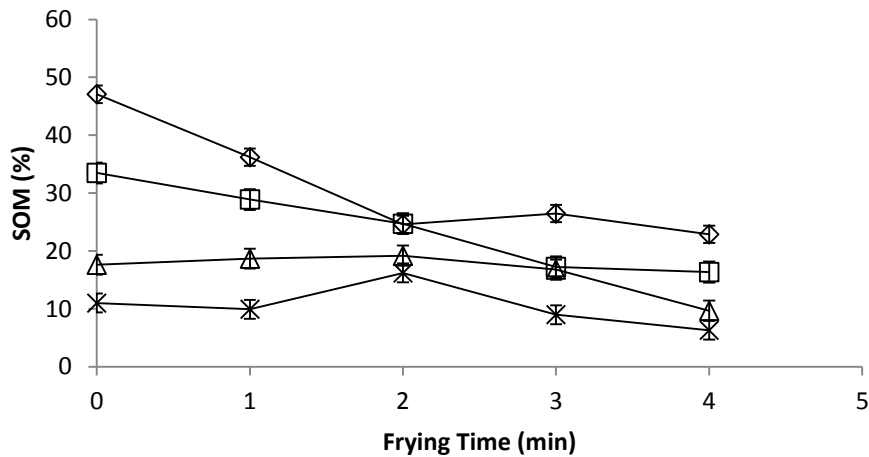


Figure 5-6 Inter-particle space occupied by moisture (SOM) during frying time of batter coating with different pre-drying times. ◇: 0MD (0 min pre-dried batter), □: 30MD (30 min pre-dried batter), Δ: 60MD (60 min pre-dried batter), and ×: 90MD (90 min pre-dried batter).

Inter-particle space occupied by moisture (SOM) range for the 0 min pre-dried fried batter was between 13.70 and 47.07%. The ranges for 30MD, 60MD, and 90MD were 9.54 - 33.47, 7.20 - 24.99 and 2.55 - 22.01%, respectively. Longer pre-drying time significantly ($P < 0.05$) decreased SOM. For 0 min pre-dried batters, the amount of moisture content is high, which means there is more space occupied by moisture than pre-dried batters. By increasing pre-drying time, more moisture is removed from the batter and therefore less space was observed to be filled by moisture. Same interpretation can explain the effect of frying time on SOM. Longer frying time, less moisture content and therefore less SOM was observed.

SOM versus moisture content of batters with different formulation is plotted in Figure 5-5b. Pearson's correlation showed that moisture content had a high positive correlation with SOM of non-dried batters ($r = 0.87$), a moderate positive correlation with SOM of 30MD ($r = 0.56$) and a low positive correlation with SOMs of 60MD and 90 MD ($r = 0.39$, and $r = 0.35$, respectively). Batters with shorter pre-drying times have bigger fraction of voids occupied by moisture than

batters with longer pre-drying times; therefore these batters showed a stronger correlation between moisture content and space occupied by moisture.

5.4.4. Inter-particle space occupied by fat (SOF)

The plots of SOF as a function of frying time at different batter formulations and pre-drying times are shown in Figures 5-7a and 5-8. Analysis of variance (ANOVA) showed that SOF of fried batter was significantly ($P < 0.05$) influenced by the main parameters pre-drying time, batter formulation and frying time. There was also interaction effect between all main parameters at a confidence interval of 95%. Increasing the amount of wheat flour in formulation of batter showed significantly ($P < 0.05$) less SOF compared to batter formulated with less wheat flour. As it was showed earlier, higher wheat flour proportion in the formulation of batter decreased the amount of fat content. Therefore, less SOF was observed for batter with higher amount of wheat flour in their formulation.

Inter-particle space occupied by fat (SOF) range for the 0 min pre-dried fried batter was between 0.48 and 11.11%. The range for 30 min, 60 min, and 90 min pre-dried fried batters were 0.60 – 5.16%, 0.44 – 5.72%, and 0.35 – 5.37%, respectively.

With longer pre-drying time, a significantly ($P < 0.05$) lower SOF was observed, which supports the idea that pre-drying decreases oil uptake. Less oil uptake was observed with longer pre-drying times (Figure 5-2b), and therefore less space was occupied by fat. Pre-drying increases the solid content with a compact matrix of batter systems which reduces the amount of oil suction after frying. For longer frying times, where again more fat is taken up, the fraction of spaces occupied by fat is higher than batters which are fried for shorter times.

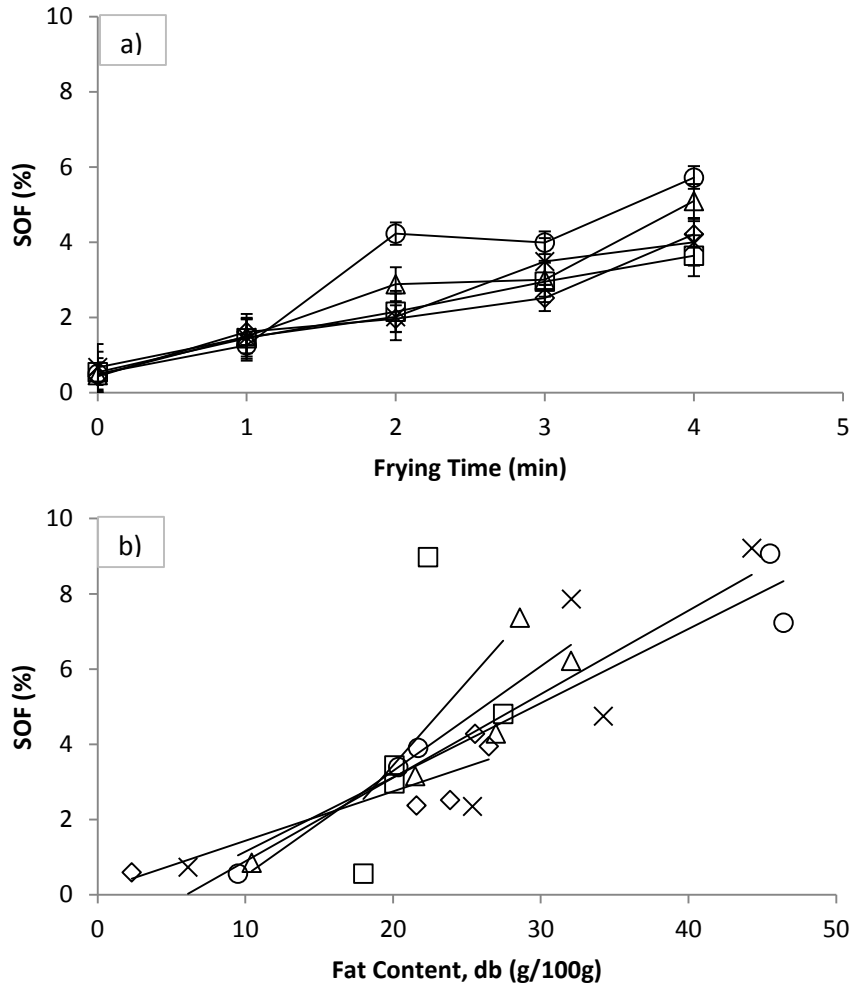


Figure 5-7 Inter-particle space occupied by fat (SOF) during frying of batter coating with different formulations (a), SOF vs. fat content (b). ◇: 100W0R (100 g wheat flour to 0 g rice flour), □: 75W25R (75g wheat flour to 25 g rice flour), △: 50W50R (50 g wheat flour to 50 g rice flour), ×: 25W75R (25 g wheat flour to 75 g rice flour), and ○: 0W100R (0 g wheat flour to 100 g rice flour).

SOF versus fat content for batters with different formulation is plotted in Figure 5-7b.

Pearson's correlation showed that fat content had a high positive correlation with SOF of 0 min pre-dried batters ($r = 0.81$), a moderate positive correlation with SOF of 30MD, 60Md, and 90MD ($r = 0.75$, $r = 0.76$, and $r = 0.65$, respectively).

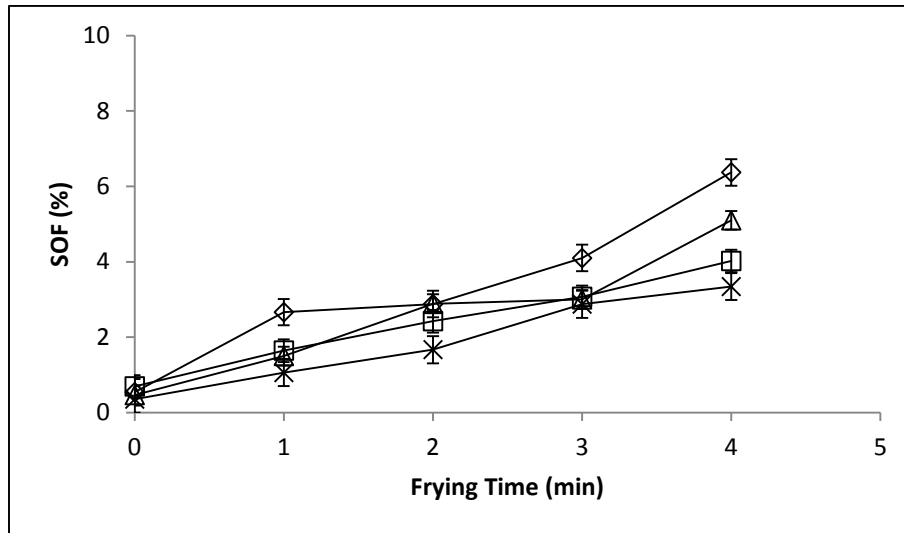


Figure 5-8 Inter-particle space occupied by fat (SOF) during frying of batter coating with different pre-drying times. \diamond : 0MD (0 min pre-dried batter), \square : 30MD (30 min pre-dried batter), Δ : 60MD (60 min pre-dried batter), and \times : 90MD (90 min pre-dried batter).

5.4.5. Inter-particle space occupied by different components

Figure 5-9a-d shows a comparison of how different components of batters dried at different times filled inter-particle spaces. As it is shown, for all pre-drying times, fat occupied the least fraction of the spaces. For non-dried batters, it was observed that moisture occupied a bigger fraction of the spaces than air. However, by drying this switched and in batters pre-dried for 90 min (Figure 5-9d), air occupied a bigger fraction of the spaces than moisture. Moisture content in non-pre-dried batters is higher than other samples and therefore it was expected to see that the fraction of spaces occupied by moisture is very high. During drying, moisture evaporation happened and moisture content decreased and that is why for pre-dried samples more spaces were occupied by air compared to non-pre-dried samples. At each particular pre-drying time, it was seen that there was no significant difference between batter formulated with different proportion of wheat and rice flour with regard to spaces occupied with different component.

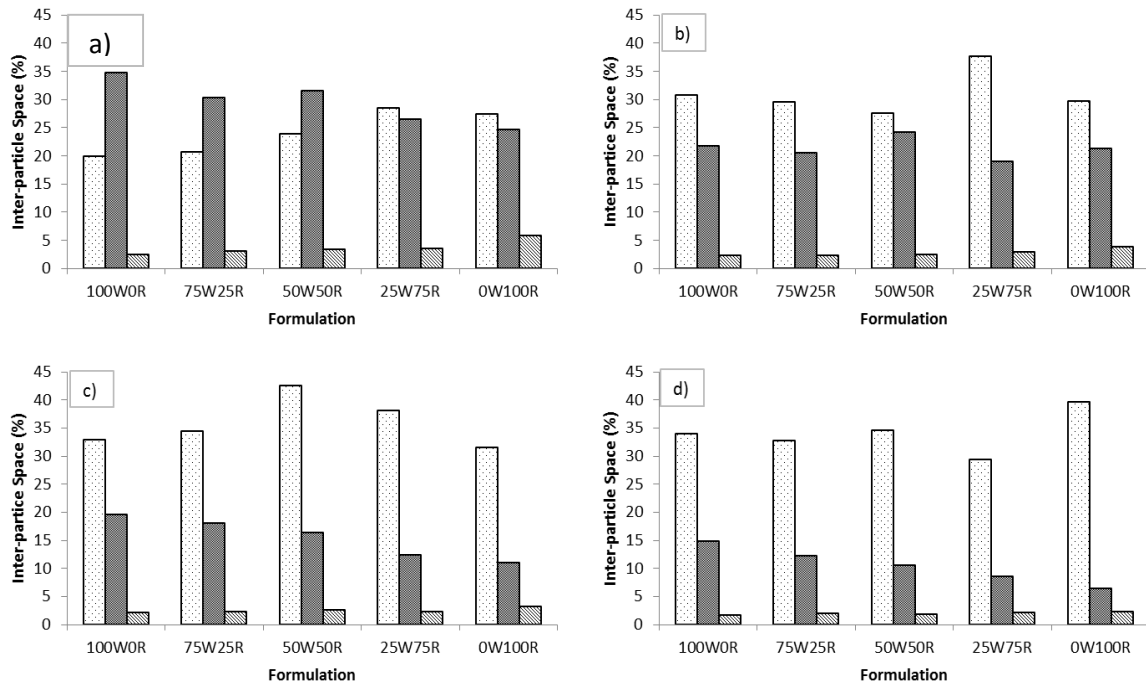


Figure 5-9 Fraction of inter-particle spaces occupied by different components (air, moisture, and fat). a): 0MD (0 min pre-dried batter), b): 30MD (30 min pre-dried batter), c): 60MD (60 min pre-dried batter), d): 90MD (90 min pre-dried batter), 100W0R (100 g wheat flour to 0 g rice flour), 75W25R (75 g wheat flour to 25 g rice flour), 50W50R (50 g wheat flour to 50 g rice flour), 25W75R (25 g wheat flour to 75 g rice flour), 0W100R (0 g wheat flour to 100 g rice flour), : SOA (space occupied by air), : SOM (space occupied by moisture), and : SOF (space occupied by fat).

