1.1. INTRODUCTION

Fruit and vegetables are low in energy and rich in vitamins, antioxidants, minerals and fibre. Anthocyanins have anti-inflammatory effects. Both alpha-carotene and betacarotene are protective against liver cancer and lung cancer in cell culture and animal studies. However, they can only obtain at the special season and spoil too quickly. Drying fruit/vegetable chips can be solved this problem. The market for dehydrated food is important for most countries worldwide (Funebo and Ohlsson, 1998), For example, US\$7.6 billion worth of dehydrated vegetables, instant dried soup, and seaweed were consumed annually in Japan in 1998, excluding uses in restaurants and institutions (Japan Statistics Bureau, 2000). In China, the production of dehydrated vegetables is worth about US\$800 million, including US\$420 million for dehydrated red pepper, about 60–70% (about 230,000 tons) for export (Liu, 2003). In Europe the market for dehydrated vegetables was estimated to be worth US\$260 million in the early 1990s (Tuley, <u>1996</u>). In the United States, there is a large market for dehydrated grapes (raisins), garlic, onions, and tomatoes (Liu, 2003). The growth in popularity of convenience foods, such as dehydrated soup mixes and instant noodles, in many Asian countries has stimulated increasing demand for high-quality dehydrated vegetables and fruits.

Freeze drying (FD) accomplishes water removal by sublimation at very low temperatures and pressures which ensures high quality of the dried products. Compared with other dehydration methods, the FD technique has important characteristics such as ability to retain original structure and color, negligible loss of nutrients, and excellent rehydration capability due to the porous structure of the product (Jiang et al., 2010a). Despite many advantages of freeze drying, the process is not commonly used in food processing because of its high cost. Conventional freeze drying requires a longer drying time, which leads to high energy consumption and high capital cost. Freeze drying costs can be 200%-500% higher than that of hot-air drying if the final moisture content is the same (Potter and Hotchkiss, 2001). As a result, FD always process medicaments, but for food raw materials rarely.

Microwave can penetrate materials and heat products without the aid of thermal gradients, which has a positive effect on dehydration (liang et al., 2010b). Microwave combined with FD, which is called microwave-assisted freeze drying (MFD), can be a potential new drying method to process food, and reduce the cost remarkably. Some investigations on MFD for several heat-sensitive materials have been conducted in the past several years. Jiang (2010a, 2010b) investigated the drying characteristics of banana chips processed by MFD, and the Physicochemical changes during processing. The result showed that the most suitable operation parameter was that the microwave was 2.5W/g, and the temperature was 55 °C, and also showed that biggest changes are found at the primary drying stage. Wang et al. (2010a) studied the effect of three different food ingredients viz. NaCl, sucrose and sodium glutamate on microwave freeze drying (MFD) of instant vegetable soup, the results showed that NaCl content and sucrose content had significant influence on drying time and sensory quality, while sodium glutamate content had insignificant effect. Jiang et al. (2010c) also did some research about comparison of the effect of MFD and microwave vacuum drying (MVD) upon the process and quality characteristics of potato banana re-structured chips, for the quality of products, MFD samples were far superior to the MVD samples but the drying time and cost was

worse. In general, compared with FD, MFD can meet the four major requirements in drying of foods: speed of operation, energy efficiency, cost of operation, and quality of dried products. The increased demand for plant-origin foods in fast-dehydrated form has increased interest in MFD and has potential to replace conventional FD (<u>Zhang and Xu</u>, <u>2003</u>). Following a brief discussion of MFD, the effects of different operating parameters, the storage stability of MFD products, and the advantage of MFD compared with the traditional FD are presented in subsequent sections.

