CHAPTER 2 BACKGROUND

This chapter provides the background and literature related to the present research. The first part of the chapter covers a brief review of the various drying methods, which were used in this study, including hot air drying, vacuum drying and low-pressure superheated steam drying. The second part of the chapter focuses on the relationship between microstructural and physical characteristic changes of foods during drying. Finally, some basic information on fractal analysis is given.

2.1 Drying of Foods

Drying is one of the oldest methods of food preservation. Water removal reduces the water activity and hence microbiological activity in food; physical and chemical changes during storage are also minimized (Mayor and Sereno, 2004). Nowadays, drying is also a method for increasing the value of food. Although all drying methods share the same basic principles of supplying heat and transporting moisture from a food product, different methods of drying affect the quality of dried food differently (Sokhansanj and Jayas, 2007); the different quality of dried food has an influence on the consumer acceptance.

The quality of dried food is often based on physical characteristics such as shrinkage and texture, which are directly affected by food microstructural changes. As a result, it is important to understand and be able to assess the effects of different drying methods on the physical characteristic and microstructural changes. Although many types of drying methods are applied to dry foods, in this study, three representative

drying methods were studied, i.e., hot air drying, vacuum drying and low-pressure superheated steam drying.

2.1.1 Hot Air Drying

Hot air drying is one of the simplest and most widely used alternatives for food dehydration because the technique requires low operating cost and is easy to operate. Many investigators have therefore studied the use of this simple technique to dry a wide array of food products.

Generally, hot air drying leads to significant microstructural and physical changes of food. For example, Lin et al. (1998) studied the effect of hot air drying on textural properties of carrot slices and compared the results obtained with that from vacuum microwave drying and freeze drying. Carrot slices were dried to a final moisture content of around 10% (d.b.). The results showed that the drying methods significantly affected the hardness of carrots. The hardness of carrots undergone hot air drying was higher than that of carrots undergone vacuum microwave drying and freeze drying, indicating that much case hardening occurred in the case of hot air drying. This is because during hot air drying liquid diffused and solutes were carried from the interior to the surface of the sample. As the moisture evaporated from the sample surface, solutes concentrated and precipitated. The skin of the sample was hence hard and dry.

Ochoa et al. (2002) studied the effects of hot air drying on shrinkage of whole sour cherry fruits. Drying was performed at temperatures of 50, 55, 60, 65, 70 and 75 °C; air velocity of 0.1, 1, 2, 3 and 5 m/s as well as air relative humidity of 5 and 50%. It was

found that only the drying temperature significantly affected the drying time. The volume and surface area changes of the samples were independent of the tested drying conditions but depended on the moisture content of the sample. A linear relationship between the dimensionless volume change (V/V_0) and the moisture content of the partially dehydrated sample was noted. Similar results were also reported for bamboo shoot and potato during hot air drying (Madamba, 2003; Khraisheh et al., 2004).

Witrowa-Rajchert and Rząca (2009) found that shrinkage of a sample (apple) dried by hot air drying was the highest when comparing with samples dried by microwave-convective drying and infrared drying. Furthermore, the results showed that shrinkage was related to the microstructural changes of the samples in all cases. SEM images showed that the sample dried by hot air drying had numerous breaks of cell walls. High temperature of hot air drying led the microstructure with much damage when comparing with the structures of the sample dried by microwave-convective drying and infrared drying.

In addition to its disadvantages when viewing from the poor product physical characteristics, the presence of oxygen in hot air also contributes to much quality degradation in terms of color, flavor and nutrients (Nijhuis et al., 1998; Araya-Farias and Ratti, 2009). In order to reduce the poor quality of food, alternative drying methods have been proposed.

2.1.2 Vacuum Drying

Vacuum drying utilizes the concept of drying at lower temperature, which is possible due to the lowering of the boiling point of water through the reduction of the system pressure (Sokhansanj and Jayas, 2007). Since the drying chamber is partially evacuated, removal of moisture takes place under the environment of lower oxygen content, leading to reduced oxidative degradation, e.g., enzymatic browning, of a final product. As the drying temperature is kept low, materials that are sensitive to oxygen and heat, like fruits and vegetables, can be more effectively dried. Vacuum also helps expand air and water vapor present in food and creates puffed structure; the final product properties depend on the level of vacuum, however (Jaya and Das, 2003). Vacuum drying has been successfully applied to many fruits and vegetables and other heat-sensitive foods. Dried foods are characterized by better retention of nutrients and volatile aroma as well as by less deformation and better rehydration characteristics (Tsami et al., 1999; Sokhansanj and Jayas, 2007).

