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Research Article

The Effect of Freeze-drying Conditions on Drying Rate and Rehydration Ratio of Dumplings

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Abstract: Freeze-dried dumplings can meet people's requirements for food with nature, nutrition, safety, health and convenience and have a potential market prospect. However, high cost and poor rehydration properties limited its development. Aiming at reducing energy consumption and increasing the rehydration properties of freeze-dried dumplings, the effects of freeze-drying conditions, including pre-freezing temperatures, pre-freezing time, drying temperatures and drying time on the drying rate and rehydration ratio of dumplings were investigated in this study. The result shows that, in the process of freeze-drying dumplings, the optimal condition is that the fresh dumplings were firstly frozen for 3.5 h and secondly dried for 20 h in the sublimation drying process; finally the dumplings continued to be dried for 6 h for the secondary drying. Under this condition, the drying rate is 2.65%/h and the rehydration ratio is 1.99.

Keywords: Dumplings, drying rate, rehydration ratio, vacuum freeze-drying

INTRODUCTION

Dumplings are not only the most widely loved traditional food, but also the world famous delicacies with nutrition, taste and easy digestion (Zhao *et al*., 1995). However, its complicated production process fails to satisfy the requirements of fast-paced lifestyle. Currently, there are mainly two kinds of instant dumplings, one is frozen dumpling and the other is dried dumpling. Frozen dumplings should be stored at low temperature because of their high percentage of water, so it is inconvenient for portability and transportation (Zhang *et al*., 2005). Dried dumplings are made primarily by fried, hot air drying and vacuum freeze-drying technology. Dumplings made by the former two technologies are poor in quality, including appearance (shrinkage, drying-up, darkening), nutrients, flavor and the low rate of rehydration; while dumplings made by vacuum freeze-drying technology can maximize their color, smell, taste, appearance and nutrition (Zhang *et al*., 2012). Moreover, this kind of dumplings are easy to carry and can be eaten directly after being soaked in boiled water (Chen *et al*., 2006; Hua *et al*., 2003). Therefore, the freeze-dried dumplings will have a broad market prospect. But the freeze-dried dumplings still present some disadvantages such as the poor rehydration property and high cost, which limit its development. Objective of the study was to expert to find the effects of pre-freezing and sublimation drying conditions on the quality of dumpling and obtain optimal freeze-drying condition, which provide theoretical and technical support for the freeze-dried industry.

MATERIALS AND METHODS

Materials: The wheat flour, fresh pork, leeks, eggs and salt used in the current study were purchased in a local supermarket.

Instruments: The vacuum freezing dryer is LGJ-12S (produced by Huaxing Technology Development LTD. Songyuan Beijing), B204-N Analytical balance (Mettler-Toledo Group), 2XZ-2 Rotary-vane Vacuum Pump (produced by Linhai Tanshi Vacuum Equipmentco ltd).

Methods: Take the flour into a stainless steel pot, add water (40% Wt) and mix them together for 15 min, by which the dough with moderate hardness was prepared. Then place the dough in a plastic bag and roll out to about 3 mm thickness and 5 cm long. After the wrappers are prepared, make the dumplings and cook them in boiling water for 7 min and then dry them in the air for 20 min.

Load the dumplings on four trays of the freezing dryer with the loadage of 4 kg/m^2, then put the trays on a shelf and freeze them in the cold trap of the freezing

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dryer. After 3.5 h, take the dumplings into the freeze-drying chamber. And then, the vacuum pump was turned on and the heating button was pressed when the pressure is below 15 Pa. The dumplings were dried at 45°C. While the temperature of the dumplings was close to the room temperature and water content of the dumplings was close to 4-5%, the process of the freeze-drying was finished.