1.2. BASIC PRINCIPLE OF MICROWAVE FREEZE DRYING

Microwaves are part of the electromagnetic spectrum. Microwave energy absorption in foods primarily involves two mechanisms: dipolar relaxation and ionic conduction, these interactions are with the electric field of the radio frequency (RF) and microwaves. Water in food is often the primary component responsible for dielectric heating. Due to their dipolar nature, water molecules attempt to follow the electric field as they alternate at very high frequencies. Such rotations of the water molecules produce heat. Ions, such as those present in salty food, migrate under the influence of the electric field, generating heat. This is the second major mechanism of heating in microwaves and RF energy. Both the dielectric constant and the dielectric loss factor measure the ability of the material to interact with the electric field of the microwaves. The dielectric constant is a measure of the food material's ability to store electromagnetic energy, whereas the dielectric loss is the material's ability to dissipate electromagnetic energy (which results in heating) (<u>Rao, 2005</u>).

The Basic principle of MFD is similar with traditional FD, there is a triple Point existed for water in raw materials. Under 611 Pa, as shown in **Figure 1.1**, the solid state can transit to gaseous state without be to liquid state. The triple point temperature was 273.16 K/0.01 °C, following this principle, the pressure of FD drying cavity can keep the pressure low than 100 pa and offer the heat energy by heat shelf. The vapour can be catch by the cold trap, in which the temperature low enough to freeze the vapour. For FD, a prominent factor is the structural rigidity afforded by the frozen substance at the surface where sublimation occurs. This rigidity prevents collapse of the solid matrix remaining after drying. The result is a porous, non-shrunken structure of the dried product that facilitates rapid and almost complete rehydration when water is added to the substance. FD can maintain the high quality of products (colour, shape, aroma, texture, biological activity, etc.) better than any other drying method due to its low processing temperature and lack of oxygen in the process (Litvin et al. 1998; Strumillo and Ada-<u>miec, 1996</u>). Other advantages of freeze drying include protection against chemical decomposition, easy rehydration, etc. (Xu et al. 2005).

However, freeze drying is an expensive and lengthy dehydration process because of low drying rates, which lead to relatively small throughputs and high capital and energy costs generated by refrigeration and vacuum systems (<u>Duan et al. 2010</u>). In MFD system, it use microwave as the heat resource, which can be directly absorbed by the water molecules for sublimation within the food material, without being affected by the dry zone. As a result, MW heating offers a good opportunity to increase the drying rate in FD. Experiments and numerical predictions show that the drying rate was significantly increased and the drying cost reduced with MW heating (<u>Wu et al. 2004</u>).



1.3. EXPERIMENTAL EQUIPMENT

A schematic diagram of MFD dryer was shown in **Figure 1.2**. There are two drying cavities where FD and MFD tests can be done. When they were dried in the FD cavity, the materials were heated by conduction from the electrically heated shelf. If the samples were dried in the MFD cavity, a microwave field supplied the required heat for drying. During drying, the pressure was maintained at 100 Pa by a vacuum pump. The temperature (-40~-45 °C) of the cold trap was low enough to condense the sublimed water vapor from the frozen banana state. To minimize the non-uniform distribution of the microwave field in the cavity, three magnetrons were used; the angle between two adjacent magnetrons was 60° . The microwave frequency was 2450 MHz. The power of microwave could be continually regulated from 0 to 2000 W. The temperature of the drying materials was detected by a special optical fiber probe which can operate in a microwave field. The microwave heating system could be automatically turned on or off during drying to control the material temperature.



Figure 1.2. Schematic diagram of the microwave freeze dryer

 Microwave freeze drying chamber 2. Optical fiber temperature sensor 3. Vacuum breakage valve, for MFD 4. Sample supporting plate 5. MFD Sample 6,7. Microwave source 8. Pressure sensor, for MFD chamber 9. Freeze drying chamber 10,12. Heating plate 11. FD sample 13. Pressure sensor, for FD chamber 14. Vacuum breakage valve, for FD 15. Temperature sensor 16. FD vacuum valve 17. MFD vacuum valve 18. Cold trap 19.Vacuum pump 20. Draining valve 21.Refrigeration compressor 22.Control system