5.5. Conclusion

Frying time, formulation, and pre-drying time had significant ($P < 0.05$) effect on moisture loss and fat content of batter coating during frying. Inter-particle spaces occupied by air, moisture, and fat were also significantly influenced by these parameters. SOA, SOM, and SOF of the batter systems ranged from 2.2 to 46.03%, 2.55 to 47.07%, and 0.35 to 11.11%, respectively. The higher the frying time, the higher the inter particle spaces occupied by air and fat. Higher proportion of rice flour in batter showed higher fraction of inter-particle spaces occupied by air, and lower fraction of spaces occupied by moisture and fat. Same results were observed with

longer pre-drying times. The fraction of spaces occupied by air was highest followed by moisture and fat. The developed technique herein may be used for other food systems and studies.

CONNECTING TEXT

In chapter 5, it was observed that it was possible to evaluate the spaces of fried batters that were occupied by air, moisture, or fat. These fractions were significantly affected during frying, and with different batter formulations and pre-drying times. Moisture evaporation and fat absorption during frying cause these changes in inter-particle fractions. These mass transfers are surface phenomena and cause some ruptures on the surface of batter being fried. In chapter 6, the surface rupture of fried batters was studied. The geometry of the rupture and its relation with moisture loss and fat uptake during frying was also evaluated.

CHAPTER 6

SURFACE RUPTURES OF FRIED BATTERS AS INFLUENCED BY BATTER FORMULATIONS

6.1. Abstract

Irregularities and microstructure of the surface of batter coatings play a critical role in oil uptake during deep-fat frying of coated products. The objective of this study was to study the influence of batter formulations on surface irregularities of fried batters. Variable pressure scanning electron microscopy (VP-SEM) was used to study the surface microstructure of batters prepared using different combinations of wheat and rice flours. Images of batter surfaces were captured, and characterized using parameters such as the total number, mean area, the biggest area, ratio and roundness of the generated ruptures on the coating surface after frying. The total number of generated ruptures on the surface of fried batters was significantly ($P < 0.05$) influenced by the batter coating formulation. Batters formulated only with wheat flour showed the highest number whereas batters formulated only with rice flour showed the least number of holes and cracks. Roundness of the ruptures ranged from 0.19 to 0.35. The higher amount of rice flour used in the formulation of batter, the higher the area of generated holes and cracks on surface was observed. Fat content was directly correlated with the ratio of the observed holes and cracks generated on the surface ($|r| = 0.72$), whereas moisture loss was correlated with ratio ($|r| = 0.67$) and mean area ($|r| = 0.77$).

6.2. Introduction

Batter is a complex system made of different ingredients such as flour, starch, hydrocolloids, salt, seasoning and water. One of the main purposes of applying batter as a coating layer is to reduce fat uptake in deep-fat fried foods (Mellema 2003, Kim, Lee et al. 2008, Albert, Perez-Munuera et

al. 2009, Adedeji, Liu et al. 2011). Quality of the final fried products is directly determined by the interaction between the frying oil and batter ingredients. Wheat flour is the most common ingredient in batter systems and it is largely available in most regions. However, rice flour has been introduced as a healthier alternative providing fewer calories and less risk of gluten intolerance (Xue and Ngadi 2007, Barutcu, Sahin et al. 2009).

Normally, quick water evaporation happens at the beginning of frying resulting in formation of a thick crust. The crust acts as a barrier that obstructs water evaporation and accumulates water vapor in the product thus increasing internal pressure (Lalam, Sandhu et al. 2013). High internal pressure may destroy the surface and create a variety of ruptures such as holes, open capillaries, channels, cracks, and crevices on the surface of the product (Saguy and Dana 2003, Dana and Saguy 2006). Steam escapes through these cracks and channels during frying; and as the process continues, adhering oil on the product's surface could enter the large voids and crevices created in the structure of the product (Kassama and Ngadi 2005, Kassama and Ngadi 2005). Since the voids are sometimes large, there will be no inner resistance due to positive water vapor pressure (Dana and Saguy 2006). This phenomenon could explain the direct relationship reported between water loss and oil uptake. Hence, the water replacement mechanism is basically related to large voids, cracks and crevices that are normally a characterization of many starchy products. These irregularities on the surface play a very important role in oil suction into fried product, particularly during cooling phase, when a vacuum pressure in the product is generated. Long continuous channels cause higher fat uptake (Mellema 2003).

The porosity which is generated through the whole body, and pore size distribution of fried foods have been largely studied. However, there has not been a thorough study on

irregularities such as holes and cracks, on the surface of foods during frying. This knowledge is needed to understand how surface ruptures occur during frying and how they influence the final characteristics of the fried product.

In recent years, much attention has been given to studying crust microstructure of fried batter coatings. Several techniques have been used for this purpose, for example, scanning electron microscopy, SEM (Barutcu, Sahin et al. 2009, Primo-Martín and van Deventer 2011, Lee, Yoo et al. 2013), confocal laser microscopy, CLM (Mukprasirt, Herald et al. 2000, Adedeji, Liu et al. 2011), light microscopy, LM (Llorca, Hernando et al. 2007), and X-ray micro-CT (Adedeji and Ngadi 2009). In particular, SEM has become the method of choice in many investigations, and it has proved to be an effective method to study the surface microstructure of many food materials. SEM investigation helps in the characterization of the physical and textural properties of food materials.

Variable pressure SEM (VP-SEM) is one of the recent advances in electron microscopy that can be applied to food structural studies. It is equipped with a differentially pumped electron column, in conjunction with one or more apparatus, which allows a pressure difference between the column and the specimen chamber. This allows the electron column to be maintained at high vacuum whereas the specimen chamber is simultaneously maintained at pressures up to around 2500 Pa (James, 2009). This technique, in contrast to conventional electron microscopy, reduces the sample preparation steps, removes the vacuum limitations, and enables the imaging of moist samples at a wide range of pressures and magnifications. Absence of freezing or drying for sample preparation makes VP-SEM a convenient technique to study the structural changes during processes such as deep-frying.

The objectives of this study were to determine the influence of batter formulations on irregularities such as holes and cracks and their geometry on the surface of batters during frying, and to examine the correlations between these surface irregularities with fat uptake and moisture loss.

6.3. Materials and methods

6.3.1. Materials

Wheat flour (Five Rose All Purpose Flour, Les Cuisines Five Roses Kitchens, QC, Canada) was purchased from a local grocery store in Montreal, Canada. Long grain rice flour, RL-100, was prepared by Rivland Partnership (Riceland Foods, Arizona, USA). Carboxymethyl cellulose (CMC) used was supplied by TIC Gums Inc., Maryland, USA. Canola oil (Les Essentiels De La Cuisine, Richmond, BC, Canada) was supplied by the Food & Dining Service, McGill University.

Five different ratios of wheat and rice flours were prepared as follows: 100 g wheat flour to 0 g rice flour (**100W0R**), 75 g wheat flour to 25 g rice flour (**75W25R**), 50 g wheat flour to 50 g rice flour (**50W50R**), 25 g wheat flour to 75 g rice flour (**25W75R**), and 0 g wheat flour to 100 g rice flour (**0W100R**). A fixed amount of salt (NaCl) and CMC were added at 2.5 and 1.5 g/100 g, respectively. Batter slurry was prepared by adding distilled water to flour mix in the ratio of 1.3:1. Then, the batter system was thoroughly mixed and poured in rectangular aluminum cells (50×25×5 mm), and transferred for frying.

6.3.2. Frying

A kitchen programmable deep fat fryer (De'Longhi, America Inc., Saddle Brooke, NJ 076663, China) was used to fry the samples at $180 \pm 2^{\circ}\text{C}$ for 1, 2, 3, and 4 min. The fryer was filled with 1.5 L fresh canola oil, preheated, and maintained at $180 \pm 2^{\circ}\text{C}$ for 2 hrs before frying. To

minimize the variation of oil properties due to degradation during frying, each batch of oil was used for only 30 min before it was replaced with a new batch of oil. All frying experiments were performed in triplicate.

6.3.3. Moisture loss

The fried batter was freeze-dried in a freeze dryer (Modulyod-115; ThermoSavant, Holbrook, NY, USA) at -50°C and 100 mbar for 36 h and was equilibrated in a desiccator for 30 min. Weight of batter, before and after freeze-drying, was measured and used to calculate the moisture content on dry weight basis (db). The difference between moisture content of non-fried batters and fried batters at each particular time point was the moisture loss at that time.

6.3.4. Fat content

Each individual freeze-dried batter (weight: 3-5 g) was placed in thimbles in a VELP SER 148 (Velp Scientifica, Usmate, Italy) solvent extraction unit and oil was extracted with petroleum ether. Oil content was computed by dividing the mass of extracted oil by the mass of freeze-dried sample.

6.3.5. Water binding capacity (WBC)

The water binding capacity (WBC) defined as the amount of water retained by the sample under low-speed centrifugation was measured based on the flour water absorption described by de la Hera, Gomez et al. (2013). Samples (1.000 ± 0.005 g) were mixed with distilled water (10 ml) and centrifuged at $2000 \times g$ for 10 min. WBC was expressed as grams of water retained per gram of solid. Flour hydration properties were analyzed in triplicate.

6.3.6. Scanning electron microscopy

Fried batter samples were prepared for scanning electron microscopy following the method of Rahimi, Khosrowshahi et al. (2007) with modifications. Batters were cut into approximately

5×5×2 mm³ cubes with a sharp razor and immersed in 2.5% glutaraldehyde fixative for 3 h. Cubes were then washed 6 times in distilled water (1 min each time), dehydrated in a graded (40, 55, 70, 85, 90, and 96%) series of ethanol for 30 min each, and defatted in 3 changes of chloroform (10 min each time). The defatted samples were kept refrigerated until SEM imaging. Samples were viewed in a scanning electron microscope (JEOL JSM-6460LV, Tokyo, Japan) operated at 20.0 kV. Digital images were acquired at 25× magnification over regions of 960×1280 pixels. Six images were captured for each sample over squared regions of 5×5 mm². Each image was digitally stored for further analysis.

6.3.7. Surface image analyses

MATLAB image processing toolbox (MATLAB, R 2008a, Version 7.6.0) was used to process the acquired images. To delineate between ruptures, such as holes, cracks and crevices on the surface of the batters and the rest of the surface, a binarization process using *bw* function was applied and the grey scale images were converted to binary (black and white) images. To convert the images, a threshold level was obtained manually using *imtool* function in MATLAB for each specific image. The pixel intensity of the ruptured area on each image was obtained by moving the mouse cursor on the interested area, and that intensity was taken as the threshold level for that particular image. The output image from the binarization process replaced all pixels in the input image with luminance greater than the threshold level with the value 1 (white) and replaces all other pixels with the value 0 (black). The white part on each image was inverted to black and vice versa using *~bw* to make the images more clear and recognizable for further image processing steps. Background noise was removed by *bwareaopen* function. Finally, number of holes and cracks and other ruptures, the biggest rupture, the average area of ruptures, ratio of the

ruptures to the total surface, and roundness of ruptures were measured using related functions on MATLAB.

6.3.8. Statistical analysis

Frying experiments for each batter formulation were applied in triplicates. Analysis of variance was performed using SAS system software (Version 9.3, SAS Institute, Inc., Cary, NC, USA) to determine the effect of formulation on surface characteristics of the samples. Duncan multiple range test was used for mean separation at $P < 0.05$ where treatment effect was significant. The “PROC Corr” of SAS software (Version 9.3, SAS Institute, Inc., Cary, NC, USA) was used for finding the Pearson’s coefficient of correlation ($|r|$) of mean area, the biggest hole, ratio, roundness, and number of ruptures with frying time, moisture loss and fat content. The degree of correlation was described as follows (Rahimi and Ngadi 2014): ($|r| < 0.20$, negligible; $|r| = 0.20$ – 0.40 , low; $|r| = 0.40$ – 0.60 , moderate; $|r| = 0.60$ – 0.80 , marked; and $|r| > 0.80$, high).

6.4. Results and discussion

Table 6-1 represents the water binding capacity of different mixtures of wheat and rice flours. Flour combinations prepared from higher amount of rice flour were significantly higher ($P < 0.05$) in water binding capacity than those of flour combinations with more wheat flour.

Table 6-1 Water binding capacity (WBC) of different mixtures of wheat and rice flours.

	100W0R	75W25R	50W50R	25W75R	0W100R
WBC	0.86 ± 0.01^c	0.85 ± 0.01^c	0.91 ± 0.02^b	1.05 ± 0.02^a	1.08 ± 0.03^a

^{a,b} Means within the same column with different subscripts differ ($P < 0.05$). 100W0R: 100g wheat flour to 0g rice flour, 75W25R: 75g wheat flour to 25 g rice flour, 50W50R: 50 g wheat flour to 50 g rice flour, 25W75R: 25 g wheat flour to 75 g rice flour, and 0W100R: 0 g wheat flour to 100 g rice flour.

The images presented in Figure 6-1 (a-d) are sample series of the images which were used to extract the characteristics of the batter surfaces. They are the grayscale and processed images of 100W0R batter fried for 4 min. Figure 6-1a is the grayscale SEM image captured from the surface of the batter. Figure 6-1b is the binarized image from grayscale image 1a. In this image, the black region is the region of interest i.e. holes and cracks and the white region is the background of the image, which is the undamaged part of the surface of the batter. To make the image clearer and easier to study, a conversion process was applied and the binarized image 1b was converted to the binarized image 1c where the black region is the background and the white region is the region of interest. Finally, the small white dots which were assumed to be noise were removed and image 1d was obtained and used for the final data extraction steps. The converted binary image may contain some noises. To remove these unwanted noises, *bwareaopen* function was used. *bwareaopen* gets rid of any detection that is smaller than a given pixel area value. Same procedure was applied to all other batter images to obtain interested information.