The drying rate: The drying rate (%/h) can be calculated according to the following formula (Chen *et al*., 2002):

$$F = 100 \times \frac{M_1 - M_2}{M_2 \times t} \tag{1}$$

In which,
M_1 = The initial mass before freeze-drying, g
M_2 = The mass after freeze-drying, g
t = The drying time, h

The rehydration: Weigh the freeze-dried dumplings and then rehydrate them in the boiling water for 10 min. After the rehydration, weigh them again and calculate their rehydration ratio. The rehydration ratio can be calculated as follows (Wang and Fang, 2005):

$$R = \frac{M_3}{M_2} \tag{2}$$

In which, M_3 is the mass of the dumplings after rehydration, g.

RESULTS AND DISCUSSION

The effects of pre-freezing temperature on the freeze-drying rate and rehydration ratio: The first stage of vacuum freeze-drying process is to freeze the dumplings until the temperature is below its eutectic point or glass transition temperature, during which the liquid water within the material become ice and the material matrix turn to be glass state. Then the whole material forms a complicated solid structure which contains both ice crystals and vitreum.

In the pre-freezing process, the drying rate and rehydration properties of freeze-dried dumplings are greatly influenced by the pre-freezing temperature. When pre-freezing temperature is -75, -60, -45, -35 and -25°C respectively, the effects of pre-freezing temperature on the freeze-drying rate and rehydration ratio were studied, which are shown in Fig. 1.

It can be seen from Fig. 1 that, with the increase of the pre-freezing temperature, the freeze-drying rate and rehydration ratio of dumplings both increase first and then decrease. When the temperature is -35°C, they both reach the maximum values.

When the pre-freezing temperature is higher than -35°C, the material can't be frozen completely and there will be a little liquid water existing. Under vacuum condition, the liquid water will evaporate quickly, which will prevent the dumplings from forming regular hole channel where the moisture sublimates and escapes in the drying process, resulting in the decrease of the drying rate. Meanwhile, as the material is not frozen thoroughly, the material will partially melt and collapse with the gradual increase of drying temperature, which influences the rehydration behavior of freeze-dried products.

And when the pre-freezing temperature is lower than -35°C, the energy consumption and cost will increase greatly. Besides, the lower the temperature, the more the frozen ice and the higher the solute

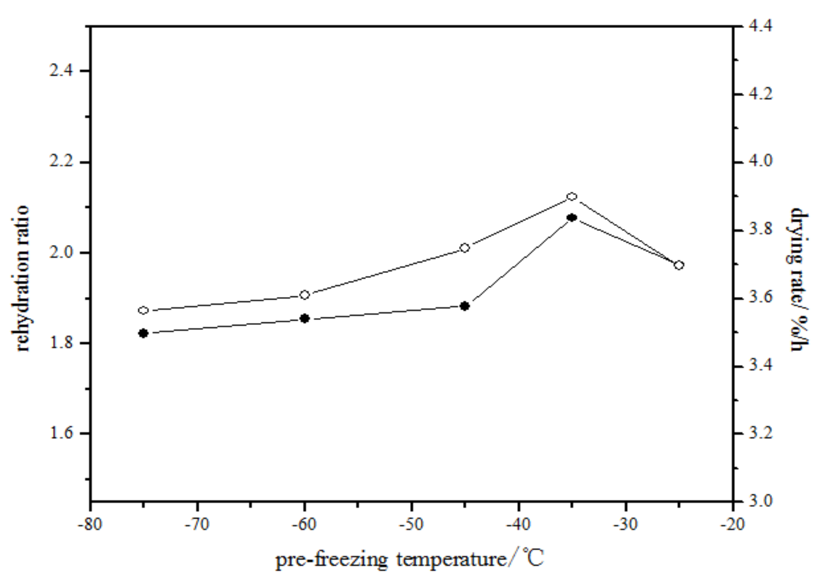

Fig. 1: The effect of pre-freezing temperature on the drying rate and rehydration ratio ○, drying rate (%/h); ●, rehydration ratio

