Optimization of process parameters for freeze-drying of *Letinus edodes*

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Abstract: The eutectic point and melting point of *Letinus edodes* were measured, and vacuum freeze drying process of *Letinus edodes* were studied. Results showed that material thickness, freezing rate, drying pressure, and heating plate temperature were main factors influencing vacuum freeze drying of *Letinus edodes*. It also determined the scope of various factors and analyzed how these factors influence drying-time by single factor experiment and four-factor and five-level quadratic regression orthogonal experiment. The degree of process parameters affecting the drying-time was in the following order: freezing rate > drying pressure > material thickness > heating plate temperature. Finally, processing parameters were optimized and the most optimal parameters were: drying pressure of 61.8 Pa, heating plate temperature of 61.7℃, freezing rate of 2.58℃/min, material thickness of 6.78 mm, and drying time of 7.27 h.

Keywords: *Letinus edodes*, freeze-drying, drying time, process parameters, optimization

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1 Introduction

Letinus edodes is rich in protein, ascorbic acid, and various amino acids. The texture of fresh *Letinus edodes* is very tender. The water content of fresh *Letinus edodes* is very high (85% to 95%w.b.). After harvesting, the freshness of *Letinus edodes* drops quickly, resulting in cap opening, lamella browning, and body withering; therefore, the flavor and value of *Letinus edodes* will be affected (Wang and Xu, 1994). Traditionally dried *Letinus edodes* is neither healthy nor aesthetically pleasing, and a great deal of nutrients is lost. The growing trend of current food processing technology is to keep the nutrition and physical characteristics of food in a maximum extent (Yang et al., 1999). When adopting vacuum freeze-drying technology to process *Letinus edodes*, the water content may become very low, and its color, smell, taste, shape, and nutrients can be maintained excellently (Krokida et al., 1998; Che et al., 2008). After rehydration, the freeze-dried *Letinus edodes* may return closer to its fresh state. This processing method can avoid the surface hardening and lose of nutrients, and can extend the shelf life of *Letinus edodes* (Milford, 1967; Li and Li, 2003).

Vacuum freeze-drying technology is also called “freeze drying”. A brief description of freeze drying process is as follows: The temperature of fresh materials or wet materials is decreased lower than the eutectic point, which freezes the water inside the materials to ice. After that, the air in the drying cabinet is extracted to create vacuum, followed by heating the plate to an appropriate temperature, which will sublimate the ice to vapor. Finally, the vapor is condensed by water

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replenishment device of vacuum system or vapor condenser of cooling system, and the dried product is obtained (Ke, 2009). Drying process is the physical process involving movement of water in the material, and the change and movement occur in low temperature and low pressure. Therefore, the basic principle of vacuum freeze-drying is the mechanism of heat and mass transfer (Hong et al., 2002).

Along with the sustainable development of the economy and improvement of people's living standard, freeze-drying of *Letinus edodes* becomes more and more popular, and is one of the high value export products (Mi and Zhang, 2007). Based on the market survey, the price of fresh *Letinus edodes* is 10–15 RMB Yuan/kg, and the price of freeze-dried *Letinus edodes* reaches 140–150 RMB/kg. Freeze-dried *Letinus edodes* has a wide rang of markets at home and abroad, including United States, Russia, Japan, Korea, and other regions (Rennie et al., 2000), in which market of Hong Kong, Japan, the United States, South Korea, and ASEAN accounted for a larger proportion of the export market. Therefore, the freeze-dried *Letinus edodes* would have a good market prospect (Zhang and Chen, 2003).

2 Materials and methods

2.1 Materials

Letinus edodes was commercially obtained.

2.2 Main equipment

1) New lyophilizer, Institute of Modern Physics, Chinese Academy of Sciences, JDG-0.2 model.

2) Haier refrigerated tank, DW40L92 model.

3) Draught drying cabinet, Wuxi Sanxin Jinggong Testing Equipment, Co., Ltd., PHG-J023A model.

4) Electronic balance, Ohaus Instrument (Shanghai) Co., Ltd., CP1502 model.