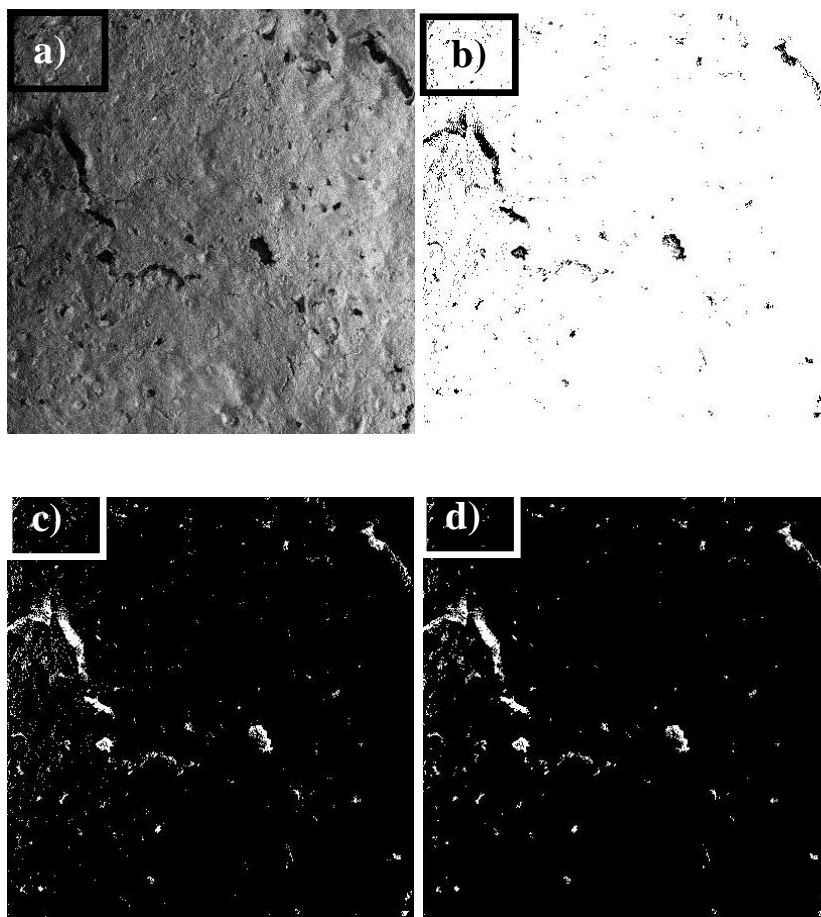


Figure 6-1 SEM images of surface of 100W0R fried batter for 4 min. a) grayscale image b) binarized image of a c) inverted image of b d) de-noised image of c.

The surface characteristics of batters with different formulations after 4 min frying are shown in Figure 6-2. Images of the surface of each of the batters with each various formulations were captured. Figure 6-2a shows the number of ruptures generated on the surface of batters after 4 min frying. Number of ruptures ranged from 181 for 0W100R to 449 for 100W0R. Batter formulation showed a significant ($P < 0.05$) effect on the number of ruptures. Fried batters prepared with higher proportion of rice flour showed less number of ruptures than batters with higher amount of wheat flour in the formulation. This might be because of the elastic properties of gluten film in batters with higher proportion of wheat flour that acts as a barrier against water evaporation. The internal vapor bubbles were probably broken to several smaller ones in batters

with stronger barrier film than in batters with weaker film. Therefore in stronger batters, instead of having low number of big holes and cracks on the surface, a high number of small holes and cracks were created. The size of the holes and cracks and their ratio on the surface of high rice flour content batters was significantly higher than batters with higher amount of wheat flour (Figure 6-2c, d and e), this supports the previous statement.

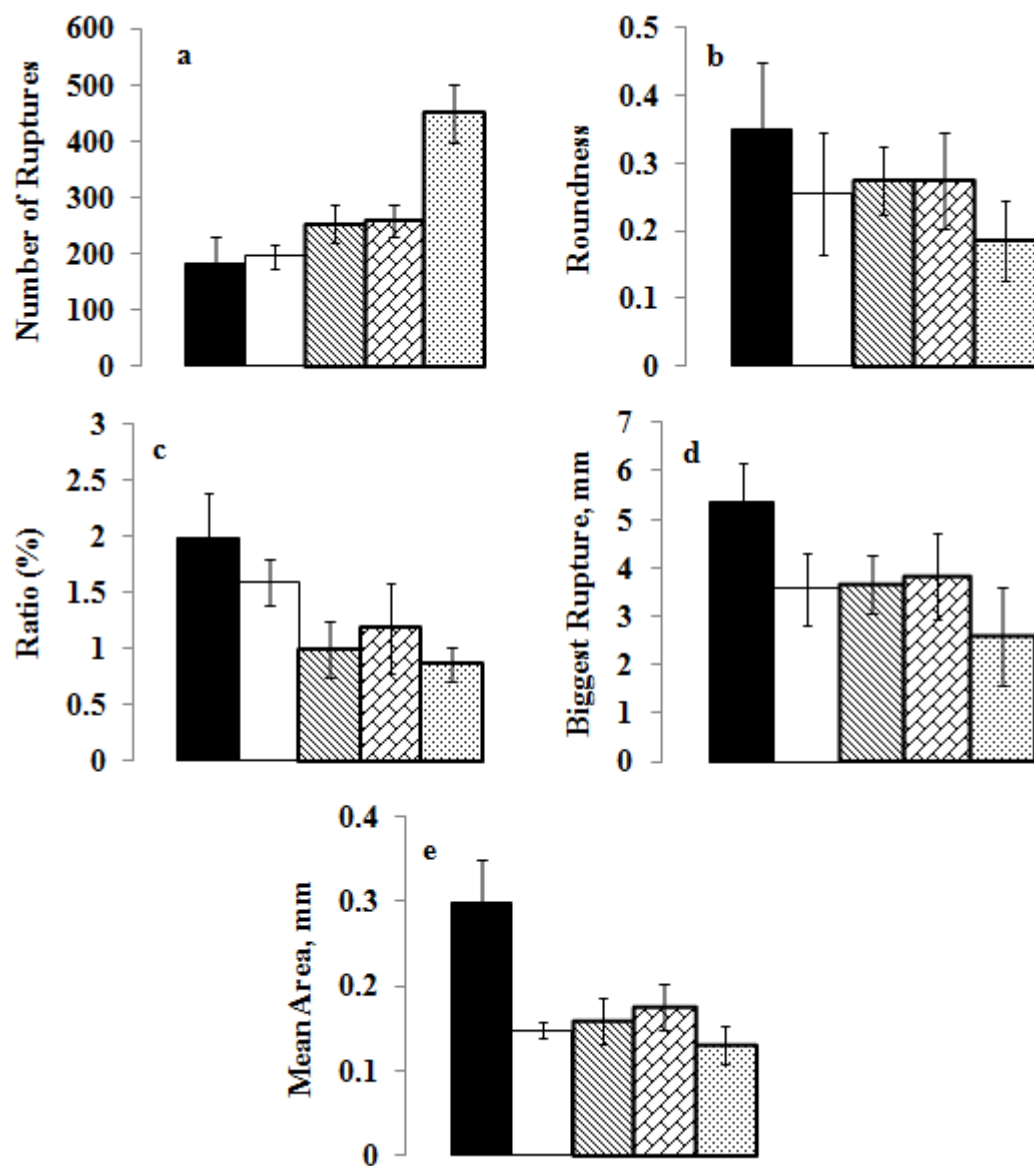


Figure 6-2 Surface characteristics of fried batters for 4 min. a) number of ruptures, b) roundness, c) ratio of the ruptures (%), d) area of the biggest rupture (mm), e) mean area of the ruptures (mm). ■: 0W100R (0 g wheat flour to 100 g rice flour), □: 25W75R (25 g wheat flour to 75 g rice flour), ▨: 50W50R (50 g wheat flour to 50 g rice flour), ▩: 75W25R (75 g wheat flour to 25 g rice flour), ▤: 100W0R (100 g wheat flour to 0 g rice flour).

Figure 6-2b shows the roundness of the ruptures created on the surface of batters after 4 min frying. Roundness is a measure of the sharpness of the corners of the solid (Mohsenin 1986), or in other words it is a measure of the extent to which the edges and corners of a particle has been rounded (Cabalar and Hasan 2013). It was proposed that the geometry of the ruptures on the surface of the batters may vary by different formulations. Several methods have been suggested for estimating roundness. Using MATLAB software, the mean area and the mean perimeter of the ruptures were already calculated and then were put in the equation below (Xie, Tang et al. 2013) to measure the roundness.

$$Roundness = \frac{4 \times MA \times \pi}{(P)^2} \quad \text{Equation (6-1)}$$

where MA is the mean area of ruptures generated on the batter surfaces, and P is the perimeter of the rupture. Roundness ranged from 0.19 to 0.35. No significant difference ($P < 0.05$) was observed between the roundness of batters with different formulations after frying for 4 min. Roundness varies between 0 and 1 (Kang, Ke et al. 2007). A roundness value equal to 1 means that the shape of the rupture is perfectly round. Hence a roundness value of a rupture on the surface which is closer to 1 than to 0 means that the shape of the rupture is most likely a circular hole and not a linear crack. The results obtained in this study (i.e. roundness between 0.19 and 0.35) showed that the number of the cracks generated on the surface is more than the number of the holes. The roundness values for 100W0R, 75W25R, 50W50R, 25W75R, and 0W100R were 0.19, 0.27, 0.27, 0.25, and 0.35, respectively. These results show that batters with higher amount of rice flour tend to show a more round-shape rupture on the surface than batters prepared with higher amount of wheat flour.

Hole-shape rupture may occur on a surface when 1) internal pressure, which is caused by saturated vapor pressure, is not uniformly distributed under the crust surface and/or 2) when heterogeneity of the mechanical strength of the crust exist. The difference between internal vapor and atmospheric pressures causes ruptures in the shape of hole over the area of the crust where is not strong enough to hold the vapor. However, the crack-shape rupture occurs when the specific volume increased in the core region exceeds the expansion of the crust during frying (Nagao, Hatae et al. 1997). The interior vapor pressure in batters with higher amount of rice flour is probably not distributed as equal as batters with higher amount of wheat flour.

Figure 6-2c shows the ratio of the ruptures generated on the surface to the whole surface area of batters fried for 4 min. The ratio ranged from 0.87% for 100W0R to 1.99% for 0W100R. Batter formulation showed a significant ($P < 0.05$) effect on the ratio of the ruptures. Batters prepared with higher proportion of rice flour showed higher ratio than batters with higher amount of wheat flour in the formulation. Figure 6-2d shows the area of the biggest rupture created on the surface of batters fried for 4 min. The biggest rupture area ranged from 3.57 to 5.65 mm. Batter with higher amount of rice flour showed a higher value for the biggest rupture on the surface than batters with higher amount of wheat flour.

Figure 6-2e shows the mean area of the ruptures generated on the surface of batters fried for 4 min. The mean area ranged from 0.12 mm for 100W0R to 0.30 mm for 0W100R. Batter formulation showed a significant ($P < 0.05$) effect on the mean area. Batters prepared with 0g wheat and 100g rice flours showed a significant higher mean area than other batters, whereas no significant difference was observed between the rest of the batters. The changes observed in the microstructure of the samples could be attributed to mass transfer processes and physicochemical transformations such as protein denaturation, and starch gelatinization during frying (Kassama

and Ngadi 2005, Adedeji and Ngadi 2009, Adedeji, Liu et al. 2011). Moisture evaporation leads to some ruptures and irregularities on the surface such as formation of voids, holes and cracks. The gluten network generated because of interaction between water and gluten protein plays a very important role in the number and size of the rupture. The network created by rice starch and water in rice-flour-based batters was weaker than the networked developed in wheat-flour-based batters, which contains gluten protein, and as a result more rupture happened on its surface. An increase in internal pressure of products during frying clearly plays an important role in occurrence of rupture on the crusted surface (Nagao, Hatae et al. 1997). A systematic study on surface rupture of batter coating has not been performed before. However, some authors concentrated on porosity change of batter during frying using microscopy techniques. Adedeji, Liu et al. (2011) used confocal laser scanning microscopy to study the microstructure of deep fat fried chicken nugget batter coating, where formation of more micropores and bigger pores during frying was observed. Pedreschi and Aguilera (2002) studied the changes experienced during frying of potato chips and observed severe changes in original shape of cells, such as shrunk and wrinkled walls, but no signs of rupture during frying at 20X magnification, which was used for their study. Restructured products such as batter show a weaker structure than naturally built materials such as potato cell walls (Ovalle, Cortés et al. 2013). The difference between these studies might be attributed to the difference in the degree of strength in these two different products. Batter products lack a strong structure and therefore show a more severe rupture than potatoes during processing.

The degree of correlation $|r|$ between the three main parameters frying time, moisture loss, and fat content and each individual surface characteristic, which were explained in Figure 6-2, was measured and then plotted in Figure 6-3. As described earlier, those $|r|$ that were less than

0.6 were considered as negligible, low or moderate correlation; and those $|r|$ that were higher than 0.6 were considered as marked or high correlation. It was decided to choose the correlations which were higher than 0.6 and plot them separately for further discussion. All chosen correlations were statistically significant at $P < 0.05$. For the biggest rupture area and roundness, none of the parameters was higher than 0.6. For mean area of the ruptures, a positive marked correlation ($|r| = 0.77$) was observed with moisture loss; whereas it was less than 0.6 for frying time and fat content.