1.4. PROCESSING

A schematic diagram of MFD dryer was shown in **Figure 1.2**. There are two drying cavities where FD and MFD tests can be done. When they were dried in the FD cavity, the materials were heated by conduction from the electrically heated shelf. If the samples were dried in the MFD cavity, a microwave field supplied the required heat for drying. During drying, the pressure was maintained at 100 Pa by a vacuum pump. The temperature (-40~-45 °C) of the cold trap was low enough to condense the sublimed water vapor from the frozen banana state. To minimize the non-uniform distribution of the microwave field in the cavity, three magnetrons were used; the angle between two adjacent magnetrons was 60° . The microwave frequency was 2450 MHz. The power of microwave could be continually regulated from 0 to 2000 W. The temperature of the drying materials was detected by a special optical fiber probe which can operate in a microwave field. The microwave heating system could be automatically turned on or off during drying to control the material temperature.

Material and pretreatment

- Material preparation including selection of fruits and vegetables of optimum maturity and rough machining, such as cleaning, sorting, peeling and slicing.
- Pretreatment including blanching, cooking, seasoning, pre-drying.

Freezing

- Freezing temperature should lower than the eutectic point of raw materials
- The materials prepared are placed in a ultra-low temperature freezer in a container directly.
- The freeze time should last 5 h at least to ensure the core of raw materials can achieve the eutectic point.

Drying

- The drying curves of different materials are necessary before drying.
- Determining the most suitable highest temperature of materials is necessary before drying
- Determining the most suitable microwave power of materials is necessary before drying
- The final moisture content of materials should lower than 5% (dry basis).

Storage

- Water sorption isotherms.
- Glass transition temperature
- Selection of storage condition and package material

1.4.1. Pretreatment procedure

MFD could produce the high quality fried foods with natural color, good flavor, and crispy texture. Some necessary pretreatment procedure like peeling, cutting should be taken during this stage. It should be mentioned that there is an edge effect existed when microwave was used (<u>James and Timothy, 1996</u>), as the result the material should be had a surface area as large as possible, and made the shape thin and flat. The drying process was taken vacuum condition, therefore it need not make colour protection course.

Some physical pre-treatment procedures have been investigated in order to improve drying kinetics and product quality. Wang et al. (Wang et al., 2010a) added the dielectric cores into the MFD instant vegetable soup and found that MFD process can be enhanced significantly by the addition of dielectric cores with high loss factor to the porous or liquid materials to be dried. The dielectric cores function as another heat source by absorbing microwave energy. In this experimental study, NaCl, sucrose and sodium glutamate were chose as the exogenous dielectric cores added into the raw materials. it was found that the soup with seasoning ingredients could reduce total drying time by 40%. Results showed that suitable addition of food ingredients could improve MFD rate of vegetable soup and reduce drying time, which was an ideal way to achieve high efficiency MFD process on the premise of good product quality.

One of the drying characteristic of MFD/FD was good rehydration, but some special environment the rehydration rate should be slow down. <u>Huang et al. (2009)</u> investigated the effect of coating on post-drying of freeze-dried strawberry pieces to control the rehydration rate. The best formula for the coating solution is found to be: whey protein 10%, glycerol 3%, lactose 10%. Color of strawberry pieces can be protected to some extent by adding Na+ and b-Cyclodextrin (b-CD) in the coating solution. The best proportions of Na+ and b-CD were 3 mg/ml and 0.5 mg/ml, respectively. Coated freeze-dried

pieces of strawberry were dried in a spouted bed. Coated freeze-dried pieces of strawberry were dried in a spouted bed. The rehydration ratio of coated and dried strawberry pieces was significant less than FD strawberry pieces in milk. It also can be used in MFD.