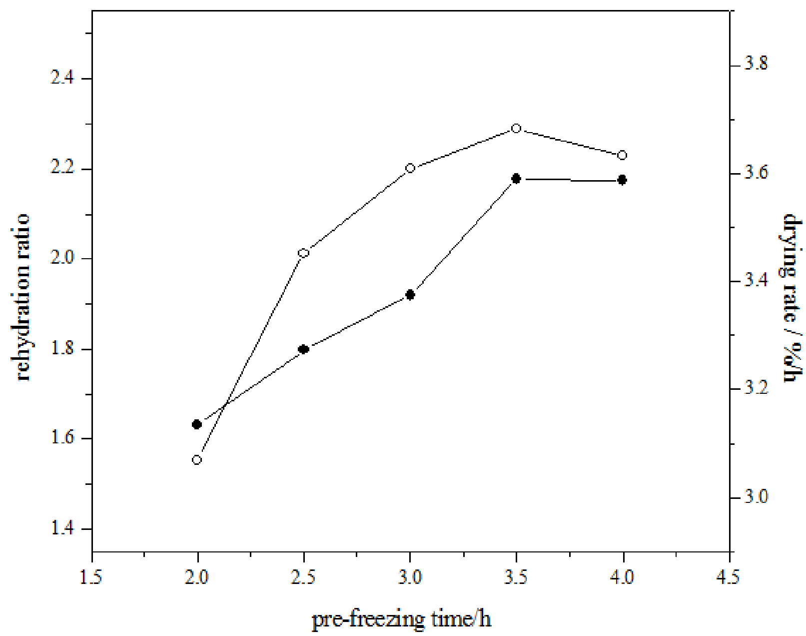

Fig. 2: The effect of pre-freezing time on the freeze-drying rate and rehydration ratio ○, drying rate (%/h); ●, rehydration ratio

concentration of unfrozen phase and glass-transition temperature, thus the drying rate is slow at the beginning. Meanwhile, if the pre-freezing temperature is too low, the ice crystal can't be removed completely in a certain time during sublimation drying process, which will make it difficult to form a loose and porous structure with the increase of the secondary drying temperature, therefore, the suitable pre-freezing temperature is -35°C.

The effects of pre-freezing time on the freeze-drying rate and rehydration ratio: The pre-freezing time has an important effect on the energy consumption and rehydration property of freeze-dried dumplings. If the pre-freezing time is too short, the dumplings can't be frozen completely and the residual moisture existing in liquid form will evaporate quickly in the drying process. This will make it difficult to form regular pores, thereby freeze-drying rate and rehydration ratio are seriously influenced. On the other hand, if the pre-freezing time is too long, the freeze-drying time are greatly extended, which will cause a great waste of energy.

When the freezing time is 2 h, 2.5 h, 3 h, 3.5 h and 4 h respectively, the effects of pre-freezing time on the freeze-drying rate and rehydration ratio were investigated and the results are shown in Fig. 2.

As is shown in Fig. 2, when the pre-frozen time are during 2-3.5 h, the drying rate and rehydration ratio both increase with the increase of pre-freezing time. The reasons may be that when the pre-freezing time is less than 3.5 h, the dumplings are not frozen thoroughly and the existing liquid water will evaporate quickly, which is not conducive to form a loose and porous structure evenly in the drying process, obstructing the hole channel where the moisture sublimates and escapes. Meanwhile, due to the dumplings are not frozen completely, the material will partially melt and collapse under dry condition and influence the rehydration ratio.

When the pre-freezing time is more than 3.5 h, the drying rate and rehydration ratio both decrease gradually with the increase of pre-freezing time, which might be attributed to that when the pre-freezing time is more than 3.5, the cooling load and energy consumption increase greatly; Moreover, with the increase of pre-freezing time, pre-freezing temperature will decrease to the freezing point of material and then the crystal nucleus is formed. In the freezing process, as the ice crystals increase, the solute concentration of unfrozen phase is improved continuously.