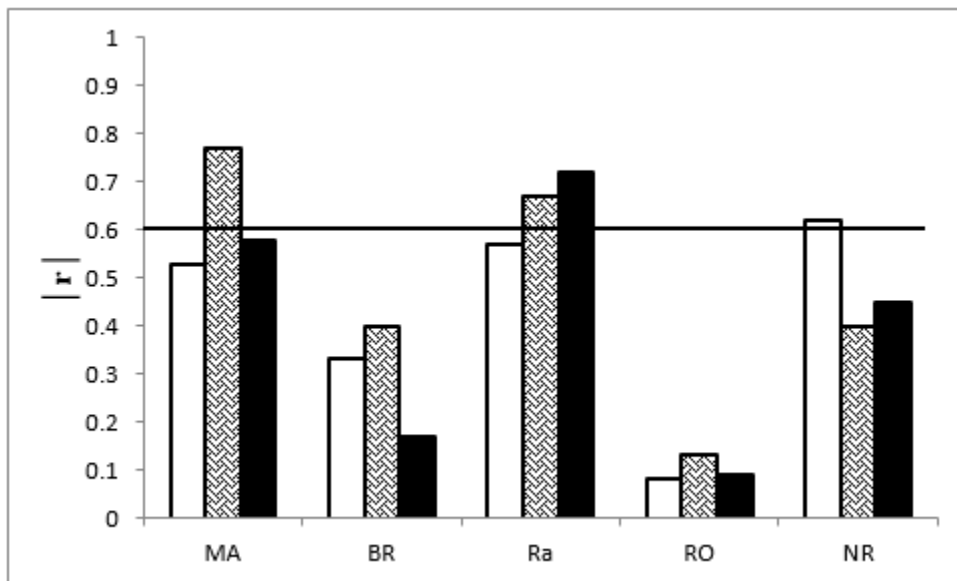


Figure 6-3 Plot of degree of correlation, $|r|$, between mean area of the ruptures (MA), the biggest rupture (BR), ratio of the ruptures (Ra), roundness of the rupturesoles (RO), and number of the ruptures (NR) on the surface of fried batters and frying time: □, moisture loss: ▨, and fat content: ■.

The degree of correlation $|r|$ between ratio of the rupture on the surface and moisture loss, and fat content were 0.67 and 0.72, respectively. However, the $|r|$ between ratio and frying time was less than 0.6. Finally, the $|r|$ between number of the ruptures and frying time was 0.62, whereas it was less than 0.6 between this characteristic and frying time and fat content.

Figure 6-4 represents the correlation between moisture loss and mean area of the ruptures generated on the surface of batters during frying. As it can be seen, for all batter formulations the mean area is positively correlated to moisture loss. The higher the moisture loss during frying, the higher the mean area of the ruptures was observed.

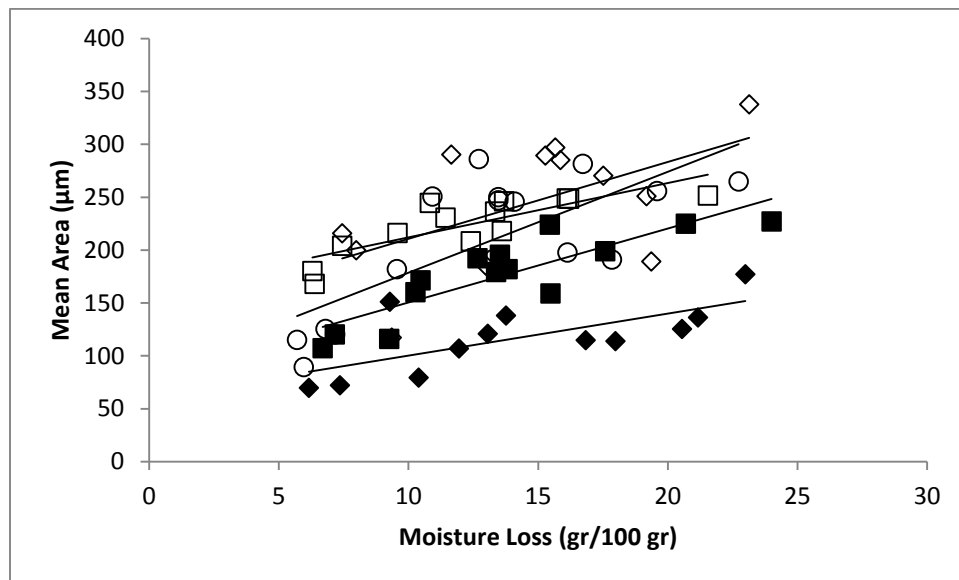


Figure 6-4 Plot of correlation between moisture loss and mean area (μm) of the ruptures generated on the surface of batters with different formulations. \diamond : 0W100R (0 g wheat flour to 100 g rice flour), \square : 25W75R (25 g wheat flour to 75 g rice flour), \circ : 50W50R (50 g wheat flour to 50 g rice flour), \blacksquare : 75W25R (75 g wheat flour to 25 g rice flour), \blacklozenge : 100W0R (100 g wheat flour to 0 g rice flour).

Figure 6-5 shows the correlation between moisture loss (%) and ratio (%) of the generated ruptures to the surface of batters. The ratio significantly increased by higher moisture loss for all batter formulations. When the food is exposed to frying temperatures, water evaporates rapidly, the outer surface becomes dry and a crust full of cracks, open capillaries and channels forms.

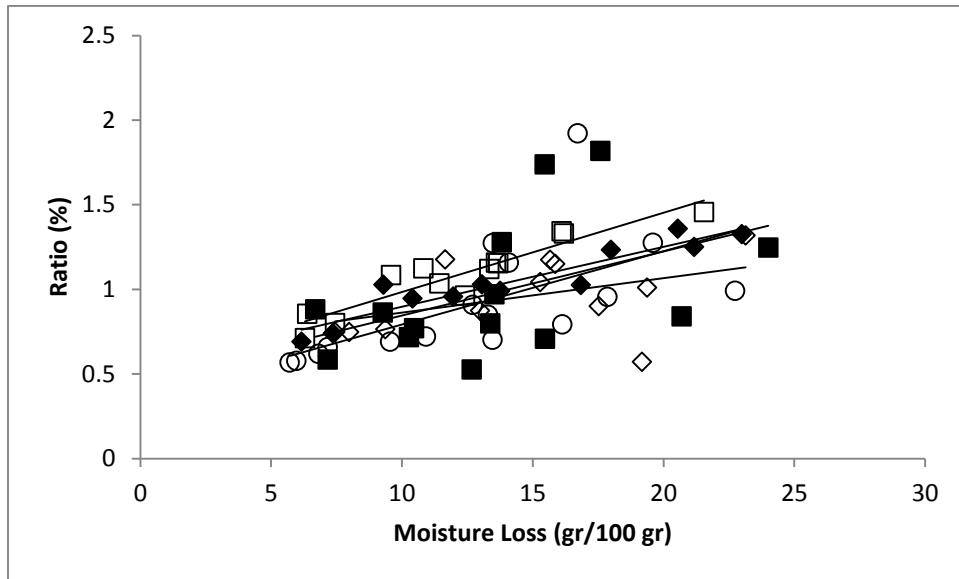


Figure 6-5 Plot of correlation between moisture loss and ratio (%) of the generated ruptures to the surface of batters with different formulations. \diamond : 0W100R (0 g wheat flour to 100 g rice flour), \square : 25W75R (25 g wheat flour to 75 g rice flour), \circ : 50W50R (50 g wheat flour to 50 g rice flour), \blacksquare : 75W25R (25 g wheat flour to 75 g rice flour), \blacklozenge : 100W0R (100 g wheat flour to 0 g rice flour).

Figure 6-6 shows the correlation between fat content (%) and ratio (%). The higher the ratio of the ruptures on the surface, the higher the fat content was observed. As observed earlier, there was a positive correlation between moisture loss during frying and area of ruptures generated on the surface. As the process of frying progresses, oil adheres to the food, entering the voids, openings and crevices left on the surface of the product by the microstructural changes. The larger the size of the ruptures on the surface, the less the inner resistance due to positive water vapor pressure, and therefore the higher the amount of oil suction into the fried food (Dana and Saguy 2006).

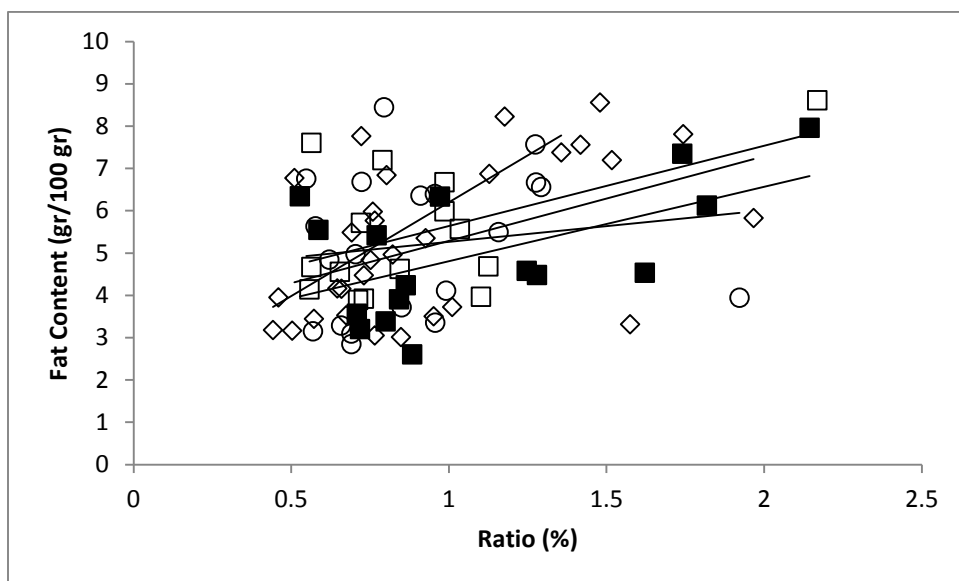


Figure 6-6 Plot of correlation between batter fat content and ratio (%) of the generated ruptures to the surface of batters with different formulations. \diamond : 0W100R (0 g wheat flour to 100 g rice flour), \square : 25W75R (25 g wheat flour to 75 g rice flour), \circ : 50W50R (50 g wheat flour to 50 g rice flour), \blacksquare : 75W25R (75 g wheat flour to 25 g rice flour), \blacklozenge : 100W0R (100 g wheat flour to 0 g rice flour).

Figure 6-7 represents the relation between number of the ruptures generated on the surface of batters during frying and frying time. The longer the frying time the more ruptures were generated on the surface.

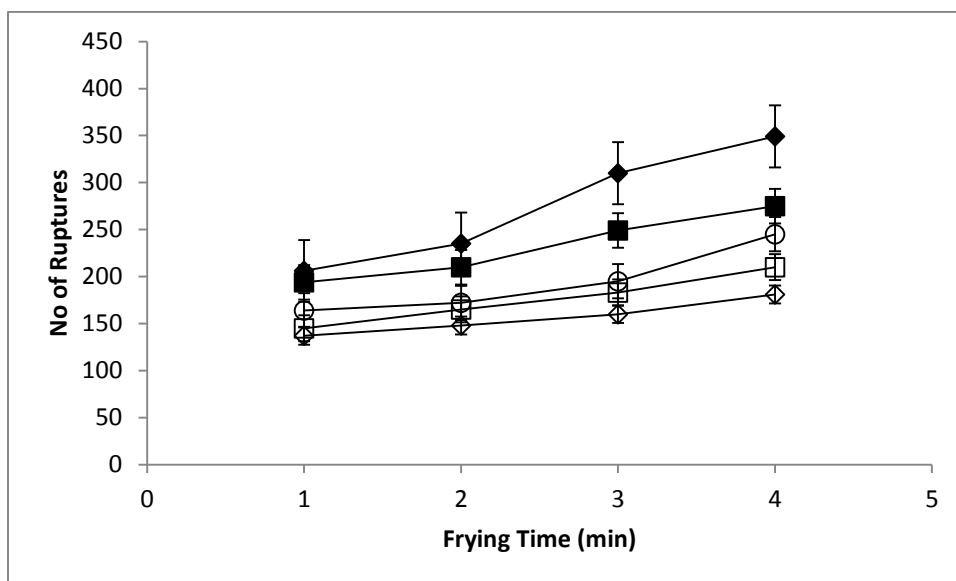


Figure 6-7 Plot of correlation between number of ruptures on the surface during fring and frying time with different formulations. ♦: 0W100R (0 g wheat flour to 100 g rice flour), □: 25W75R (25 g wheat flour to 75 g rice flour), ○: 50W50R (50 g wheat flour to 50 g rice flour), ▲: 75W25R (75 g wheat flour to 25 g rice flour), ✖: 100W0R (100 g wheat flour to 0 g rice flour).

6.5. Conclusions

Variable pressure scanning electron microscopy has great potential for characterizing surface properties of deep-fat fried food coating as discussed. Images of the surface of non-fried and fried batters prepared with different formulations showed that some ruptures happened on the surface of batters when affected by a presure difference or by frying. Different formulations used for batter preparation showed significantly different behaviour in ruptures that occur on their surfaces. Batter prepared from only wheat flour showed the highest number of ruptures whereas batters formulated only with rice flour showed the least number of ruptures. Roundness of the ruptures was affected by batter formulation. The higher amount of rice flour used in the formulation of batter, the higher the area of generated holes and cracks on surface was observed. Fat content, moisture loss, and frying time were positively correlated with some microstructural

characteristics of the surface. Fat content was directly correlated with the ratio of the ruptures generated on the surface, whereas moisture loss was correlated with ratio and mean area.