1.4.2. Freezing

The freezing stage represents the first separation step in the freeze drying process, and the performance of the overall freeze drying process depends significantly on this stage (Liapis et al., 1996). The material system to be processed is cooled down to a temperature that is always below the glass transfer temperature (T_g) of the material system. Glassy state is one kind of stage. In this stage, the energy of thermal motion of molecules is very low, which can freeze over the molecular chain. Material at this time showed similar mechanical properties with glass. Glass transfer temperature can be measured by DSC, DMTA and some other measuring method, the value is close to eutectic temperature. For instance, if the material to be freeze-dried is a solution with an equilibrium phase diagram that presents a eutectic point, then the value of the final freezing temperature must be below the value of the eutectic temperature; in this case, the material becomes wholly crystalline.

The shape of the pores, the pore size distribution, and pore connectivity of the porous network of the dried layer formed by the sublimation of the frozen water during the primary drying stage depend on the ice crystals that formed during the freezing stage; this dependence is of extreme importance because the parameters that characterize the mass and heat transfer rates in the dried layer are influenced significantly by the porous structure of the dried layer (Liapis et al., 2007; Millman et al., 1985; Liapis and Litchfield, 1979; Genin and Rene, 1996). If the ice crystals are small and discontinuous, then the mass transfer rate of the water vapor in the dried layer could be limited. On the other hand, if large dendritic ice crystals are formed and homogeneous dispersion of the preand post eutectic frozen solution can be realized, the mass transfer rate of the water vapor in the dried layer could be dried more quickly. Thus, the method and rate of freezing, as well as the shape of the container of the solution and the nature of the product, are critical to the course of lyophilization because they affect the drying rate and the quality of the product. Always, we prefer high quality to high drying rate.

Some methods can be used to control the size of ice-crystal. Application of an electric or magnetic field can significantly affect the freezing characteristics of water. A DC electric field will tend to induce ice nucleation at a lower degree of super cooling, and there is evidence to show that an AC electric field delays the onset of ice nucleation (Woo and Mujumdar, 2010). Water consists of dipole molecules. Due to the structure of water molecules, which are composed of two hydrogen atoms and one oxygen atom, the oxygen side of the molecule is partially negative, whereas the hydrogen side of the molecule is partially positive. When an electric field is applied across a water sample, water molecules tend to realign themselves along the direction of the field (Shevkunov and Vegiri, 2002). In the manipulation of the electric field, application of DC and AC fields may have opposite effects. The former tends to induce nucleation, whereas the latter has been shown to delay nucleation. These effects are due to the alignment effect of the dipole water molecules under an electric field (Sun et al., 2008; Orlowska et al., 2009; Do et al., 2004; Kim et al., 2007).

Water is a diamagnetic material and hence is susceptible to be magnetized by a magnetic field. Industrial research has shown that a magnetic field can be used to delay nucleation and to induce small, unclustered ice. Under a magnetic field, the hydrogen bonds between the water molecules are stronger, giving a more ordered and stable configuration (Tagami et al., 1999; Ueno and Iwasaki, 1994). Norio and Satoru (Norio and Satoru, 2001) of ABI Limited in Japan found that the magnetic field can help in maintaining water in a metastable state to even larger degree of supercooling. In other words, a magnetic field will tend to affect water in the opposite manner to that of a DC electric field.

Immersion freezing with ultrasound also can influence the freezing characteristics of water. Ultrasound plays an effective role in the formation of nuclei and subsequent crystal growth. During the freezing period, it would lead to fine ice crystals and shorten the time from the onset of crystallization to the formation of ice, thus reducing damages to cellular structure. This may due to acoustic cavitation, which consists of the formation, growth, and violent collapse of small bubbles or voids in liquids (Simal et al., 2008). A Schematic of the ultrasonic assistant freezing pretreatment system was shown in **Figure 1.3**. Xu et al. (2009) investigated the effect of power ultrasound pretreatment on edamame prior to freeze drying, the objective of her research was to evaluate factors influencing the effects of power ultrasound on freezing efficiency in immersion freezing and obtain proper pretreatment operation conditions. The results of response surface analysis show that the texture and the water-holding power of edamame after thawing are 6050 and 92% when 58 W ultrasound power is applied for 0.7 min, 50% intermittency at a coolant temperature of -20 °C.