In addition, when the temperature is below -2°C (the eutectic point temperature of dumplings), theoretically, the material and its water should have formed eutectic. With the further increase of pre-freezing time, a growing number of ice crystals are formed and the solute concentration of unfrozen phase goes higher. When the temperature reaches the glass-transition temperature, the solute concentration even reaches the maximum. Then the unfrozen phase forms the glass state and the whole material forms a complicated solid structure. Therefore, at the beginning of the drying process, the drying rate is slow correspondingly. But if the pre-freezing time is enough, material will be frozen thoroughly and under dry condition, water can escape from the structural channel easily, thus it can form loose and porous structure that contributes to the improvement of drying and rehydration of dumplings.

Table 1: The factors and levels for the orthogonal experiment

Factor	Code	Level 1	Level 2	Level 3
Pre-freezing temperature (°C)	A	-25	-35	-45
Pre-freezing time (h)	B	2	3	4
Pre-freezing methods	C	Slow freezing	Fast freezing	Freezing in stages

Table 2: The orthogonal experiment results

No	A	B	C	Result 1 Rehydration ratio	Result 2 Drying rate (%/h)
1	1	1	1	2.104	3.802
2	1	2	2	2.152	3.842
3	1	3	3	1.974	3.696
4	2	1	2	1.931	3.824
5	2	2	3	2.720	3.877
6	2	3	1	2.078	3.800
7	3	1	3	2.311	3.936
8	3	2	1	2.528	3.958
9	3	3	2	2.412	3.905
K_1	6.230	6.346	6.713	-	-
K_2	6.729	6.729	6.495	-	-
K_3	7.251	6.464	7.005	-	-
k_1	2.077	2.115	2.238	-	-
k_2	2.243	2.467	2.165	-	-
k_3	2.417	2.155	2.335	-	-
R_1	0.340	0.352	0.170	-	-

K_i presents the sum of level i of A, B, C respectively and k_i is the average value of level i

The effects of prefreezing methods on the freeze-drying rate and rehydration ratio of freeze-dried dumplings: It is prone to mechanical effect (Qin, 2006) and solute effect (Zhang, 1999) in the pre-freezing process of dumplings and pre-freezing methods have an important effect on the dumplings` structure during sublimation drying. Slow freezing allows the dumplings to form larger but irregular crystals, which are likely to cause mechanical damage of cells and tissues and make it difficult to re-hydrate. Nevertheless, the larger crystals leave large pores after sublimation drying, which would provide big hole channels for water to escape and thus improve the drying rate. On the other hand, fast freezing allows dumplings to form small crystals with much less damage to the cells and tissues, but the smaller crystals leave small pores in drying process, which increases the resistance of water vapor escaping from the hole channels, as a result, water vapor can only pass through the dried layer by slowly infiltrating and decrease the drying rate. Conclusively, the ice crystal size in dumplings can greatly influence the drying rate and rehydration and the freezing methods should be determined by experiments.

Multi-factor orthogonal test in the pre-freezing stage: Based on the experiments of single factor above, it was found that the pre-freezing temperature, pre-freezing time and pre-freezing methods are the main factors. In order to investigate the effects of these factors on drying rate and rehydration ratio of freeze-drying dumplings and to find the optimal conditions, the orthogonal experiments including three factors and three levels were designed and carried out, which are shown in Table 1. The experimental results based on the orthogonal array $L_9(3^3)$ are listed in Table 2.

From the results shown in Table 2, it can be seen that the important factor is pre-freezing temperature and then in sequence is pre-freezing time and pre-freezing methods. The optimal conditions obtained from the orthogonal experiments are that the pre-freezing temperature is -45°C, pre-freezing time is 3 h and the method is the staged freezing, but it is not among the above single factor experiments. Therefore, another single factor experiment was done around pre-freezing time, which revealed that the optimal pre-freezing time is 3.5 h. In conclusion, taking comprehensive consideration of energy consumption, freeze-drying time and the quality of the dumplings, the freezing conditions are that the dumplings were firstly frozen for 1.75 h at -30°C and then for another 1.75 h at -45°C.