CONNECTING TEXT

As was explained earlier, a big part of oil is carried on the surface of fried batter when it is removed from the frying oil. This fraction of oil is assumed to be directly dependent on the surface roughness and irregularities of fried batters. In chapter 7, fractal dimension and lacunarity were used as useful tools to study the surface roughness and irregularities of fried batters.

CHAPTER 7

STRUCTURE AND IRREGULARITIES OF SURFACE FRIED BATTERS STUDIED BY FRACTAL DIMENSION AND LACUNARITY ANALYSIS

7.1. Abstract

Fractal dimension and lacunarity are the fractal geometry parameters used effectively in describing images of different objects. These tools were applied to study the structure and irregularities of the surface of batters with different formulation during frying. Using scanning electron microscopy, images of batter surfaces were captured, and analyzed via ImageJ software. Fractal dimension (FD), as a method to measure the surface roughness of fried batters, ranged between 1.79 and 1.81. Application of higher amount of wheat flour in batter preparation resulted in higher FD values compared to batters with higher amount of rice flour. FD significantly ($P < 0.05$) increased during frying. There was a high positive correlation between FD and fat uptake for all batter formulations ($|r| = 0.91-0.99$) that means that surface roughness is an important factor affecting the amount of fat remaining on the surface of batters. Lacunarity, as a measure of degree of heterogeneity of batter ruptures, ranged between 1.19 and 1.25. The lacunarity results showed that the size and shape of ruptures, i.e. cracks and holes, generated on the surface of batters during frying was approximately uniform. This is the first reported use of lacunarity for the characterization of images of fried batter surfaces.

7.2. Introduction

Surface topography, i.e., structure, morphology, or texture, is an essential physical feature of solid foods impacting not only their sensorial and optical feature, but also their behavior during processing and storage. Different food processing operations cause new surfaces with new

characteristics. Roughness, as a surface characteristic, is entirely scale sensitive, which means a surface that normally looks smooth and flat to the naked eye may be rough when examined using advanced microscopic techniques. These microscopy techniques provide high quality digital images for further processing. In a digital image of a surface, data is stored as an array of pixels with different intensity or grey scale. Therefore, the local deviation of brightness from one pixel to another pixel or within a small area is defined texture. The texture of images is a useful method applied for pattern identification to characterize the arrangement of basic constituent of a material in a surface (Quevedo, Carlos et al. 2002).

Conventionally and based on Euclidean geometry, the dimension of an object is expressed as an integer number. However, the dimension of some patterns such as contours of biological cells, fractals and some other natural objects is very difficult to describe based on Euclidean geometry, but can be quantitatively evaluated using measures of complexity. Fractal geometry is considered as an extension of Euclidean geometry. The word fractal was first coined by Mandelbrot (1982) from the Latin adjective “fractus”, which corresponds to the Latin verb “frangere” meaning “to break”, or “to create irregular fragments”. Fractals have been used to describe and measure irregular fragments or complex shapes of materials such as mountains, shorelines, clouds, stars, plants, brain cells, and gold colloids (Mandelbrot 1982, Kerdpi boon, Kerr et al. 2006). Many foods, like many other natural objects, show a number of specific properties such as complexity and inhomogeneity in their structure. Study of complexity and inhomogeneity of food surfaces using some measures such as fractal dimension (FD) and lacunarity (λ) can become a great interest for many scientists and provide useful information for food processors (Smith Jr, Lange et al. 1996).

Fractal dimension is a very important parameter for topographical measurement of various materials (Chen 2007). It is used in several applications, such as, measurement of irregularities in an image, texture segmentation, surface roughness estimation and many other functions (Biswas, Ghose et al. 1998). It has also found vast applications in fields ranging from material science, power technology, computer vision, and micro-electronics. The morphological characteristics of a fractal remain the same regardless of the scale of observation and the level of magnification (Quevedo, Carlos et al. 2002). Fractal functions can provide suitable information and model for explaining rough surfaces. Different methods have been used in the literature to estimate fractal dimension (Biswas, Ghose et al. 1998, Quevedo, Carlos et al. 2002, Zheng, Sun et al. 2006). These methods include differential fractal Brownian motion (FBM) method, ε -blanket method, variation method, two-dimensional variation method, differential box counting (DBC) method and frequency domain method.

Studies have shown that fractal dimension is not a sufficient metric for the characterization of most textures (Valous, Sun et al. 2010, Iqbal, Valous et al. 2011), since it only measures how much space is filled. Lacunarity complements fractal dimension by measuring how the data fill the space. Lacunarity is a term that is used to describe the morphology of objects (Dàvila and Parés 2007, Dàvila, Toldrà et al. 2007). It is accounted as the gappiness or the visual texture of an image because it explains the heterogeneity of the gaps or the degree of structural variance within an object (Smith Jr, Lange et al. 1996). Lacuna (related to lake) is originally a Latin word for gap (Mandelbrot 1982), and a fractal is said to be lacunar if the gaps in an object have large intervals, holes, and voids. Objects are more lacunar if gap sizes are distributed over a wider range (Dàvila, Toldrà et al. 2007). Lacunarity calculates the distribution of gap sizes along datasets. Heterogeneous, high-lacunarity sets cover gaps of

divergent lengths while sets with even gaps are homogeneous and exhibit low lacunarity (Charisis, Hadjileontiadis et al. 2014). Lacunarity is a scale dependent measure of heterogeneity of texture since a heterogeneous set at small scale might be considered homogenous at larger scales and vice versa. For fried batters, these gaps refer to holes, cracks and any other ruptures on the surface, where open channels are available for water and oil to transfer. The study of gap distribution i.e. heterogeneity of the rupture, by means of lacunarity provides information about the surface microstructure of fried batter.

Previous studies revealed that the surface attributes of fried foods and especially the geometrical irregularity or roughness highly influence the oil uptake kinetic (Quevedo, Carlos et al. 2002, Thanatuksorn, Pradistsuwana et al. 2005, Moreno, Brown et al. 2010). Using scanning laser microscopy, Moreno, Brown et al. (2010) studied the relationship between surface roughness and oil gain in fried formulated products. Area-scale fractal analysis approach was used to measure surface topography. The surface topographic analysis showed that potato flake based products are considerably rougher than gluten based products. The authors also found that there was a good agreement between oil uptake and surface description within each product category. Quevedo, Carlos et al. (2002) employed area-scale fractal analysis technique to quantify morphological changes in images acquired by microscopy for starch gelatinization during frying process and chocolate blooming during storage. In the study by Thanatuksorn, Pradistsuwana et al. (2005), the correlation between changes in fractal dimension, as measure of roughness surface, due to initial moisture level and the final oil content were considered for a wheat flour and water food model.

Several factors including porosity, hydrophobic behaviour and topography, determine the characteristics of a food and therefore, transport phenomena during its frying. The purpose of

this study was to: measure surface topography, particularly fractal dimension as a metric of roughness of batters as affected by different combinations of wheat and rice flours during frying; assess the effect of the surface roughness on fat content; and to study the level of heterogeneity using lacunarity analysis of fried batter surfaces.

7.3. Materials and methods

7.3.1. Materials

Wheat flour (Five Rose All Purpose Flour, Les Cuisines Five Roses Kitchens, QC, Canada) was purchased from a local grocery store in Montreal, Canada. Long grain rice flour, RL-100, was prepared by Rivland Partnership (Riceland Foods, Arizona, USA). Carboxymethyl cellulose (CMC) used was supplied by TIC Gums Inc., Maryland, USA. Canola oil (Les Essentiels De La Cuisine, Richmond, BC, Canada) was supplied by Food & Dining Service, McGill University.

7.3.2. Batter preparation

Five different ratios of wheat and rice flours were prepared namely: 100 g wheat flour to 0 g rice flour (100W0R), 75 g wheat flour to 25 g rice flour (75W25R), 50 g wheat flour to 50 g rice flour (50W50R), 25 g wheat flour to 75g rice flour (25W75R), and 0 g wheat flour to 100 g rice flour (0W100R). A fixed amount of salt (NaCl) and CMC were added at the amount of 2.5 and 1.5 g/100 g, respectively. Batter slurry was prepared by adding distilled water to flour mix in the ratio of 1.3:1. Then, the batter system was thoroughly mixed and poured in rectangular aluminum cells (50×25×5 mm), and transferred for frying.

7.3.3. Frying

A kitchen programmable deep fat fryer (De'Longhi, America Inc., Saddle Brooke, NJ 07663, China) was used to fry the samples at $180 \pm 2^{\circ}\text{C}$ for 1, 2, 3, and 4 min. The fryer was filled with 1.5 L fresh canola oil and preheated and maintained at $180 \pm 2^{\circ}\text{C}$ for 2 hrs before frying. To

minimize the variation of oil properties due to degradation during frying, each batch of oil was used for only 30 min before it was replaced with a new batch of oil. All experiments were performed in triplicate. Excess oil on the surface was mopped off using paper towel. Fried samples were then allowed to cool under ambient conditions.

7.3.4. Moisture loss

The fried batter was freeze-dried in a freeze dryer (Modulyod-115; ThermoSavant, Holbrook, NY, USA) at -50°C and 100 mbar for 36 h and was allowed to equilibrate in a desiccator for 30 min. Weight of batter before and after freeze-drying was measured and used to calculate the moisture content. Moisture loss was the difference between moisture content of batters at each specific frying time and moisture content of non-fried batter.

7.3.5. Fat content

The freeze-dried batters were ground in a coffee grinder (Bodum 5678-57; C-Mill, Bodum Inc., New York, NY, USA), and then 3-5 g of samples was placed in thimbles in a VELP SER 148 (Velp Scientifica, Usmate, Italy) solvent extraction unit, and oil was extracted with petroleum ether. Oil content (dry basis) was computed by dividing the mass of extracted oil with the mass of freeze-dried sample.

7.3.6. Scanning electron microscopy

Fried batter samples were prepared for scanning electron microscopy at 4 min of frying following the method of (Rahimi, Khosrowshahi et al. 2007). Batters were cut into approximately $5 \times 5 \times 2 \text{ mm}^3$ cubes with a sharp razor and immersed in 2.5% glutaraldehyde fixative for 3 h. Cubes were then washed 6 times in distilled water (1 min each time), dehydrated in a graded (40, 55, 70, 85, 90, and 96%) series of ethanol for 30 min each, and defatted in 3 changes in chloroform (10 min each time). The defatted samples were kept refrigerated until

SEM imaging. Samples were viewed in a scanning electron microscope (JEOL JSM-6460LV Tokyo, Japan) operated at 20 kV. Digital images were acquired at 25× magnification over regions of 960×1280 pixels. Six images were captured on each sample over squared regions of 5×5 mm². Each image was digitally stored for further analysis.

7.3.7. Fractal dimension measurement

Fractal dimension (FD) of SEM images captured from the surface of batters was measured by the public domain java-based image processing software package (ImageJ 1.47v, National Institute of Health, USA) with the accompanied FracLac v2.5w plug-in (Karperien 2007). This plug-in employs a fractal analysis technique called box-counting method to measure the fractal dimension as the degree of irregularity. FD value is interpreted as the surface roughness index for the actual surface. The box counting method involves covering the whole space of an image, where boxes with different sizes (P) are mounted into the images. The number of boxes (N) which are formed on the image is then counted. The relationship between the number of boxes covering a surface contour N and the size of the grids P corresponds to fractal dimension (Moreno, Brown et al. 2010). The fractal dimension is obtained from calculation using the scaling rule, expressed as (Dàvila and Parés 2007):

$$D = -\frac{\log N}{\log P} \quad \text{Equation (7-1)}$$

where N is the number of boxes covering the contour, P is the size of the boxes, and D is the fractal dimension. Since the calculation of FD by image analysis is performed on a two-dimensional space, an extra dimension should be added to D value to represent the three-dimensional features of images of batter surfaces:

$$FD = 1 + D \quad \text{Equation (7-2)}$$

7.3.8. Lacunarity measurement

Lacunarity (λ) of batter surface images was computed by applying FracLac v2.5w plug-in available for imageJ software. A short description of lacunarity measurement is given as follows (Velazquez-Camilo, Bolaños-Reynoso et al. 2010, Iqbal, Valous et al. 2011). The algorithm for the lacunarity computation measures deviation from translational invariance of an image's intensity distribution using gliding box sampling. Lacunarity is based on the pixel distribution for an image, which is basically obtained from scans at different box sizes at different grid orientations. A square moving window of side length of r pixels is placed in the upper-left hand corner of a binary image of side length T pixels, such that $r \leq T$. The algorithm counts the number or mass m of pixels that are associated with the image underneath the moving window. The window is then translated by one pixel to the right and underlying mass is again recorded. When the moving window reaches the right-hand side of the image, it is placed back to its starting point at the left hand side of the image and is translated by one pixel downward. The computation is repeated over the entire set until the moving window reaches the lower right-hand edge of the image. This procedure produces a frequency distribution of the box masses $n(m, r)$. This frequency distribution is converted into a probability distribution $Q(m, r)$ by dividing by the total number of boxes $N(r)$ of size r . Lacunarity is then calculated by using Equation (7-3):

$$\lambda = (\sigma^2/\mu^2) + 1 \quad \text{Equation (7-3)}$$

where σ is standard deviation and μ is mean. The ratio of σ to μ changes with the size of window covering the images, which shows that lacunarity is related to the scale of measuring windows (Iqbal, Valous et al. 2011).