2.3 Methods

2.3.1 Determination of eutectic point and melting point of *Letinus edodes*

The eutectic point and melting point of *Letinus edodes* were determined according to electric resistance method (Cui et al., 2008).

2.3.2 Process

Sample preparation — Pre-freezing — Drying — Drying analysis — Packaging

2.3.3 Determination and calculation of experimental indicators

1) Drying rate (R_{dry})

$$R_{dry} = G_g / G_o$$

Where, G_g = weight of fresh *Letinus edodes* before freeze-drying; G_o = weight of *Letinus edodes* after freeze-drying.

2) Nutrients

a. Water was determined according to National Food Safety Standard GB/T 5009.3–2003.

b. Crude protein was determined according to National Food Safety Standard GB/T 5009.5–2003.

c. Crude fat was determined according to National Food Safety Standard GB/T 5009.6–2003.

d. Polysaccharide was determined according to NV/T 1676–2008.

e. Vitamin D was determined according to AM-AM-FS-Jp-0017.

f. Riboflavin (Vitamin B2) was determined according to National Food Safety Standard GB/T 5009.85–2003.

g. Preservation rate of nutrients (ξ)

ξ = (composition rate of dry sample/drying rate)/ (composition rate of fresh sample)×100%

3) Rehydration ratio (R_f)

$$R_f = G_t / G_g$$

Where, G_t = drained weight of freeze-dried *Letinus edodes* after rehydration; G_g = weight of freeze-dried *Letinus edodes* before rehydration.

4) Sensory evaluation

Sensory quality for *Letinus edodes*' color, aroma, and appearance was evaluated with 10-point weight method. Table 1 gives sensory quality of the weighted value of *Letinus edodes*.

Table 1 Sensory quality of the weighted value of *Letinus edodes*

Index	Status	Weight	Remarks
Color	Uniform color, showing dark brown, consistent with the fresh *Letinus edodes*	4	
	Relatively uniform color, showing light brown, sometimes dark brown	3	
	Relatively uniform color, showing dark brown and yellow	2	
	Uneven color, showing light brown, dark brown, and yellow	1	

Index	Status	Weight	Remarks
Aroma	Aroma is rich and pure, with aroma of *Letinus edodes* after rehydration	3	
	Aroma is light, with aroma of *Letinus edodes* after rehydration	2	
	Aroma is light, with aging taste after rehydration	1	
Appearance	Tissue loose, no shrinkage, a gas chamber	3	
	Tissue loose, significant shrinkage, a gas chamber	2	
	Tissue closer, dense contraction of the surface layer	1	

3 Results and discussion

3.1 Single factor experiment and analysis

1) Material thickness: The thicker the *Letinus edodes* slice, the lower the drying rate in the whole freeze-drying process, and accordingly the rate of water escape slowed. Therefore, the damage of material cellular tissue became smaller and the recovery ability of materials to the original features was enhanced with an increase in material thickness of *Letinus edodes*. Conversely, freeze-drying rate increased and freeze-drying time became shorter with the decreased slice thickness of *Letinus edodes*. At last, in view of the shape characteristics of *Letinus edodes*, slice thickness is chosen to be 5–9 cm.

2) Cooling rate: Drying rate was accelerated and rehydration of sliced *Letinus edodes* reduced with a decrease in pre-freezing rate. At last, in view of cooling effect of machine itself, pre-freezing rate was controlled at 2.133–2.575℃/min.

3) Drying pressure: With the increase of drying pressure, water loss from *Letinus edodes* increased and drying rate accelerated. However, if drying pressure was too high, dried product quality was affected. Therefore, drying pressure was controlled at 55–75 Pa.

4) Heating plate temperature: With the increase of heating plate temperature, drying rate of *Letinus edodes* slice increased. But, if heating plate temperature was too high, ice crystal melted, and *Letinus edodes* slice burned and collapsed. Deformation of *Letinus edodes* slice became relatively larger and rehydration time became smaller with an increase in heating plate temperature. Therefore, the heating plate temperature was controlled at 55–70℃.

3.2 Quadratic orthogonal regression test and analysis

Table 2 shows regression analysis of variance, and Table 3 shows variance analysis of the regression equation. The regression equation was established using SAS software (drying time for Y value, drying pressure for x_1, heating plate temperature for x_2, cooling rate for x_3, and material thickness for x_4).