A number of investigators have studied vacuum drying of many food products in terms of their physical characteristics. Wu et al. (2007), for example, studied shrinkage of eggplants during vacuum drying. It was found that shrinkage was not dependent on the drying temperature and a linear relationship between the volume change and the moisture content was found. On the other hand, Panyawong and Devahastin (2007) noted a second-order relationship between the volume change and the moisture content of vacuum dried carrot. Nevertheless, if the information during the first period of drying was neglected, the trend of such relation would be similar as that reported by Wu et al.

(2007) and other investigators who studied hot air drying (Ochoa et al., 2002, Madamba, 2003, Khraisheh et al., 2004).

Zotarelli et al. (2012) found that shrinkage of the samples (banana and mango) dried by vacuum drying was less than those dried by hot air drying. These results were related to the microstructural changes of the samples because large moisture gradients developed within the samples during hot air drying led to microstructural stresses and collapse of most capillaries that in turn led to shrinkage. Vacuum drying, on the other hand, created a porous structure of the samples and hence less shrinkage. SEM images of the sample microstructure have indeed shown that the samples dried by vacuum drying had larger pores than those dried by hot air drying.

2.1.3 Low-Pressure Superheated Steam Drying

A concept of low-pressure superheated steam drying (LPSSD) has been proposed as an alternative to drying heat-sensitive products since the technique combines the advantages of drying at reduced temperature to those of superheated steam drying (SSD) (Devahastin and Suvarnakuta, 2008). The notable advantages of SSD that are of interest to food industry include the absence of oxidative reactions (e.g., enzymatic browning, lipid oxidation) due to lack of oxygen, high drying rates in both constant and falling rate periods, depending on steam temperature and pressure, and its ability to yield a high porosity dried product due to an evolution of steam within the product. However, most foods or other heat-sensitive products are damaged at the saturation temperature of superheated steam corresponding to the atmospheric or higher pressures. Therefore, LPSSD is operated at reduced pressure to obtain superheated steam at lower

temperatures to prevent the products from being damaged in a high-temperature environment of atmospheric-pressure SSD.

Devahastin et al. (2004) were the first to study both the drying kinetics and quality (e.g., volume, shrinkage, color and rehydration ratio) of food viz. carrot cubes undergoing LPSSD. Vacuum drying experiments were also conducted to compare the results with those of LPSSD. It was found that although LPSSD required longer time to achieve the same final moisture content of carrots than vacuum drying, LPSSD led to the product with superior quality attributes than vacuum drying.

Since then a number of studies have been made on LPSSD of different food products, both from the fundamental and application points of view (Barbieri et al., 2004; Methakhup et al., 2005; Leeratanarak et al., 2006; Pimpaporn et al., 2007; Kingcam et al., 2008). The use of LPSSD is thus recommended because it leads to the many advantages in terms of the quality of dried food products.

In an interesting study of Panyawong and Devahastin (2007) who investigated the relationships between the volume change and moisture content of carrots undergoing vacuum drying and low-pressure superheated steam drying it was shown that the trends of shrinkage in both drying cases were different. Although the two samples had similar volume changes, when comparing the results at the same drying temperature, the sample dried by vacuum drying deformed more rapidly and shrunk less uniformly than that dried by low-pressure superheated steam drying. This result supports that of Devahastin et al. (2004) who observed that the carrots dried by vacuum drying had a rather dense

layer, while the sample pore distribution was rather nonuniform as compared to that of the sample dried by low-pressure superheated steam drying.

From the above-mentioned arguments, it is recognized that the different drying methods affect the quality changes of foods differently. Although it is well recognized that physical characteristic changes are due to changes of the food microstructure, not much quantitative information is available on the effects of drying methods on the changes of microstructure, which in turn affects many physical characteristics of food. For this reason, the relationships between the microstructural and physical changes of dried food should be investigated.

2.2 Relationships between Microstructural and Physical Changes of Foods during Drying

During drying moisture migrates from the inner cells through the porous structure to the surface of food and then to the surrounding environment. Loss of moisture leads to deformation of food microstructure; this in turn leads to changes of physical characteristics (i.e., shrinkage and hardness) of dried food, which directly affect its quality and consumer acceptance. Therefore, in order to control food quality, it is important to investigate the relationships between microstructural and physical changes of food during drying; the relationships should be useful as they can be used to help design and identify an appropriate drying process and condition to obtain a high-quality dried food product.