7.3.9. Statistical analysis

Frying experiments for each batter formulation were conducted in triplicates. Analysis of variance was performed using SAS system software (Version 9.3, SAS Institute, Inc., Cary, NC, USA) to determine the effect of formulation and frying time on fractal dimension of the samples. Duncan multiple range test was used for mean separation at $P < 0.05$ where treatment effect was significant. The “PROC Corr” of SAS software (Version 9.3, SAS Institute, Inc., Cary, NC, USA) was used for finding the Pearson’s coefficient of correlation (r) of fractal dimension with moisture loss and fat content.

7.4. Results and discussions

Figure 7-1 shows images (left) and their respective gray level intensity maps (right) of the surfaces of batters formulated with 100 g wheat flour and fried for different times. Concerning the variations in grey level intensities of surface images, Quevedo, Carlos et al. (2002) were able to compare the surface roughness of chocolate and pumpkin. They showed a more jagged map of surface intensity of pumpkin than the map of chocolate. The surface of some products like pumpkin is that rough that even the naked eye can recognize. However, as it is clear from Figure 7-1, the surface images of our batters and their intensity maps did not clearly show any difference, and they all showed approximately a same range of pixel intensity variation. Therefore, it was necessary to use more complex techniques such as fractal dimension and lacunarity measurements to study the irregularities of the surface of fried batters.

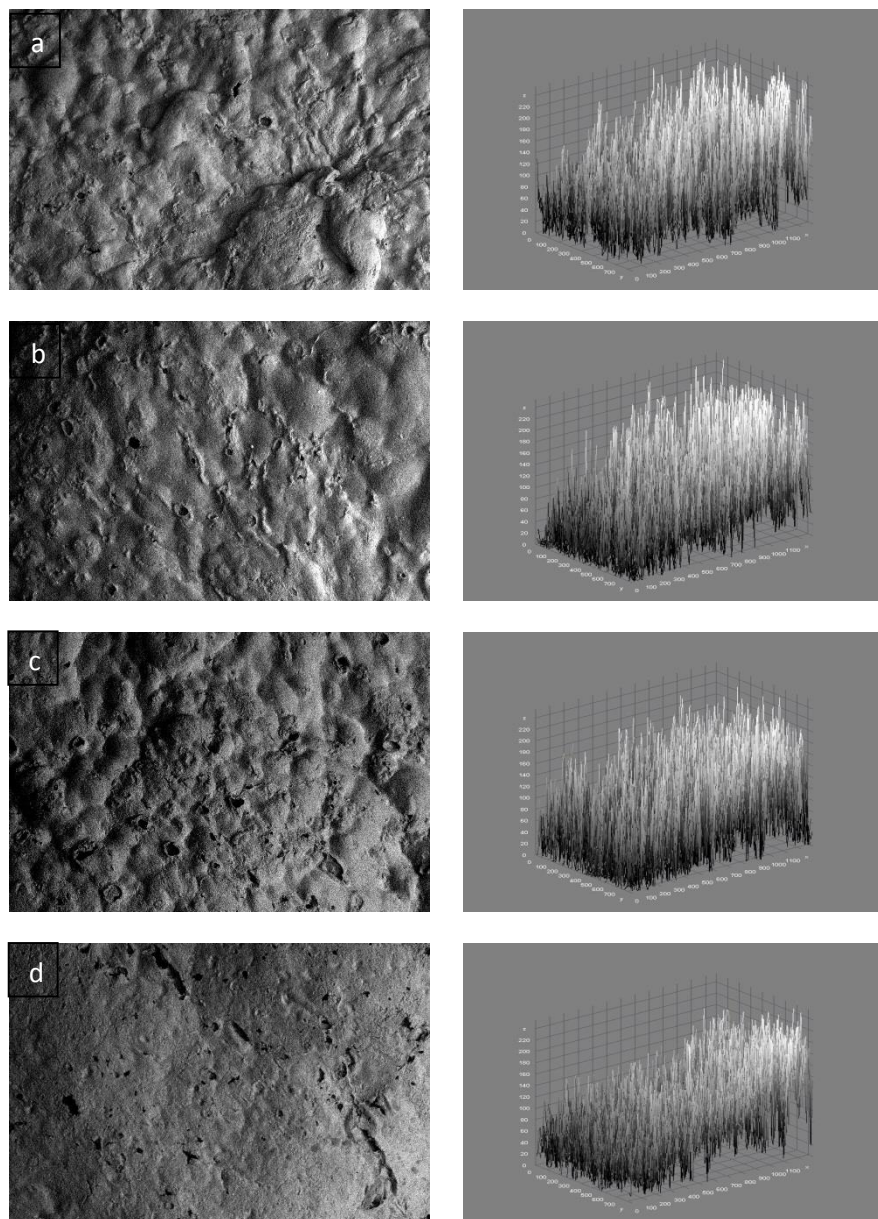


Figure 7-1 Images of the surfaces (left) and their respective gray level intensity plots (right) of 100 g wheat flour batter fried at different frying times. a: 1min frying, b: 2 min frying, c: 3 min frying, d: 4 min frying.

Fractal dimension (FD) was recently introduced as a good tool to describe the surface of foods (Quevedo, Carlos et al. 2002, Zheng, Sun et al. 2006, Moreno, Brown et al. 2010). Figure 7-2 illustrates the FD during frying of batters with different formulations. FD values varied in a very narrow range between 2.79 and 2.82. This narrow range indicates that within products with similar specification, such as these batters which are all restructured of flour and water, and then

fried, the fractal dimension does not show a big difference. The main effects of frying time and formulation were significant ($P < 0.05$) on FD of the batters. However, no interaction effect of frying time and formulation on variation was observed in FD at a confidence interval of 95%. Longer frying time showed significantly higher FD values. Higher wheat flour proportion in the formulation of batter coating also showed higher FD.

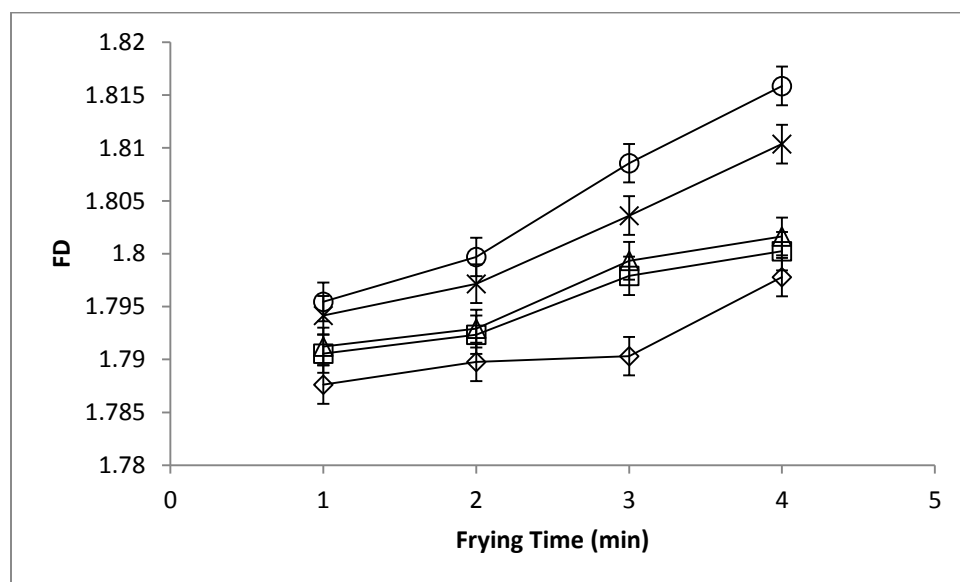


Figure 7-2 Fractal dimension during frying of batters with different formulations. O: 100W0R (100 g wheat flour and 0 g rice flour), ×: 75W25R (75 g wheat flour and 25 g rice flour), Δ: 50W50R (50 g wheat flour and 50 g rice flour), □: 25W75R (25 g wheat flour and 75 g rice flour), ◇: 0W100R (0 g wheat flour and 100 g rice flour).

Significant physical, chemical and structural changes occur during frying at the usual high frying oil temperatures. Water changes from liquid phase to vapor, evaporates at the product surface and leaves the product. Starch gelatinization and protein denaturation also occur at frying temperature in flour based products like batter (Addo, Xiong et al. 2001, Yusop, Maskat et al. 2011). These phenomena create a porous structure crust on the surface of fried batters (Barutcu, Sahin et al. 2009), which becomes thicker with increased frying times as more water was evaporated (Pedreschi, Aguilera et al. 2001, Yamsaengsung and Moreira 2002). Batters prepared

from higher amount of wheat flour were composed of more gluten protein than batters prepared from higher amount of rice flours. Therefore, different structural behavior of the developed crust was observed during the heating process (Champenois, Rao et al. 1998, Addo, Xiong et al. 2001). The different structural changes on the surface of batters with different formulations during frying explain the variations in roughness of the surfaces, and therefore different FD values were obtained.

FD versus moisture loss of batters with different formulations is plotted in Figure 7-3. Pearson's correlation showed that moisture loss had a high positive correlation with FD of all batters (coefficient of correlation ranged between 0.87 and 0.97). As mentioned, development of a dry surface, i.e. crust, is a major structural transformation occurring during the frying of foods. The formation of the crust greatly reduces the rate of moisture transfer and induces an increase in pressure inside the product (Yamsaengsung and Moreira 2002). The inner moisture is converted to vapor, creating a pressure gradient that leads to expansion of the pores and therefore, aggressive damage to the surface to create channels and ways for evaporation (Bouchon 2009). Pedreschi and Aguilera (2002) observed a drastic change in original shape of raw potato cells as a result of starch swelling and moisture loss at the crust region of the product during frying. In a study conducted in our laboratory on the surface rupture of batter during frying (results not published yet), it was observed that the ratio and number of rupture happened on the surface of batters significantly increased by higher amount of moisture loss. Therefore, the possible reason for higher FD value at higher moisture loss could be the structural changes during water evaporation.

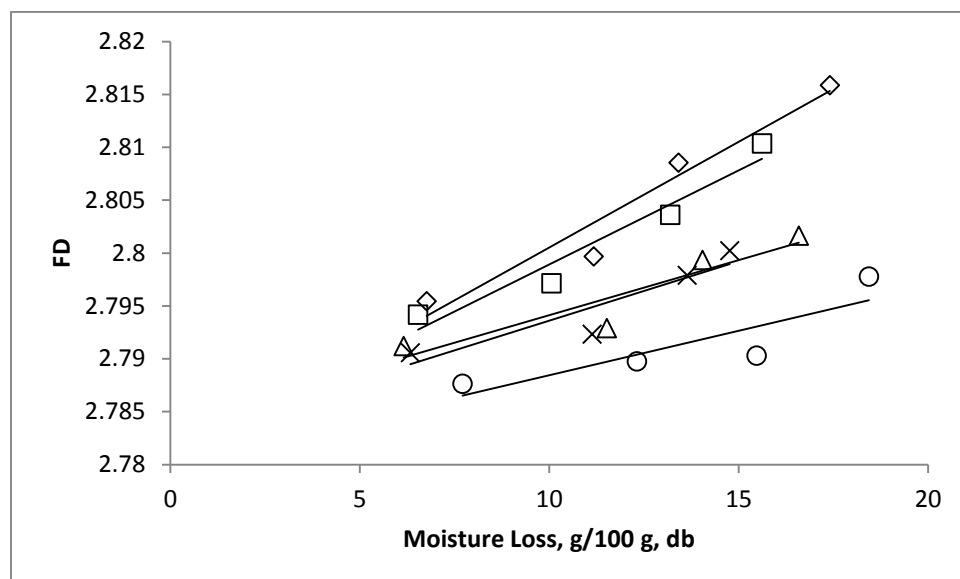


Figure 7-3 Plot of correlation between FD and moisture loss, db (%) of fried batters with different formulation. O: 100W0R (100 g wheat flour and 0 g rice flour), ×: 75W25R (75 g wheat flour and 25 g rice flour), Δ: 50W50R (50 g wheat flour and 50 g rice flour), □: 25W75R (25 g wheat flour and 75 g rice flour), ◇: 0W100R (0 g wheat flour and 100 g rice flour).

It is suggested that oil absorption is primarily a surface phenomenon, involving equilibrium between adhesion and drainage of oil as the food is removed from the oil bath (Bouchon, Aguilera et al. 2003). Three different oil fractions have been identified in fried foods namely structural, penetrated and surface oils (Durán, Pedreschi et al. 2007, Pedreschi, Cocio et al. 2008). Surface oil fraction represents the oil which remains on the surface during frying and might be absorbed during cooling phase, immediately after taking the food out of the fryer. Depending on the product, the fraction may constitute the highest amount (up to 80%) of the total fat content in the product. Therefore, it is crucial to study the correlation between surface roughness and the amount of surface fat content. Plots of correlation between FD and surface fat content of batters with different formulations are shown in Figure 7-4. Pearson's correlation showed that fat content had a high positive correlation with FD of all batters (coefficient of correlation ranged between 0.91 and 0.99). This confirms that a higher FD value, which shows a rougher surface, leads to higher amount of oil remaining on the surface of the batter and

consequently higher total fat content. Several studies have also demonstrated that the surface characteristics and specifically roughness play important roles in oil absorption (Thanatuksorn, Pradistsuwana et al. 2005, Moreno, Bouchon et al. 2010, Moreno, Brown et al. 2010). Moreno, Brown et al. (2010) worked on a food model comprised of various wheat flour and water mixtures, and reported that the products which showed rougher surfaces during deep fat frying retained more oil after the process. Similarly, Thanatuksorn, Pradistsuwana et al. (2005) observed that the surface roughness that was generated during frying of potato flake and wheat gluten based products increased the quantity of adhered oil during cooling period. These authors stated that the relationship between surface roughness and oil content is restricted to products with similar compositions, and a product with less irregular surface might show higher fat content than a product with higher irregular surface but different composition. In fact, surface irregularity is not the only factor affecting oil uptake, and the balance between oil that is in the crust and the residual surface oil is the result of a competition between capillary suction into the crust and drainage along the surface of the product. Consequently, some other factors such as initial moisture content (Rahimi and Ngadi 2014), micro-structure of the crust, particularly porosity and pore size distribution (Adedeji, Liu et al. 2011), and formation of surface active agents, and wetting and hydrophobic nature of the surface (Dana and Saguy 2006) may also play important roles in oil absorption.