Effect of each factor on experimental index was determined through analysis of variance. The regression equation between drying time and various factors is:

$$Y=-277.165000+3.396167x_1+2.226333x_2+48.966667x_3+14.187500x_4-0.032588x_1^2+0.032625x_2x_1-0.036338x_2^2-0.339750x_3x_1-0.077250x_3x_2-3.758750x_3^2-0.064875x_4x_1+0.067375x_4x_2-0.346250x_4x_3-0.990938x_4^2$$

Table 2 Regression analysis of variance

Source	Degrees of freedom	Sum of squares	Mean square	F value	P value
Model	14	103.617005	7.401215	13.29	<0.0001
Error	15	8.350492	0.556699		
Total	29	111.967497			

Table 3 Variance analysis of the regression equation

Source	Degrees of freedom	Sum of squares	Mean square	F value	P value
Monomial	4	12.247950	3.0619875	5.05	0.0089
Quadratic term	4	64.606567	16.151642	16.70	< 0.0001
Cross terms	6	26.762488	4.460415	8.94	0.0003
Lack of fit	10	6.608158	0.660816	1.90	0.2488
Error	5	1.742333	0.348467		
Error total	15	8.350492	0.556699		

Significance analysis of the regression equation resulted in $F_1=13.29$, the corresponding probability value was <0.0001, and lack of fit provided F_2 = 1.90, the corresponding probability value was 0.2488, coefficient of determination was 0.9254. Thus, the regression equation is extremely remarkable. There was no evidence for lack of fit. At the same time, measured values were fitted well by the regression model. Each index of regression equation was significant, and the fitting of test data with the quadratic regression model was good. F values of monomial, quadratic term, and cross terms were above 0.01, indicating that effect of each factor on drying time was extremely significant.

3.3 Analysis and impact of interactions on the drying-time

3.3.1 Interaction analysis of drying pressure and heating plate temperature

Figure 1 shows response surface and contour lines of drying time as a function of pressure and temperature. When drying pressure is 55.00–63.00 Pa and heating plate temperature is 55–63℃, drying time increases with an increase in drying pressure and heating plate temperature. Thus, there is a synergistic effect between drying pressure and heating plate temperature. However, when drying pressure is 63–75.00 Pa and heating plate temperature is 63–75℃, drying time decreases with an increase in drying pressure and heating plate temperature, and thus, there is a strong opposition between drying pressure and heating plate temperature. Moreover, when drying pressure is 63 Pa and heating plate temperature is 63℃, the interaction between drying pressure and heating plate temperature is obvious and reaches the maximum value.

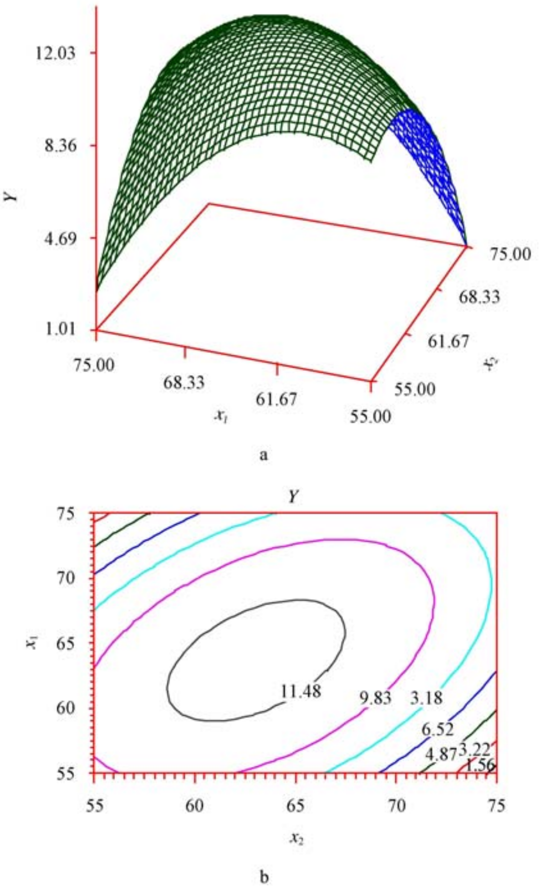

Figure 1 (a) Response surface and (b) contour lines of drying time (Y) as a function of drying pressure (x_1) and heating plate temperature (x_2)