2.2.1 Microstructural Changes of Foods during Drying

Prior to being able establish the relationships between microstructural and physical changes of food, an appropriate means must be identified to obtain quantitative information on the microstructural changes, which are more difficult to obtain compared with the apparent physical characteristics.

In general, the microstructure of food can be characterized in the form of images using various techniques, e.g., light microscopy (LM), scanning electron microscopy (SEM) or stereomicroscopy. These techniques are very useful tools to observe changes that take place during the drying process. Several studies have indeed reported the use of such techniques to investigate the microstructural changes of food during drying; investigations have also been made to study the relationships between microstructural and physical changes of various food products undergoing different drying processes at different conditions.

Mayor and Sereno (2005), for example, studied microstructural changes of apple slices during hot air drying and related these changes to the product shrinkage. The microstructural changes of apple tissues were represented by the changes of cell size as well as the changes of the cell shape. It was found that the size (cell area and intercellular spaces) and shape factor (roundness) decreased linearly with the moisture content during drying. Moreover, the apparent dimensional change (shrinkage) of apple slices, which represents the macroscopic change, were closely related with the size and shape changes of the sample cells.

Nimmol et al. (2007) used SEM to study microstructural changes of banana slices undergone combined vacuum drying and far-infrared radiation (VACUUM-FIR) and combined LPSSD and far-infrared radiation (LPSSD-FIR). SEM images showed that banana slices dried by VACUUM-FIR at lower drying temperature (80 °C in this case) had more dense structure (smaller and less pores), leading to harder texture than those dried by LPSSD-FIR. Léonard et al. (2008) later applied X-ray microtomography coupled with image analysis to evaluate the microstructural information of banana slices undergone both LPSSD-FIR and VACUUM-FIR. These investigators characterized the pore structure, in terms of the total pore volume, pore size distribution and porosity. The results showed that higher drying temperature and the use of FIR could increase the final porosity of the samples and the pore size distribution shifted toward larger pore sizes. Although the structural image of banana slices dried by both methods was similar (in the case of drying 90 °C), the porosity value of the LPSSD-FIR sample was higher than that of the VACUUM-FIR sample. This is due to rapid increase in the sample temperature during an initial stage of LPSSD-FIR, resulting in rigorous boiling of moisture within the sample.

Acevedo et al. (2008) studied the relationships between microstructural changes of apples and their physical properties during vacuum drying and freeze drying. Environmental scanning electron microscopy (ESEM) was used to observe the microstructure of the samples. It was found that microstructural changes were directly related to the final texture (hardness) of the samples. When comparing the two drying methods the results showed that the samples dried by vacuum drying had higher cellular collapse and tissue disruption than those dried by freeze drying. The collapse and disruption led to a higher mechanical resistance, hence higher hardness values when the

results were compared at the same moisture content. Nevertheless, the observed microstructural changes was not quantified.

Askari et al. (2009) studied microstructural changes of apples and strawberries underdone hot air drying and microwave-assisted hot air drying. SEM was used to observe the sample microstructure. It was found that the surface of samples dried by microwave-assisted hot air drying was more rigid due to greater migration rate of soluble solids and more case hardening, leading to higher values of hardness than the samples dried by hot air drying. However, the information on microstructural changes was not again quantified.

It can be seen from the above sample studies that despite investigations on relationships between microstructural and apparent physical changes of food, not much quantitative information is available to describe in detail such relationships. Although microstructural changes of food can be investigated using various techniques, it is not easy to quantify the microstructural changes without the use of appropriate evaluation algorithm. Recently, fractal analysis is among the techniques that has been proposed to tackle this difficulty.

2.3 Fractal Analysis

2.3.1 Definition of Fractals

Fractals or fractal geometry refers to geometry of complex and irregular objects, which are found in nature such as coastlines, clouds, rivers, mountains, islands, plants, etc.

(Mandelbrot, 1983). The dimensions of fractals can be non-integer and are called fractal dimension. Fractal dimension (FD) contains three characteristics, which consist of being described as self-similarity, not being described by a mathematical formula but by recurrent dependencies and containing a dimension that is not an integer (Dziuba et al., 1999). Hence, fractal geometry can be used to describe irregular objects. Fractal dimension consists of the dimensionless unit; the value of FD is in the range of 1-2 if an image has two dimensions, while if an image has three dimensions, the value is 2-3. For example, FD of a straight line is equal 1, while the FD of a plane is 2.