The effects of drying temperature on the freeze-drying rate and rehydration ratio of freeze-dried dumplings: The frozen material is heated at low temperature and low pressure so that the ice crystals can be sublimated and thus leave a regular network structure. After the freeze-drying, material can be preserved for a long time at room temperature if sealed tightly. During drying, the initial heating temperature should be controlled around the eutectic point temperature of the material and it shouldn't be too high, otherwise, the ice crystals will partially melt and the water will evaporate directly, thereby hindering the formation of regular network structure, which greatly affects the dumplings` drying and rehydration (Jiang *et al*., 2007). When it appears some drying layer on the surface of the material, increase the heating temperature slowly and make sure that the dried layer will not disintegrate. The DSC curve of freeze-dried dumplings

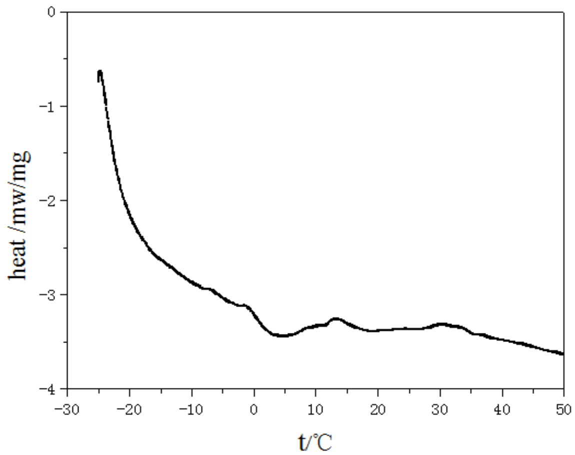

Fig. 3: The DSC curve of the freeze-dried dumplings' glass transition temperature

is shown in Fig. 3, it can be seen that the eutectic point temperature of dumplings is about -8°C and the melting point is about -2°C.

In order to make sure that the dumplings will not melt at the beginning of drying, the initial drying temperature should not be higher than eutectic point temperature. The effects of initial drying temperature (-20, -15, -8, -5, 0°C, respectively) on the freeze-drying rate and rehydration ratio were investigated. The result is shown in Fig. 4.

It can be seen from Fig. 4 that drying rate and rehydration ratio both increase firstly and then decrease swiftly with the increase of temperature. When the initial temperature is -8°C, rehydration ratio and drying rate both reach the highest values. It is due to that the higher the temperature, the more the heat energy transferred from the frozen layer, the higher the driving force of mass transfer, the faster the escape rate of water and the faster the drying rate. But when the initial temperature is higher than -8°C, ice crystals will melt and disintegrate, obstructing the hole channel where the moisture sublimates and escapes. Therefore, under this condition the moisture in the dumplings can't be dried thoroughly which will influence the drying rate and rehydration. What's more, if the initial temperature continuously rises to 0°C, the structure of the dumplings is seriously damaged because of the melting and cracking of the ice, which certainly influences the quality of dumplings and rehydration ratio.

After being dried at -8°C for 5 h, a drying layer is generated on the surface of the dumplings, then the heating temperature can be increased to improve the drying rate. It is noted that the drying temperature should not exceed the melting point to avoid the melting of the ice crystals inside the dumplings. It can be observed from Figure 4 that, the melting point of dumplings is 3°C, so the effects of drying temperature on the freeze-drying rate and rehydration ratio at the temperatures of -4, -2, 0, 2 and 4°C, respectively were investigated under the condition that the initial temperature is -8°C. The result is shown in Fig. 5.

