International Journal of Engineering and TechnologyVol. 1, No.4, October, 2009
ISSN: 1793-8236

Study on the Preeze-drying Experiment of Kuala Fragrant Pear Slice

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Abstract—**The Kuala fragrant pear produced in Xinjiang Province of China is the materials of many kinds of heath food and drug. The measurement results showed that the average moisture content of fresh fragrant pear is 87%; the eutectic temperature is in the range of -17℃~-23℃ and is regarded as -20℃ averagely; the melting point is -15℃. For the fragrant pear of 4mm thick, the freeze-drying experiment were carried out as follows: the pre-freezing time is 1.5h, and the pre-freezing temperature is -35℃; the time for freeze dehydration is 7.5h, and the temperature is respectively 0℃, 10℃, 20℃; the time for desorption drying is 2h, and the temperature is 50℃. The total time is 11h. The freeze-drying process curve is given. The quality of the freeze-dried fragrant pear was tested and the result is comparatively well. By a series of experiments, the effect of freeze-drying parameters on the freeze-drying time and product quality were discussed, the parameters including the freezing rate, heating temperature, pressure of drying chamber and thickness.**

Index Terms—**Freeze-drying, Kuala fragrant pear, eutectic temperature, physical parameters, process optimization**

I. Introduction

In recent years, much attention has been paid to the quality of foods during drying. Both the method of drying and physicochemical changes that occur in tissues during drying affect the quality of the dehy-drated product. It is well known that vacuum freeze-drying produces the highest quality processed food products[1 -2]. Compared to classical dehydration techniques, the main advantages of the vacuum freeze-drying process are: (1) the preservation of most of the initial raw material properties such as shape, appearance, taste, color, flavour, texture, biological activity, etc. and (2) the high rehydration capacity of the freeze-dried product. This is largely because the structure of the food is not severely damaged as in other preservation procedures.Freeze drying is completed by three steps[3]: freezing, sublimation drying, and desorption drying.

The Kuala fragrant pear produced in Xinjiang Province of China is the materials of many kinds of heath food and drug. It has very high economic value. The study on freeze-drying experiment for Kuala fragrant pear is carried out. The aim was to determine the influence of processing conditions: operating pressure and heating plate temperature, on both the freeze-drying time and the final product quality (appearance, colour, rehydration ratio and texture).

Manuscript received May 10, 2009.

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II. The Measurement of Kuala fragrant pear's physical parameters

The moisture content, the eutectic temperature and the melting point were measured. The moisture content of fresh fragrant pear was measured with the halogen moisture content determinator (MB45,OHAUS Ltd. USA). The result is in the range of 82%~90% and is regarded as 87% averagely. The resistance - temperature curve during freezing process is measured that is shown in Fig 1 by self-made resistance-type eutectic temperature determinator. Likewise, the resistance - temperature curve during melting process is shown in Fig 2. According to the figures, the eutectic temperature is in the range of -17℃~-23℃ and is regarded as -20℃ averagely; the melting point is about -15℃.

The eutectic and melting temperature of the fragrant pear was also measured with differential scanning calorimetry (DSC-Q100, TA, USA). And the eutectic and melting point were determined from heat flow curves with professional software (Universal Analysis, TA, USA), as shown in Fig.3.

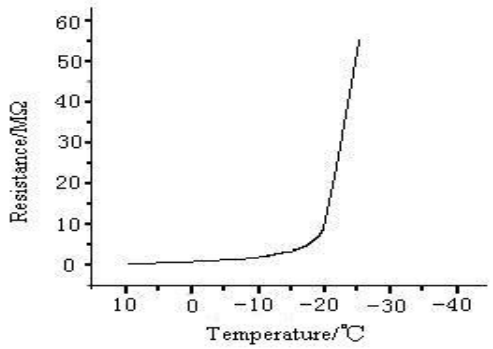


Fig. 1 The resistance- temperature curve for measuring fragrant pear's eutectic point