Fractal dimension can be calculated by many methods such as the box counting method (BCM), fractal Brownian motion method (FBMM) and frequency domain method (FDM). BCM is nevertheless one of the most popular methods used to calculate fractal dimension of images (Turner et al., 1998). In such a case FD can be calculated according to the following equation:

$$\mathrm{FD} = \lim_{r \to 0} \frac{\log(N_r)}{\log(\frac{1}{r})} \tag{2.1}$$

Cubic boxes of different sizes (r) are mounted into an image. The number of boxes intercepted with the image for each iteration of r value (N_r) is counted. Fractal dimension is determined from the slope of the least-square linear regression of the logarithmic plot of N_r versus $1/r$ (Quevedo et al., 2002).

2.3.2 Applications of Fractal Analysis in Foods

Several researchers have used fractal analysis to study microstructural changes of foods undergoing different processing. Fractal dimension has indeed proved to be capable of being a quantitative indicator of the microstructural changes of many food products such as cane sugar and cocoa butter undergoing crystallization, dough undergoing baking, protein gels preparing at different pH conditions and shrimp undergoing boiling (Marangoni and McGauley, 2003; Dàvila et al., 2007; Niamnuy et al., 2008; Pérez-Neito et al., 2010; Velazquez-Camilo et al., 2010).

Quevedo et al. (2002), for example, conducted the fractal analysis of the microstructure of potato cells and used the calculated fractal dimension to describe the microstructural changes of potato during frying. Fractal dimension was noted to be useful in numerically describing the microstructural changes of foods during processing.

Marangoni and Narine (2002) used fractal dimension to quantify the microstructure of fat crystal networks formed during the crystallization process. Fractal dimension of polarized light microscopic images were determined by the box counting method. It was found that fractal dimension of the crystal network structure was closely related to the polymorphism of the solid state.

Marangoni and McGauley (2003) established the relationship between crystallization behavior and structure in cocoa butter using fractal dimension. Fractal dimension of the polarized light micrographic images was used to quantify the microstructure of cocoa butter crystallized at different temperatures for up to 45 days. It was found that fractal dimension values were in the range of 1.47-1.88 at crystallization temperatures of -14 to

26 °C; the values changed with the microstructural changes of the sample and the crystallization kinetics. Brunello et al. (2003) later applied fractal dimension to correlate the microstructural changes to mechanical properties of cocoa butter crystallized at different temperatures (5-24 °C). It was found that fractal dimension of microstructural change images was closely related to the mechanical strength of the samples.

Dàvila et al. (2007) investigated microstructural changes of plasma protein gels at different pH conditions using fractal analysis. It was found that the values of fractal dimension of SEM images of plasma protein gels increased with pH, indicating that fractal dimension could be used to monitor the growth of protein aggregates during gelation. Fractal dimension was also found to positively correlate with the hardness of plasma protein gels.

Niamnuy et al. (2008) also developed relationships between microstructural changes, in terms of fractal dimension, and physical changes, in terms of cooking loss and hardness, of shrimp during boiling in salt solution. It was found that the normalized change of fractal dimension correlated well with the cooking loss and hardness, indicating that the changes of the shrimp microstructure had a direct effect on the apparent changes of shrimp.

Velazquez-Camilo et al. (2010) applied fractal analysis to monitor the crystallization process evolution of cane sugar. Fractal dimension was used to quantify the rough surface of cane sugar crystallization images at different crystallization time. It was found that fractal dimension of crystallization images of the samples increased with the crystallization time, which is also related to the formation of larger clusters. Fractal

dimension could thus be considered as an indicator of the amount of the formed crystals.

Pérez-Neito et al. (2010) studied structural changes of dough/crumb during bread baking. Fractal dimension was applied to characterize the texture of the crumb and the perimeter of the pores. It was found that both fractal dimension of the texture of crumb and fractal dimension of the perimeter of the pores were capable of being a quantitative indicator of bubble coalescence during baking.

In terms of drying Kerdpiboon and Devahastin (2007) were among the first to propose the relationships between microstructural and physical changes of food during drying using fractal dimension. The investigators developed relationships between microstructural changes, which were represented quantitatively in terms of the normalized change of fractal dimension of the microstructural images ($\Delta FD/FD_0$), and physical characteristics, which were represented in terms of the percentage of shrinkage and rehydration ability, of carrot cubes during hot air drying and low-pressure superheated steam drying. The results showed that the tested physical changes correlated well with $\Delta FD/FD_0$ of the microstructural images. However, such relationships have not yet been generalized and are specific to the tested processing methods and conditions.