3.3.2 Interaction analysis of drying pressure and cooling rate

Figure 2 shows response surface and contour lines of drying time as a function of pressure and cooling rate. When drying pressure is 55.00–64.00 Pa and cooling rate is 1.00–2.58℃/min, drying time increases with an increase in drying pressure and cooling rate. Therefore, there is a synergistic effect between drying pressure and cooling rate. However, when drying pressure is 64.00–75.00 Pa and cooling rate is 2.58–3.50℃/min, drying time decreases with an increase in drying pressure and cooling rate indicating that there is a strong opposition between drying pressure and cooling rate. Moreover, when drying pressure is 64.00 Pa and cooling rate is 2.58℃/min, the interaction between drying pressure and cooling rate is obvious and reaches the maximum value.

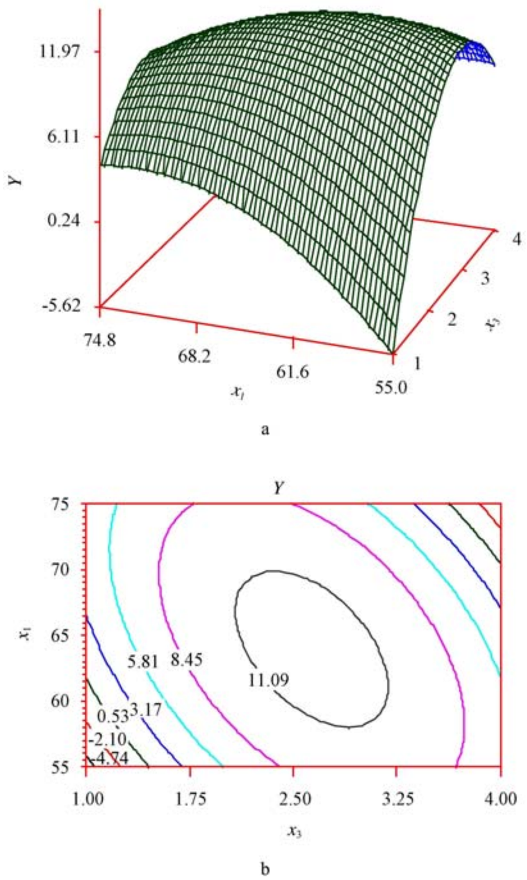

Figure 2 (a) Response surface and (b) contour lines of drying time (Y) as a function of drying pressure (x_1) and cooling rate (x_3)

3.3.3 Interaction analysis of drying pressure and material thickness

Figure 3 shows response surface and contour lines of drying time as a function of pressure and material thickness. When drying pressure is 55.00–64.80 Pa and material thickness is 4.0–7.0 mm, drying time increases with an increase in drying pressure and material thickness. Thus, there is a synergistic effect between drying pressure and material thickness. However, when drying pressure is 64.80–74.90 Pa and material thickness is 7.0–10.0 mm, drying time decreases with an increase in drying pressure and material thickness, and thus, there is a strong opposition between drying pressure and material thickness. Moreover, when drying pressure is 64.80 Pa and material thickness is 7.0 mm, the interaction between drying pressure and material thickness is obvious and reaches the maximum value.