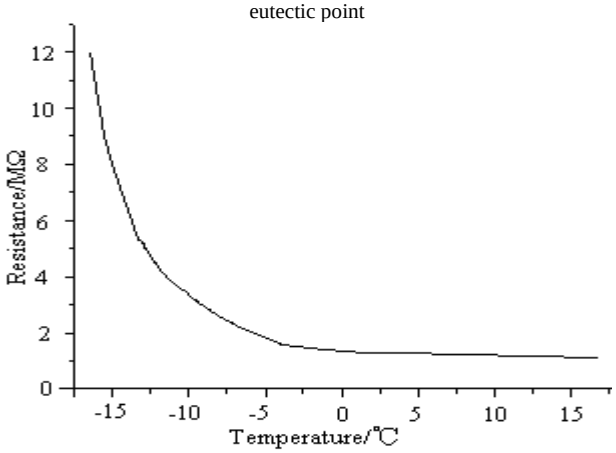


Fig. 2 The resistance- temperature curve for measuring pear's melting point

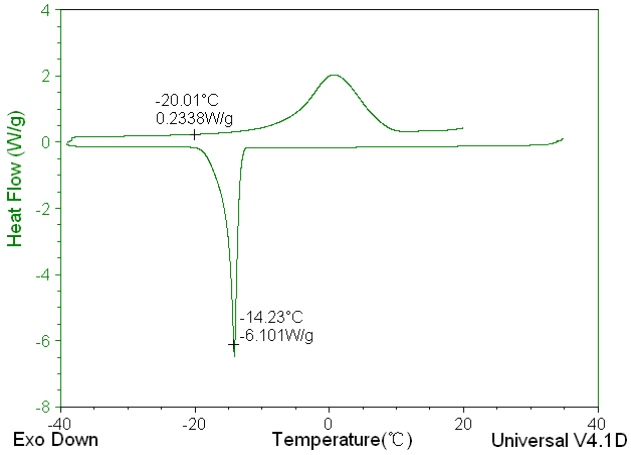


Fig. 3 Eutectic and melting point determination on DSC thermogram

III. FREEZE-DRYING PROCESS OF KUALA FRAGRANT PEAR

Through many experiments carried out on GLZ-0.4 vacuum freeze-drying equipment, a mature freeze-drying process of fragrant pear pieces is summarized as follows:

A. Pretreatment

The pears with good quality and uniform maturity were picked out and cleaned. Their skin was peeled after draining. Their weight and moisture content were measured. They were cut into slices with thickness of about 4mm, paved in the pallet and placed on the shelf of the freeze-drying chamber.

B. Pre-freezing

Considering that the pear's eutectic temperature is -20℃ and the temperature difference between the shelf and material is 10~15℃, the shelf temperature was set to be -35℃. After refrigerating for 1.5h, the pear slices have been totally frozen and the temperature of them was -30℃.

C. Sublimation Drying

The condenser of freeze-dryer began to be refrigerated. The temperature of condenser decreased to -40℃ in 3~5 minutes. Then the vacuum pump was started. The shelves were heated when the pressure of freeze-drying chamber reached 40Pa. The heating power is determined based on the regulation of the shelf temperature. Firstly, the shelf temperature is increased to 0℃ in 30 min and kept for 2 h; secondly, the shelf temperature is increased to 10℃ in 30 min and kept for 1.5 h; and at last, the shelf temperature is increased to 20℃ in 30 min and kept for 2.5 h. The heating power should be controlled to keep the temperature of the pear slice below its melting point temperature. When the ice in pear slices has sublimated through the top and bottom surface, the ice-boundary receded to the center of the pear slice, the temperature of product is very close to shelf's The sublimation drying process is finished and desorption drying process was started. In order to shorten the time of sublimation drying, the cyclic pressure method was adopted and the gas pressure is controlled in the range of 20Pa ~ 60Pa. The water 97% was removed in the sublimation drying process and 7.5 h was spent.

D. Desorption Drying

The heating temperature continue to be increased and the stage of desorption drying is started. The shelf temperature should not exceed 55℃ and the pear slice temperature should not exceed 50℃. The gas pressure is always 20 Pa. The time of the desorption drying is 2 h. When the temperature of product is close to shelf's, the whole process of freeze-drying is finished.

The termination of desorption drying process can be judged by two methods. (i)Temperature approaching method. In the late stage of freeze-drying, the water evaporation quantity is very few so that the product temperature will increase continuously. When the product temperature is close to shelf's, the freeze-drying process is over. (ii)Pressure method. Turn off the valve between the freeze-drying chamber and condenser for a period of time, and observe the pressure change in the freeze-drying chamber. If it's no longer change, then the drying process is over. The two methods are both used in these experiments.