It can be seen from Fig. 5 that the drying rate and rehydration ratio reach the peak at 0°C. When the drying temperature is lower than 0°C, it is helpful to water escapes when dried and the water quickly and efficiently infiltrates into the inner of the dumplings when rehydrated, which can increase the drying rate

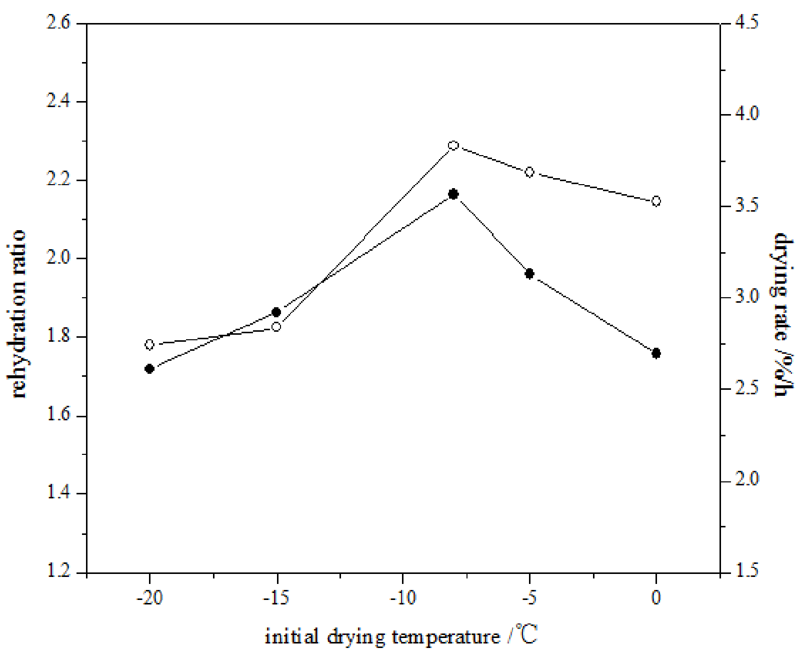

Fig. 4: The effect of initial drying temperature on the drying rate and rehydration ratio ○, drying rate (%/h); ●, rehydration ratio

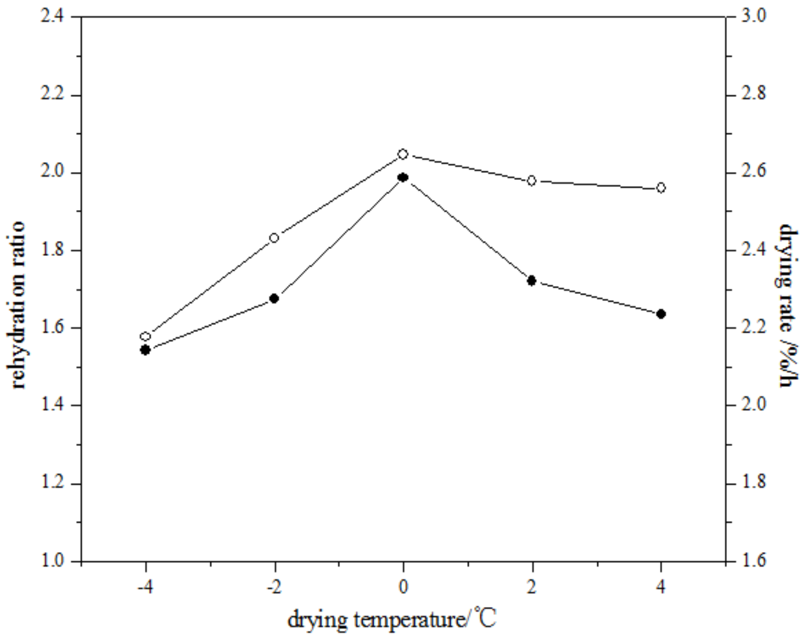

Fig. 5: The effect of drying temperature on the drying rate and rehydration ratio ○, drying rate (%/h); ●, rehydration ratio