1. compression tank; 2. condenser tube; 3. receiver (refrigerating medium); 4. pressure valve; 5. evaporator; 6. reaction vessel; 7. fan; 8: ultrasonic transducer; 9. external thermocouple; 10. internal thermocouple; 11. temperture control system; 12. ultrasonic generator.

Figure 1.3. Schematic of the ultrasonic assistant freezing pretreatment system. (Xu et al., 2009)

1.4.3. Drying procedure

During the MFD drying, drying parameters as following would significantly affect the properties of dried food, such as moisture content, color, and texture of dried foods.

• drying temperature/microwave power

- drying time
- vacuum degree
- cold trap temperature

This drying temperature means the highest temperature which materials can be achieved. Higher drying temperature and microwave power can lead a fast drying rate but if they exceed a certain degree it is easy to make the materials charred and some nutrition lost. Some pre-test is necessary before drying to fix the most suitable drying temperature and microwave power which can prevent drying fail. <u>Duan et al. (2007)</u> used microwave as a heat source for freeze drying cabbage; they noted that at suitable parameters MFD can reduce more than half of the drying time compared to FD without significant differences in product quality. There was an adjustment among the sublimation drying stage and desorption drying stage. Considering efficiency and quality, higher microwave power should be adopted in the sublimation phase, and lower microwave power should be introduced in the desorption phase. Wang et al. (2010b) did some researches about the effect of food ingredient on microwave freeze drying of instant vegetable soup, in this experimental investigation, a mild drying condition: drying processes were carried out at microwave power of 1.0 W/g and material temperature of 55 °C were used to compare the different drying rate of samples added different additives. <u>liang et al. (2010a)</u> compared the drying quality of banana chips disposed by different microwave powers, the result showed 2 W/g microwave would lead the highest sense score, and it is very east to cause the plastic container melting if the drying temperature higher than 70 °C.

For the basic principle of FD, the pressure of drying cavity must lower than 611 Pa. there is a plasma discharge phenomenon existed when microwave used in low pressure condition, which would lead locally charring (<u>Wu et al. 2010</u>). **Figure 1.4** shows that in about 50 Pa it is prone to make plasma discharge happened. In practice we always make the pressure low than 100 - 200 Pa to prevent this phenomenon. Relatively higher pressure is a good technique to save energy. In Duan's research (2007), 50, 100, 180 Pa were chose as the contrastive conditions. The results showed low cavity pressure was necessary to reduce the drying time. In fact, different cavity pressures had no significant effect on the sublimation phase, but it made the drying rate in the desorption phase change significantly. As a result, at the beginning of drying, higher pressure could be adopted, and in the desorption phase lower cavity pressure should be used. However, lower pressure needs more power consumption by the vacuum pump. Therefore, in the sublimation phase, 180 Pa cavity pressure is recommended along with 100 Pa pressure in the desorption phase.

Cold trap in freeze trap can catch the vapour which comes from samples by extra low temperature. It can prevent the vapour absorbed into the vacuum pump to protect the vacuum pump. The inner wall of cold trap can frost the vapour. Some research showed the heat transfer coefficient would depress as the thickness of frost increasing and the vacuum degree in cold trap decreasing (<u>Deng and Xu, 2003</u>). The temperature in cold trap need to maintain a certain temperature, as author's experience, -30 °C can meet the demand.