E. Post-processing

The average moisture content of the freeze-dried pear slices is about 2%. The freeze-dried pear slices are easy to absorb moisture and easy to be metamorphic, therefore, they must be care of storage and transportation. A relatively successful method is to keep the freeze-dried pear slices in vacuum package. In these experiments, the products have been storage for 80 days and were not deteriorated. If the pear slice has not been freeze-dried completely, however, it would regain moisture and change color.

F. Freeze-drying Process Curve of Kuala Fragrant Pear

In the experiment, freeze-drier can automatically record the temperature of the fluid, condenser, shelves and three groups of the product. Temperature-time curve is shown in Fig.4. It shows the tendency of temperature change in stages of pre-freezing, sublimation drying and desorption drying. The totally time expended is 11 h.

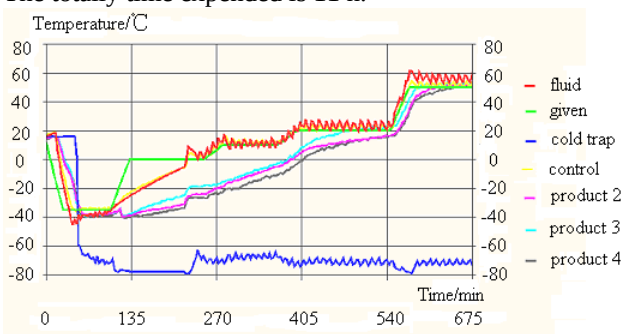


Fig. 4 The curve of vacuum freeze-drying for fragrant pear slice

IV. QUALITY TEST OF FREEZE-DRIED PEAR SLICES

A. Sensory Evaluation

Sensory character of high quality pear slices is: pulp is quite dried and the color is light yellow; the tissue is loose and there is no hardening on the surface; the shape is plump and the thickness is close to that of fresh material; it tastes crisp and keeps the original aroma.

B. Component Measurement

(a) Contents of heavy metals: Cu, Fe and Mn are not detectable; Sn < 0.1mg/kg; Ng < 0.01mg/kg; Pb < 0.1mg/kg;

(b) Contents of mineral: Mg > 60mg/kg; K > 510 mg/kg; Na > 80mg/kg; Ca > 80mg/kg; P > 140 mg/kg; S > 6 mg/kg; Si > 30 mg/kg;

(c) Contents of microbe: total bacterial count < 100 ind/mg; coliform count < 6 ind/mg; pathogenic bacterial is not detectable;

(d) Moisture content: < 4%, and the average value is 2%.

C. *Rehydration Properties*

Rehydration is one of the important evaluation factor of quality. It can be represented as rehydration rate, i.e., the ratio of sample weight after rehydration to the fresh fruit weight before freeze-dried. It reflected the similarity of freeze-dried fruit with the fresh one. It can also be represented as the net increased water content of the sample after rehydration (wet basis) W or the water absorption of dry basis R directly. The formulae of them are respectively:

$$W = (G_R - G_{dry}) / G_R \quad (1)$$

$$R = G_R / G_{dry} \quad (2)$$

Where, G_{dry} is the weight of the freeze-dried fruit before rehydration; G_R is the weight of the sample after rehydration. The testing method of G_R is as follow: The sample is immersed in the water for a period of time, then picked out, drained and weighed. If the weight is not changed more than 10% in 10 min, the data of last time is the result.

The test data of rehydration properties of freeze-dried pear slices in different water temperature conditions are listed in Table1. The best result is the water temperature 50℃ and the rehydration time 10 min.

Table1 The rehydration rate of fragrant pear slice*

Water temperature ℃	Rehydration time min	Rehydration rate %	W %	R %
25	35	79.8	83.3	6
50	10	87.5	84.8	6.6
75	5	85.3	84.4	6.4

*moisture content of fresh fragrant pear slice is 87%, the sample of freeze-dried pear slice is 2%

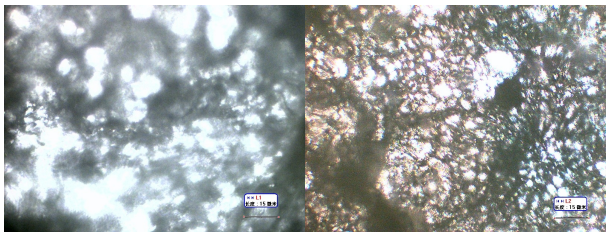
V. FACTOR ANALYSIS IN THE FREEZE-DRYING PROCESS OF KUALA FRAGRANT PEAR SLICES

In order to investigate the influence of process parameters on the freeze-drying process and quality of the product, a series of single factor experiments of freezing rate, temperature of shelf, gas pressure of freeze-drying chamber and thickness of samples have been respectively carried out.