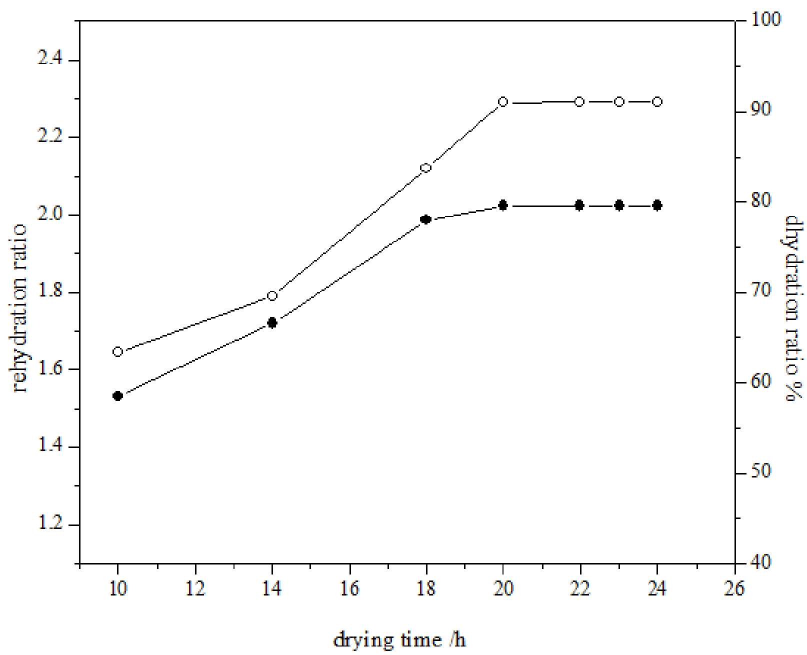

Fig. 6: The effect of drying time on the dehydration ratio and rehydration ratio ○, dehydration ratio (%); ●, rehydration ratio

and rehydration ratio. However, when the temperature is too high and even more than the melting temperature, the ice crystals will partially melt and collapse and water will evaporate directly, resulting in an irregular hole channel, which in turn hinders the water escaping from the dumplings.

The effects of drying time on the dehydration ratio and rehydration ratio of freeze-dried dumplings: In order to determine the time of sublimation drying process, the moisture content of dumplings was measured. When the moisture content is less than 10%, the sublimation drying process is finished. The relationship between dehydration ratio and drying time during the sublimation drying period is shown in Fig. 6.

It can be seen from Fig. 6 that, with the increase of the drying time, the dehydration and rehydration ratio both increase rapidly at first and then nearly keep constant. When the dumplings are dried for 20 h, the dehydration ratio is more than 90% and the rehydration ratio reach the maximum. When the drying time is less than 20 h, the free water in the material can’t be removed completely and the residual moisture is hold back in the hole channels, which may lead to a collapse of the internal structure when the secondary drying temperature is heated up and then influence the rehydration.

When the sublimation drying time is more than 20 h, the dehydration and rehydration ratio nearly remain unchanged, which indicates that the free water in the dumplings was removed completely and the primary drying was finished.

In order to remove the remaining 10% of adsorption water, the secondary drying is followed by the primary drying. And during this process, the material temperature is close to the plate temperature, so the heating temperature can be high enough as long as it doesn't influence the quality of the product. Therefore, the dumplings are dried at 40°C for 6 h for the secondary drying.

CONCLUSION

Effects of pre-freezing temperature, pre-freezing time, drying temperature and drying time on the drying rate and rehydration ratio of dumplings were studied. The results revealed that the optimal condition for the freeze-dried dumplings is that in freezing process, the fresh dumplings were frozen for 1.75 h at -30°C first and then for another 1.75 h at -45°C; and then in sublimation drying process, the frozen dumplings were dried for 20 h at -8°C and 0°C; after the first drying process, dumplings continued to be dried at 40°C for 6 h for the secondary drying. Under this condition, the drying rate is 2.65%/h and the rehydration ratio is 1.99.

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