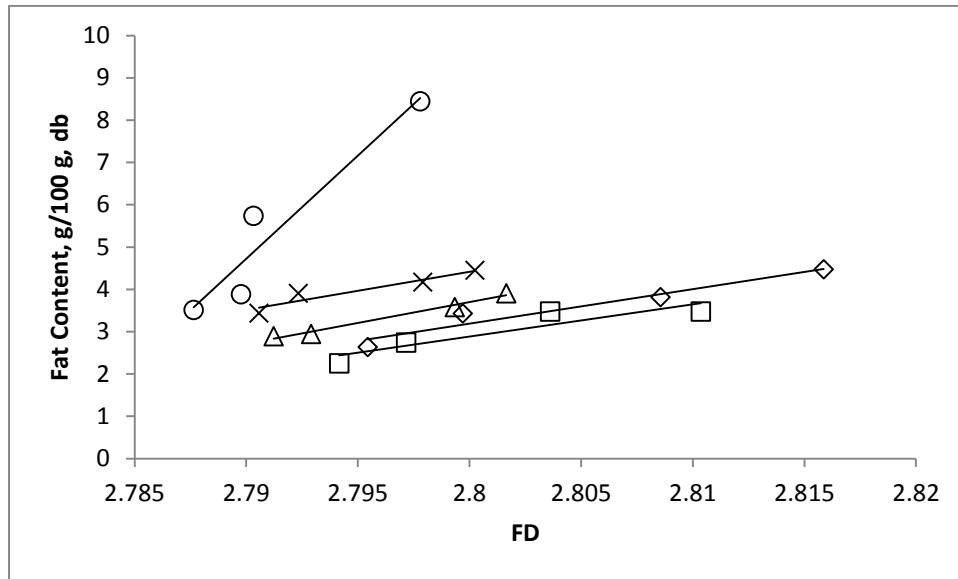


Figure 7-4 Plot of correlation between FD and fat content, db (g/100 g) of fried batters with different formulation. O: 100W0R (100 g wheat flour and 0 g rice flour), ×: 75W25R (75 g wheat flour and 25 g rice flour), Δ: 50W50R (50 g wheat flour and 50 g rice flour), □: 25W75R (25 g wheat flour and 75 g rice flour), ◇: 0W100R (0 g wheat flour and 100 g rice flour).

Lacunarity (λ) is basically developed to describe a property of fractal (Mandelbrot 1982) and measure the spatial distribution of data (Iqbal, Valous et al. 2011). It was used in this work to complement fractal dimension by computing how the data fill the space. In other words, it was applied to measure the heterogeneity of the structure or the degree of structural variance within batter surfaces. The study of surface rupture distribution by means of lacunarity could provide information about microstructure of the batters and how the property is related to surface damage due to water evaporation during frying. Lacunarity of all batter formulations was observed to be in a narrow range between 1.19 and 1.25. Lacunarity is always greater than 1. If it tends to 1, it means that the image approaches a homogenous structure (Velazquez-Camilo, Bolaños-Reynoso et al. 2010). Since lacunarity measures the variation of the pixel counts performed at each box size, it is correct to say that lacunarity reflects the deviation of the sizes of the ruptures on the batter surfaces. When all ruptures have the same area, lacunarity tends to 1. Otherwise, when there are large and small ruptures, and the deviation shows inhomogeneity of sizes, lacunarity

tends to larger values. This is the first reported use of lacunarity for the characterization of images of fried batter surfaces. However, there are some reports on application of this parameter in other food processing units. Velazquez-Camilo, Bolaños-Reynoso et al. (2010) reported almost the same range of lacunarity for the images of cane sugar crystals. Dàvila and Parés (2007) represented lacunarity as a powerful tool to analyze changes in microstructural properties of protein gels.

Figure 7-5 shows the lacunarity of batters with different formulation in function of frying time. In total, as it is clear, no meaningful trend was found for variation in lacunarity of images when only the batter formulation was considered, whereas frying time showed a significant decrease or increase in lacunarity. It can be observed that the lacunarity of surface images of 100W0R and 0W100R batters did not change during frying, while that of 75W25R and 50W50R significantly ($P < 0.05$) decreased; and that of 25W75R significantly ($P < 0.05$) increased. Lower lacunarity value obtained during frying of 75W25R and 50W50R shows that the ruptures generated on the surface became more homogenous in shape and size. In contrast, increase in lacunarity of 25W75R during frying indicates that the heterogeneity of the size of ruptures on the surface of these batters increased during frying. It reveals that the size of the vapor bubbles generated during frying in these batters was not uniform; and therefore created holes and cracks with different sizes when evaporating through the surface.

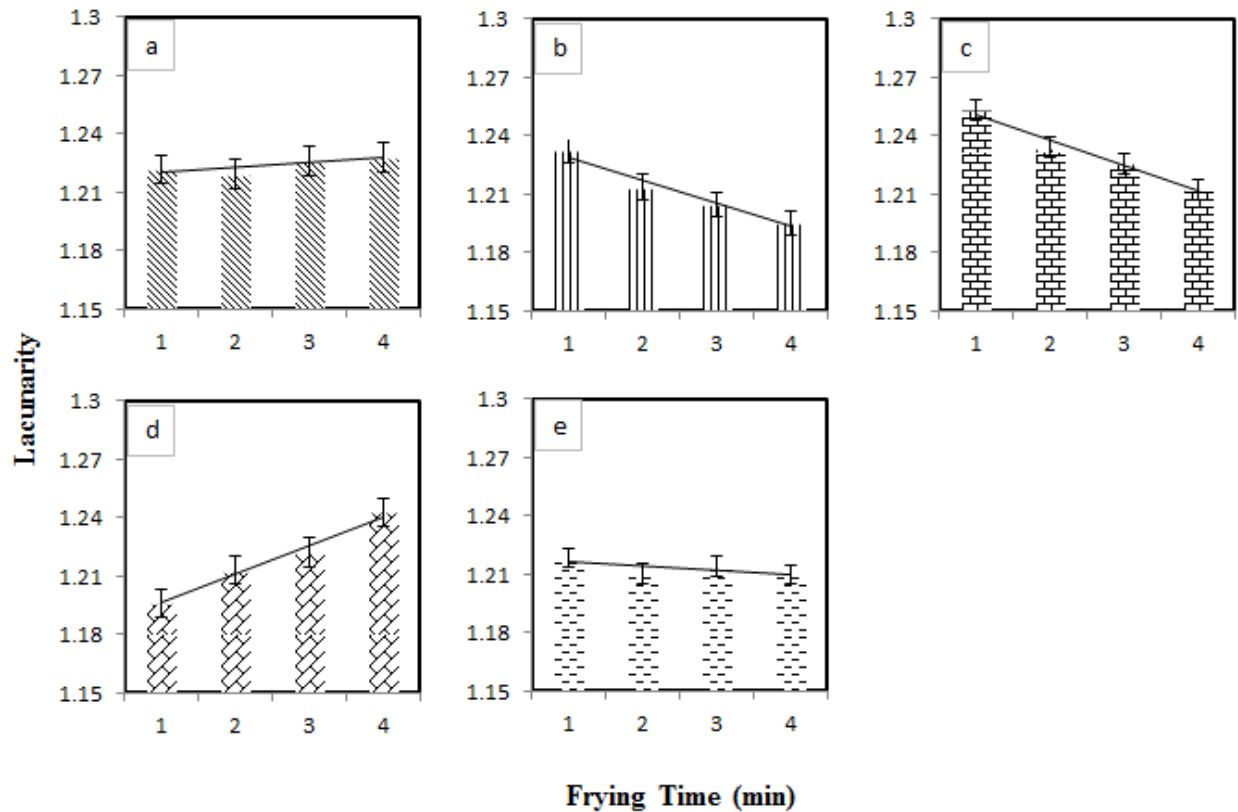


Figure 7-5 Lacunarity during frying of batters with different formulations. a) 100W0R (100 g wheat flour and 0 g rice flour), b) 75W25R (75 g wheat flour and 25 g rice flour), c) 50W50R (50 g wheat flour and 50 g rice flour), d) 25W75R (25 g wheat flour and 75 g rice flour), e) 0W100R (0 g wheat flour and 100 g rice flour).

In general, surface irregularities play a significant role in post-frying oil absorption in wheat and rice flour based batters. Therefore, it is very important to apply a good method to remove the oil which remains on the surface of food products during frying. Two approaches that were utilized to effectively reduce oil uptake after frying were blowing away the surface oil by using hot air (Dana and Saguy 2006), and blotting the surface with an adsorbent clean tissue. In two different studies conducted by the authors on the effect of batter formulations on fat absorption (chapters 3 and 4), it was observed that in the study that oil was mopped off after frying wheat-flour-based batter decreased the amount of oil absorption; whereas in the other study that oil was not removed rice-flour-based batter showed less oil content. In these studies, it

was also reported that around 80% of the total oil content was the fraction which remained on the surface during frying and was absorbed during cooling phase. It means that combination of two simple techniques i.e. 1) using wheat flour for batter preparation and 2) removing the surface oil could significantly reduce the amount of total fat uptake.

7.5. Conclusions

This study confirmed that surface roughness highly affects the amount of fat content in fried products, and post frying process is very important in decreasing this parameter. Batter formulation and frying time influenced the fractal dimension, which was found to be a useful tool in studying the surface roughness of fried batters. Higher the amount of wheat flour in formulation of batter significantly increased fractal dimension. Although the results of this study supported that surface roughness plays a crucial role in remaining oil on the surface of batters during frying, it is not the only parameter affecting oil content, and other parameter such as initial moisture content influence fat content. Lacunarity results suggested that the ruptures happened on the surface during frying was homogenous, and therefore batter formulation and frying time does not result a significant difference in shape and size of holes and cracks on the surface of these products.

CHAPTER 8

GENERAL SUMMARY AND CONCLUSIONS

Deep-fat frying is a widely used method for processing of batter coated foods. Batter frying forms a continuous and uniform layer over the food surface that provides a tasty and crispy texture crust. The crust acts as a barrier against the loss of moisture by protecting the natural juices of foods. Batter formula and processing methods, such as frying time, pre-heating temperature, and pre-drying time can influence the quality of the crust by controlling the moisture evaporation and fat absorption during frying. There is a lack of knowledge about how surface and microstructural changes during frying affect the mechanism of these mass transfers. Study of mechanism of moisture and fat transfers, when this barrier layer is applied, provides useful information for further quality improvement and more reduction of fat content in the final product.

In this study, mass transfer of deep-fat fried batter coating was studied when different combinations of wheat and rice flours, as the main ingredients, were used. Different pre-heating temperatures and pre-drying times were also applied to study their influence on mass transfer of batter during frying. Different fractions of fat in fried batters were evaluated to understand whether fat absorption happens mostly during frying or after frying. Inter-particle spaces of the fried batters are occupied primarily by three main components that are air, moisture, and fat. Therefore, fractions of these spaces as occupied by air, moisture, or fat were measured as a tool to better understand the mechanism of fat uptake. The geometry and number of ruptures that are generated during frying due to moisture evaporation were investigated as the open ways for fat absorption. Finally, surface irregularity and roughness of fried batters were evaluated as a very

important parameter in carrying fat on the surface of batter when it is removed from the oil medium.

The effect of pre-heating temperature, batter formulation, and frying time on porosity, moisture content, and fat content of batters fried at 180°C was initially investigated. All main parameters showed significant ($P < 0.05$) effects on moisture loss, fat uptake and porosity. Pre-heating significantly decreased the amount of fat absorption during frying. Pre-heating at 60°C showed the highest influence on reduction of fat uptake which could be because of less moisture loss and porosity created during frying. Therefore, this temperature was used to investigate the properties of batters formulated with different combinations of wheat and rice flours. Batters prepared from higher amount of wheat flour significantly decreased fat absorption when compared to batters prepared from higher amount of rice flour. Batters formulated with higher amount of wheat flour represented a stronger film barrier against moisture evaporation and showed less porosity than batters with higher rice flour content.

Different fractions of fat namely total fat content, fat penetrated to the inner structure, and fat remained on the surface of batter during frying were measured. Frying time and pre-drying had significant ($P < 0.05$) effect on moisture loss of batter coating during frying. Frying time, batter formulation, and pre-drying had significant ($P < 0.05$) effect on oil uptake of batter coating. A higher proportion of wheat flour showed higher surface and total fat content, and lower penetrated fat content. Pre-drying significantly ($P < 0.05$) reduced surface, penetrated and total fat contents. Moisture diffusivity and kinetics of fat penetration during frying were evaluated using different mathematical models. The Fick's diffusion equation, Page, and modified Page models adequately predicted kinetics of mass transfer in batters with different formulations and pre-dried at different times.

Evaluation different fractions of the spaces in a fried product that are occupied by air, moisture, or fat provides useful information for researcher and manufacturer to better understand the mechanism of oil absorption during frying.