A. *Freezing Rate*

Two projects have been designed in this paper that was quick-freezing and slow-freezing. (i) Quick-freezing. The temperature of shelf and pallet was lowered to -35℃ in 0.5 h and then the pear slices were paved in the pallet quickly. The temperature of shelf and pear slice respectively decreased to -37℃ and -35℃ in 1 h. (ii) Slow-freezing. The pear slices and pallet were put into the freeze-drying chamber at normal temperature and the cooling rate was controlled in the range of 1~2℃/min. The temperature of shelf and pear slices respectively decreased to -36℃ and -28℃. After frozen by two methods, the products were dried and tested as normal.

The micro-observation of freeze-dried product section by the reversed-biomicroscope (AE31, Motic Ltd., China) shows that the diameter of micro-holes for quick-freezing pear slices is about 5~8μm, while that for slow-freezing pear slices is about 9~12μm, as shown in Fig 5. So it directly illustrates that the ice crystal for quick-freezing pear slices is small and the micro-holes channel is fine and tortuous. Therefore, the time for sublimation drying is longer and the effect of rehydration is not very well. The effect of two freezing methods on the freezing time and product quality is shown in Table 2.



(a) quick-freezing (b) slow-freezing
Fig. 5 The micrograph of freeze-drying fragrant pear slices

Table 2 the effect of freezing ways on freeze-drying time and quality

Pre-freezing method	Sublimation time h	Rehydration ate %	Appearance	Micro void μm
quick-freezing	8.5	80.2	yellow, no crisp	5-8
slow-freezing	7.5	87.5	pale yellow, crisp	9-12

B. *Heating Temperature of Shelves*

In the sublimation drying stage, the heating temperature is usually raised as soon as possible in order to accelerate the drying rate. But the phenomenon such as melting and disintegration should be avoided. In order to determine the suitable heating temperature, the highest shelf temperatures in the sublimation drying process were respectively set at 30℃, 25℃ and 20℃ for 3 kinds of experiments. The results showed as follows: When the sublimation drying temperature was 30℃, the phenomenon of melting and brown stain would appear, and the pear slices would shrink obviously; when it was 25℃, the rehydration rate of product is very low; even when it was 20℃, the gas pressure was rising if the temperature increased too early, because the heating rate is too fast, and the ice sublimating rate is beyond the condensing capacity of condenser. At last, the "three-step" heating method as mentioned earlier was determined.

In the desorption drying stage, the heating temperature and time directly affect the final water content of product. The highest temperature is limited by the heat-durability of material instead of the melting temperature. Some experiments have been carried out when the temperature 50~70℃ and the gas pressure 30 Pa. When the sample temperature was beyond 55℃, some hardening pieces and dark spots appeared on the surface, resulting in the worse quality. So the desorption temperature was determined at 50℃ at last.

C. *Gas Pressure*

The influence of gas pressure in the drying chamber on the freeze-drying process is shown as the variation of thermal

conductivity and mass transfer coefficient through the dried layer. The increase of gas pressure will increase the thermal conductivity of the dried layer, but will decrease the mass transfer coefficient. Some freeze-drying experiments of fragrant pear slices have been respectively carried out under constant pressure of 30 Pa, 45 Pa and 70 Pa. Results show that under the pressure of 45 Pa the drying time is relatively shorter. The sublimation drying time was 10 h and the desorption drying time was 2.5 h.

In order to balance the heat and mass transfer, the "cycle-pressure" method was adopted, i.e., the gas pressure in drying chamber is increased and decreased periodically, so that the thermal conductivity and mass transfer coefficient are increased alternatively. Experiments were respectively carried out when the gas pressure in the range of 20~60Pa, 30~70Pa and 40~80Pa. The results showed that the drying effect is the best when the pressure 20~60 Pa. The time of drying and rehydration both are the shortest.