Figure 1.4. The relationship between electric field threshold value and pressure (<u>Wu et al., 2010</u>)

1.5. HEAT AND MASS TRANSFER MODELS OF MFD

The Heat and Mass Transfer Models of MFD can be divided into three parts, the heat and mass transfer equations in the sublimation condensation region, drying region and sublimation front, as shown in Fig. 5.1. <u>Wang and Shi (1998a)</u> developed a sublimation-condensation model based on an unsteady-state model. It illustrates an one-dimensional unsaturated porous medium slab to be freeze-dried with microwave heating. For this research, the heat and mass transfer equations in the dried region can be written as:

Mass transfer

$$\varepsilon \frac{\partial p_{\nu}}{\partial \tau} = \frac{\partial}{\partial x} \left(D_e \frac{\partial p_{\nu}}{\partial x} \right) \tag{1}$$

Heat transfer

$$\rho c \frac{\partial T}{\partial \tau} = \lambda \frac{\partial^2 T}{\partial x^2} + q \tag{2}$$

The heat and mass transfer equations in the sublimation condensation region are as follows:

Heat transfer

$$(\rho c + \varepsilon (1 - S) \frac{dp_v}{dT} \Delta H) \frac{\partial T}{\partial \tau} = \frac{\partial}{\partial x} ((\lambda + \Delta HKs) \frac{\partial T}{\partial x}) + q$$
(3)

Mass transfer

$$-u_{sat}\frac{\partial S}{\partial \tau} = -\frac{\partial}{\partial x}(Ks\frac{\partial T}{\partial x}) + f_T\frac{\partial}{\partial x}(\lambda + \Delta HKs)\frac{\partial T}{\partial x}) + f_{Tq}$$
(4)

$$f_{T} = \frac{\varepsilon(1-S)}{(\rho c + \varepsilon(1-S)\frac{d\rho_{\nu}}{dT}\Delta H)}\frac{d\rho_{\nu}}{dT}$$
(5)

The heat and mass transfer equations of the sublimation front are as follows: Heat transfer:

$$\left(-De\frac{\partial\rho v}{\partial x}\right)\Big|_{x=X^{+}} - \left(-Ks\frac{\partial T}{\partial x}\right)\Big|_{x=X^{-}} = J_f$$
(6)

Mass transfer:

$$\left(-\lambda e \frac{\partial T}{\partial x}\right)\Big|_{x=x^{-}} - \left(-\lambda \frac{\partial T}{\partial x}\right)\Big|_{x=x^{-}} = J_f \Delta H$$
(7)

$$J_f = \left(-u_{sat}S\frac{dX}{d\tau}\right)\Big|_{x=X}$$
(8)

This model could simulate the drying process well according to some reports (Wang and Shi, 1999; Wang and Shi, 1997; Wang and Shi, 1998b). Besides that, Tao et al. (2005) give a numerical simulation of conjugate heat and mass transfer process within cylindrical porous media with cylindrical dielectric cores in MFD, Chen and Wang (2005) developed a heat and mass transfer model of dielectric-material-assisted MFD of skim milk with hygroscopic effect.



Figure 1.5. Physical model of sublimation-condensation. I-Sublimation-condensation region; II-Sublimation front; III-Dried region. 1-vapor (ϵ (1-S)); 2-ice (ϵ S); 3-solid body (1- ϵ).(Wang et al., 1998a)

1.6. CONCLUSION

The main advantage of MFD is to sharply reduce drying times and save energy consumption. Many researches showed that MFD can save more than 50% drying time compare with FD. As long as the drying parameters are controlled well, MFD can ensure the same product quality as conventional FD. However, there are still some problems needing further research to successfully introduce MFD to industrial applications:

1. In order to bridge the gap between laboratory research and industrial applications, scale-up of MFD equipment should be developed. MFD dryer is an important facet to ensure the uniformity of drying. The locality of microwave sources should be settled suitable and the samples can move is better than keep still.

2. During the MFD process, the distribution of the electromagnetic fields is affected by the dielectric properties of the materials, so the drying control program should be based on the changes of dielectric properties of the materials. Additives can affect the dielectric properties obviously.

3. Freezing can affect the dried samples quality obviously, always we prefer good samples quality to fast drying rate, as a result we choose fast freeze.

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