Helium pycnometry technique was used to study the effect of batter formulation, pre-drying time, and frying time on inter-particle spaces of batters occupied by components namely air, moisture, and fat. Frying time, formulation, and pre-drying time showed significant ($P < 0.05$) effect on these fractions of inter-particle spaces. The spaces occupied by these three components increased during frying. Higher proportion of rice flour in batter showed higher fraction of inter-particle spaces occupied by air, and lower fraction of spaces occupied by moisture and fat. This means that more moisture loss during frying underwent in batters prepared from higher amount of rice flour; and therefore more air filled spaces was left after frying in these batters compared to batters formulated with higher amount of wheat flour. Bigger fraction of spaces was occupied by air, and smaller fraction of spaces was occupied by moisture and fat when longer pre-drying times was conducted. SOA, SOM, and SOF of the batter systems ranged from 2.20 to 46.03%, 2.55 to 47.07%, and 0.35 to 11.11%, respectively. The fraction of spaces occupied by air was highest followed by moisture and fat. The developed technique herein may be used for other food systems and studies.

Rapid moisture evaporation during frying generates some ruptures, such as voids, holes, and cracks on the surface of fried batters. VP-SEM technique showed a great potential in characterization of these surface irregularities. Analysis of the images captured from the surface of non-fried and fried batters with different combinations of wheat and rice flours showed some extensive ruptures formed on the surface of the batters. Batters formulated with only wheat flour showed the highest number of ruptures whereas batters formulated only with rice flour showed

the least number. Batter formulation significantly ($P < 0.05$) influenced the roundness of the ruptures. The area of holes and cracks, generated on the surface during frying, was increased by applying more amount of rice flour in batter formulation. It was observed that more severe ruptures on the surface of fried batters resulted in higher fat content, which means that these ruptures provide some open channels and ways for fat migration.

This study confirmed that the amount of fat that is remained on the surface of fried foods is highly dependent on surface roughness and post frying process is very important in decreasing this parameter. Batter formulation and frying time influenced the fractal dimension, which was found to be a useful tool in studying the surface roughness of fried batters. Batters formulated with higher amount of wheat flour showed significantly higher fractal dimension than batters with higher amount of rice flour. Although the results of this study supported that surface roughness plays a crucial role in remaining oil on the surface of batters during frying, it is not the only parameter affecting oil content, and other parameter such as initial moisture content influence fat content. Lacunarity results suggested that the ruptures happened on the surface during frying was homogenous, and therefore batter formulation and frying time does not result a significant difference in shape and size of holes and cracks on the surface of these products.

Batters prepared with a higher amount of wheat flour showed higher fat content when no post frying processing was applied to remove the excess fat from the surface. In contrast, it was observed that batters with higher amount of wheat flour showed significantly less fat content than batters prepared from higher amount of rice flour when the excess fat on the surface was removed by mopping of the surface. This finding showed that wheat flour batters carry more fat on its surface than rice flour batter when it is removed from the oil medium. Study of surface roughness of batters with different formulation confirmed this observation where batters

prepared with higher amount of wheat flour showed higher fractal dimension. It can be concluded that post frying process is very critical in controlling the amount of fat content in fried products. Therefore, wheat flour can be an effective ingredient in reducing fat content of fried breaded products when an appropriate post frying process is applied.

CHAPTER 9

CONTRIBUTIONS TO KNOWLEDGE

This thesis was designed to contribute to the scientific knowledge by studying the microstructure and surface irregularities of fried batter coatings and finding the correlation between these parameters and fat absorption during frying. The following are a few of the several commendable contributions of this research.

1. Pre-heating at 60°C was found to be an effective method in reducing fat adsorption during frying. Pre-heating causes starch gelatinization and protein denaturation which form a barrier film. The barrier film prevents moisture evaporation and oil uptake. Temperatures higher than 60°C weaken the generated film and increase fat absorption.
2. Wheat flour was used as an appropriate ingredient in formulation of batter to reduce fat absorption during frying, especially when a post frying process was applied to remove the excess fat on the surface of fried batters.
3. Pre-drying was introduced as a pre-treatment technique to reduce initial moisture content of batter coating and to significantly decrease fat content of fried batter.
4. Different fractions of fat in fried batter were for the first time. Fat absorption was observed to be mostly a surface related phenomenon and a big part of fat was absorbed after frying process and during cooling phase from the surface.
5. Studying the surface roughness of batters showed that surface of batters prepared with higher amount of wheat flour was rougher than surface of batters prepared with higher amount of rice flour. This confirmed that wheat flour batter carried more fat on its surface than rice flour batter after removing the fried batters from fryer. However, wheat flour batters absorbed less fat than rice flour batters during frying. Therefore, it was

recommended to apply post frying techniques such as mopping the surface of fried foods or blowing air on the surface in purpose of removing extra fat from the surface to reduce the total fat content.

6. A useful protocol for evaluation the different fractions of inter-particle spaces of fried batters was developed. Using this protocol, three different fractions of spaces when occupied by air, fat, and moisture were for the first time reported. The developed technique can be used for other food systems and studies.
7. Lacunarity as a measure of batter surface irregularities was studied. SEM images captured from the surface of fried batters were analysed using ImageJ software, and useful information was extracted. The obtained information was used to study the geometry of generated ruptures on the surface of fried batters due to moisture evaporation during frying.
8. VP-SEM imaging was confirmed to be an appropriate technique to study the surface irregularities of fried batter.

CHAPTER 10

RECOMMENDATIONS FOR FUTURE STUDIES

This study has explored promising pre-treatment techniques for decreasing fat uptake during frying of batter coating. It has also evaluated the correlation between microstructure and surface irregularities of batters and fat absorption. However, fat absorption during frying is a complex phenomenon and there is still a need for future studies to understand it better. Some of the recommendations for future research are as follows:

1. All studies have been conducted on frying of batter coating. Using the aluminum cells which were used in this study, it is recommended to investigate the influence of core part formulation (food model) on oil absorption of batter coating and core part during deep fat frying.
2. Study of the distribution of the components of a fried food provides useful information to understand the mechanism of oil absorption. Therefore, it is recommended to identify the distribution of different components of fried batter samples by Fourier transform infrared spectroscopy (FTIR).
3. It has been reported that oil absorption is mostly a surface phenomenon and a big part of oil is absorbed from the surface of fried foods and during cooling phase. In addition to surface roughness, the wetting characteristics of food surfaces and oil/food surface tension might also play an important role in the remaining oil on the surface. Then, study of the wetting characteristics of food surfaces is recommended to understand the fat absorption mechanism.

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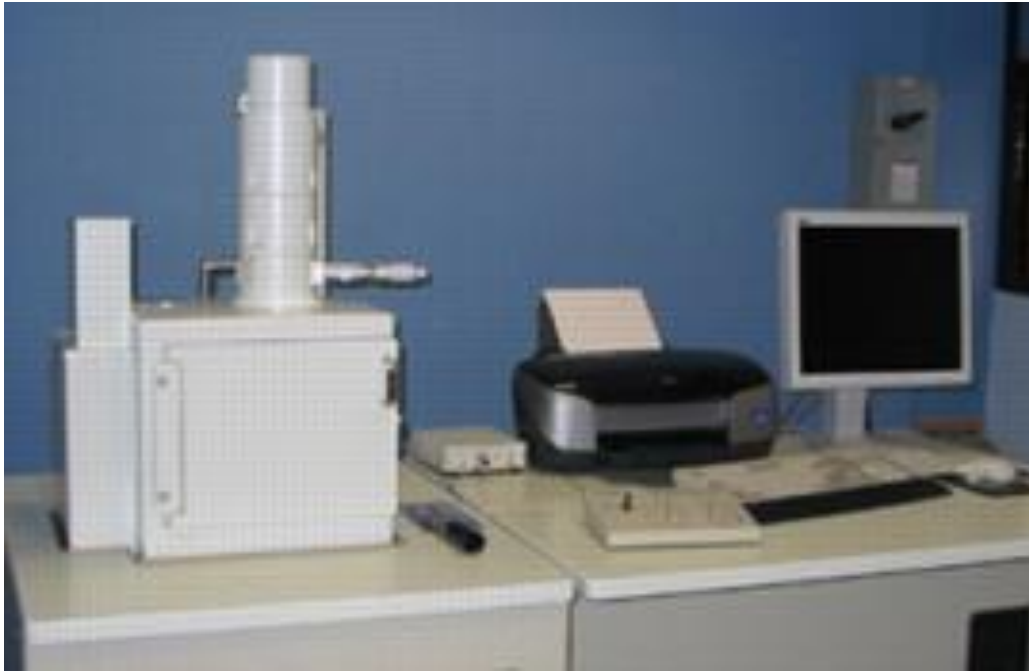
APPENDICES

Appendix 1. MATLAB codes used for surface rupture

characterization.

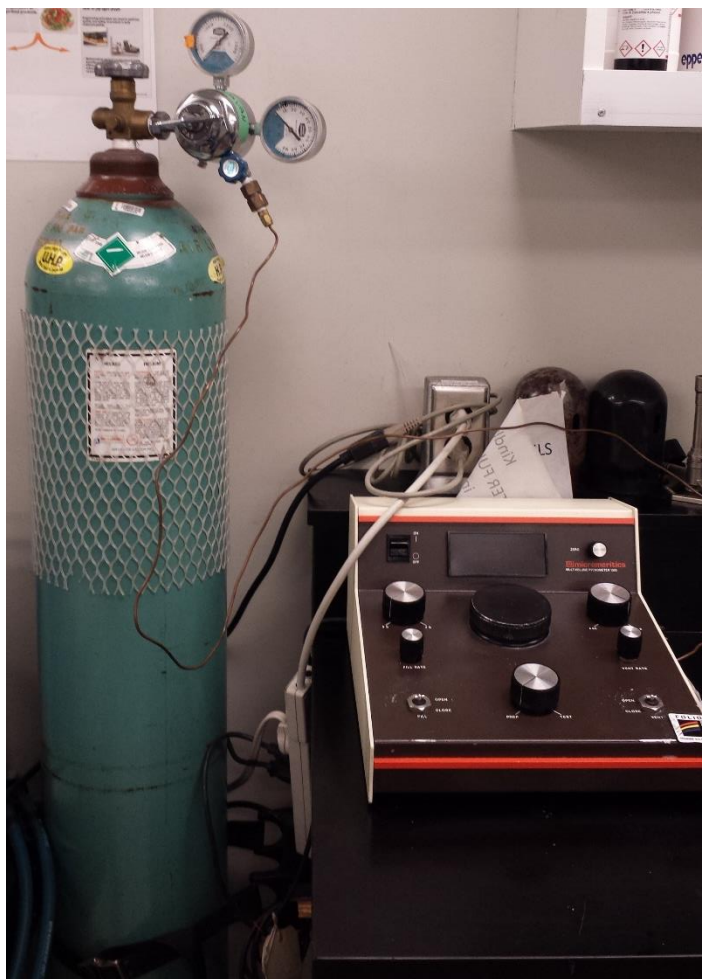
```
%% This codes are to measure some properties of batter surface images.
% peoperties like number of objects of interest in the image, area of the
% objects, and some statistical properties like max, min, mean, and
% histogram
%%% to read the image, first the imread function is run in MATLAB command
>window and then the flollowing command are run;
%%% to binarize the image( the thresholding level is obtained useing below
% procedure:using pixel region function on imtool, it was found the pixel
%with inensity equal and less than 20 are the holes and therfore 25 was
%divided to 256 (8 bytes grayscale images (0 - 255)), so 0.082 was decided
%as the thresholding level.
%%%to binirize the image:
imshow (I);
bw = im2bw(I, 0.);
%%% to invert the image:
bwinv = ~bw;
%%%to remove the noise
rem = bwareaopen(bwinv,5);
figure;
imshow(rem);
%the threshold was selected 5 because using imtool it was found that white
%dots smaller than 5 pixles are noise.
%%%to measure the number of objects:
[labeled, numobjectcs] = bwlabel(rem,8);
% the number 8 in the command above is about the connectivity of pixels.
% to obtain some morphological properties:
data = regionprops(labeled, 'all');
%%%statistical analysis
numobjectcs
maxarea= max([data.Area])
meanarea = mean([data.Area])
figure;
hist([data.Area],20)
maxperimeter = max([data.Perimeter])
meanperimeter = mean([data.Perimeter])
Roundness = (4*meanarea*pi)/(meanperimeter.^2)
%to measure to ration:
a = sum(sum(rem));
b = 960*1280;
ratio=a/b*100;
ratio
```

Appendix 2. Variable-Pressure Scanning Electron Microscopy.



The scanning electron microscope (JEOL JSM-6460LV Variable Pressure Scanning Electron Microscope (LV-SEM)) shown in this picture was used for this study. It is located in Laboratory of Calcified Tissues and Biomaterials, Department of Stomatology, Faculty of Dentistry, Université de Montréal, Montreal, Canada.

Appendix 3. Helium Pycnometer.



The helium pycnometer (Model 1305 Multivolume, Micromeritics Instrument Corporation, Norcross, GA) is used from Laboratory of Food and Bioprocess Engineering, Department of Bioresource Engineering, Faculty Agriculture and Environmental Sciences, McGill University, Montreal, Canada.

Appendix 4. Freeze Dryer.



The freeze dryer (Modulyod-115; ThermoSavant, Holbrook, NY, USA) is located in Laboratory of Food and Bioprocess Engineering, Department of Bioresource Engineering, Faculty Agriculture and Environmental Sciences, McGill University, Montreal, Canada.

Appendix 5. Solvent Extraction Unit.



The solvent extraction unite (Velp Scientifica, Usmate, Italy) solvent extraction unit is located in Laboratory of Food and Bioprocess Engineering, Department of Bioresource Engineering, Faculty Agriculture and Environmental Sciences, McGill University, Montreal, Canada.