The temperature-time curves of freeze-drying at the pressure 45 Pa was compared with that at the pressure 20~60 Pa in Fig.6. The temperature rising had to be slowed down at the latter stage of sublimation drying when the pressure 45 Pa, because it has been limited by the mass transfer ability. the 'cycle-pressure' method of 20~60 Pa was adopted as mentioned above.

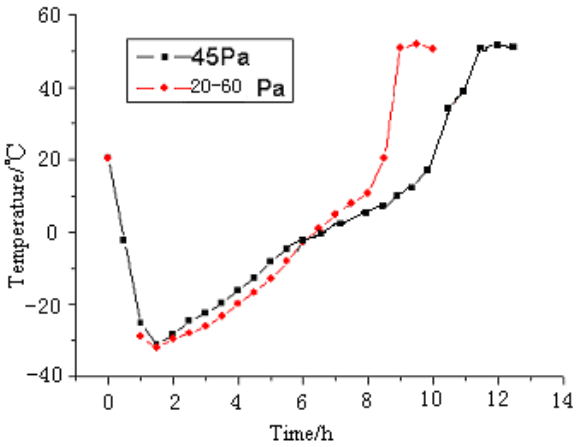


Fig. 6 Freeze-drying curve of fragrant pear for different pressure

D. *Thickness of Material*

The determination for the thickness of pear slices is to get the balance between the material loadage and the freeze-drying time. Theoretically, the thinner it is, the less the drying time, but the lower the production efficiency. Experiments of pear slices for thickness 2mm, 4mm and 6mm have been carried out respectively. The temperature-time curves of different thickness materials are shown in Fig.7, and the time for them are listed in Table 2.

Table 2 The effect of the thickness on freeze-drying time

Thickness mm	Pre-freezing rate ℃/min	Sublimation time h	Desorption time h	Rehydration rate %
2	1.2	4.5	1.5	80.3
4	0.9	7.5	2	87.5
6	0.7	10.5	2.5	75.3

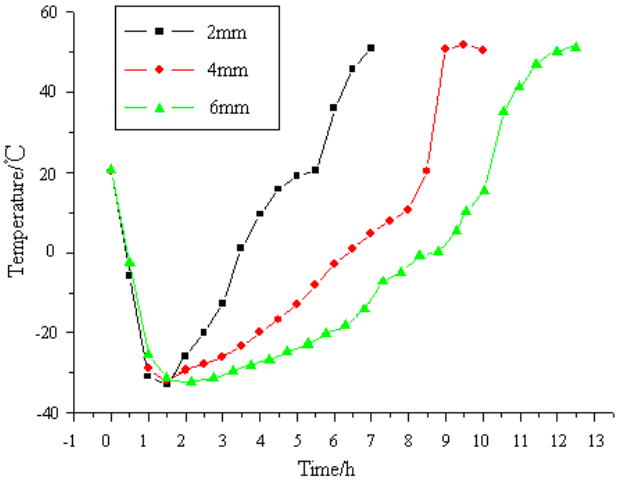


Fig. 7 Freeze-drying curve for fragrant pears slice of different thickness

Because the sublimation and desorption drying time is proportional to the thickness and the auxiliary time is same, the production efficiency of pear slices for thickness 6mm is the highest. But the thicker the material is, the more difficult the process control. It is hard to guarantee the product quality for thickness 6mm. On the other hand, considering the actual size of Kuala fragrant pear, the product output ratio of pear slices for thickness 6mm is far lower than that for 4mm. So the freeze-drying process of pear slices with thickness 4mm is recommended.

VI. Conclusion

1）According to the testing data, the initial water content of Kuala fragrant pear is 87%, the eutectic temperature is -20℃ and the melting point is -15℃.

2） The best freeze-drying process parameters are: the sample thickness is 4mm; the pre-freezing temperature is -35℃ and the time is 1.5 h; the sublimation drying temperature is increased slowly from 0℃ to 20℃ and time is 7.5 h, the cycle-pressure is 20~60 Pa; the desorption drying temperature is 50℃ and the time is 2h.

3）The water content of the freeze-dried fragrant pear slice is about 2%. The freeze-dried fragrant pear slice holds the original color, aroma, taste and nutritional components, and tastes crisp. It can be directly eaten as instant food. It also has a good Rehydration property and the rehydration rate is up to 87%.

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