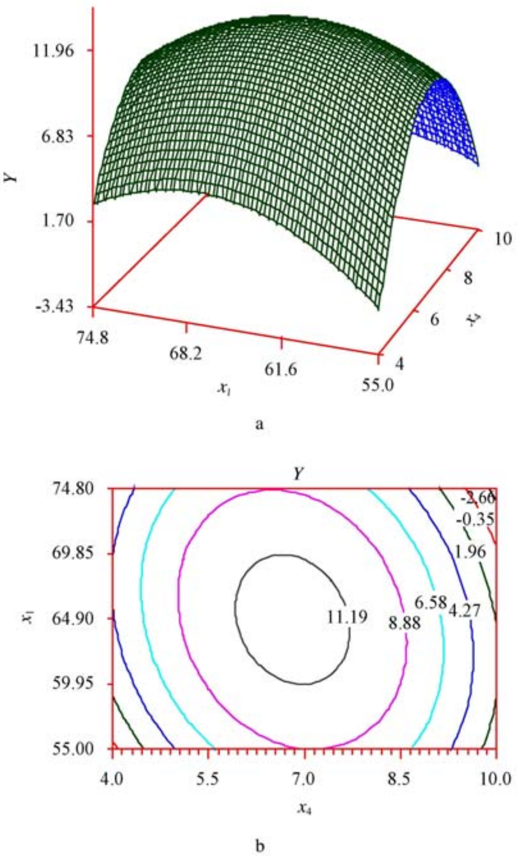

Figure 3 (a) Response surface and (b) contour lines of drying time (Y) as a function of drying pressure (x_1) and material thickness (x_4)

3.3.4 Interaction analysis of heating plate temperature and cooling rate

Figure 4 shows response surface and contour lines of drying time as a function of temperature and cooling rate. When heating plate temperature is 55.00–62.95℃ and cooling rate is 1.00–2.61℃/min, drying time increases with an increase in heating plate temperature and cooling rate. This shows that there is a synergistic effect between heating plate temperature and cooling rate. However, when heating plate temperature is 62.95–74.90℃ and cooling rate is 2.61–4.00℃/min, drying time decreases with an increase in heating plate temperature and cooling rate indicating that there is a strong opposition between heating plate temperature and cooling rate. Moreover, when heating plate temperature is 62.95℃ and cooling rate is 2.61℃/min, the interaction between heating plate temperature and cooling rate is obvious and reaches the maximum value.

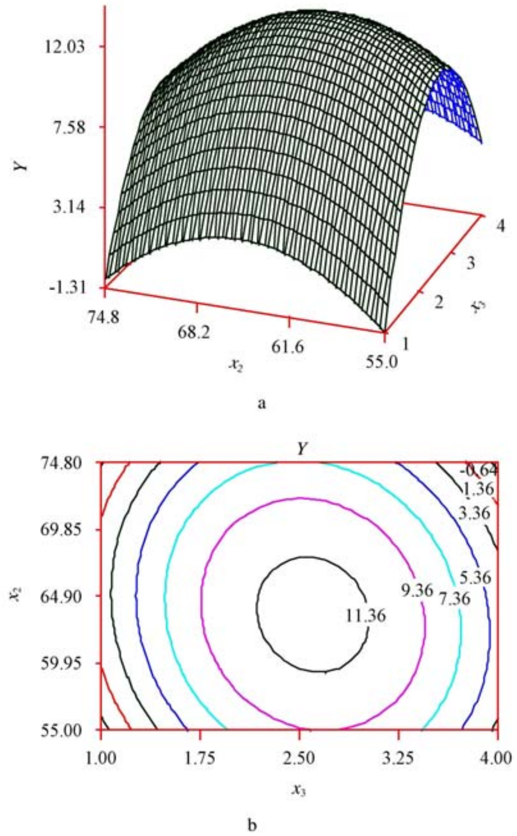

Figure 4 (a) Response surface and contour lines of drying time (Y) as a function of heating plate temperature (x_2) and cooling rate (x_3)

3.3.5 Interaction analysis of heating plate temperature and material thickness

Figure 5 shows response surface and contour lines of drying time as a function of temperature and material thickness. When heating plate temperature is 55.00–62.55℃ and material thickness is 4.00–6.35 mm, drying time increases with an increase in heating plate temperature and material thickness. This indicates that there is a synergistic effect between heating plate temperature and material thickness. However, when heating plate temperature is 62.55–74.90℃ and material thickness is 6.36–10.0 mm, drying time decreases with an increase in heating plate temperature and material thickness, and thus, there is a strong opposition between heating plate temperature and material thickness. Moreover, when heating plate temperature is 62.55℃ and material thickness is 6.35 mm, the interaction between heating plate temperature and material thickness is obvious and reaches the maximum value.

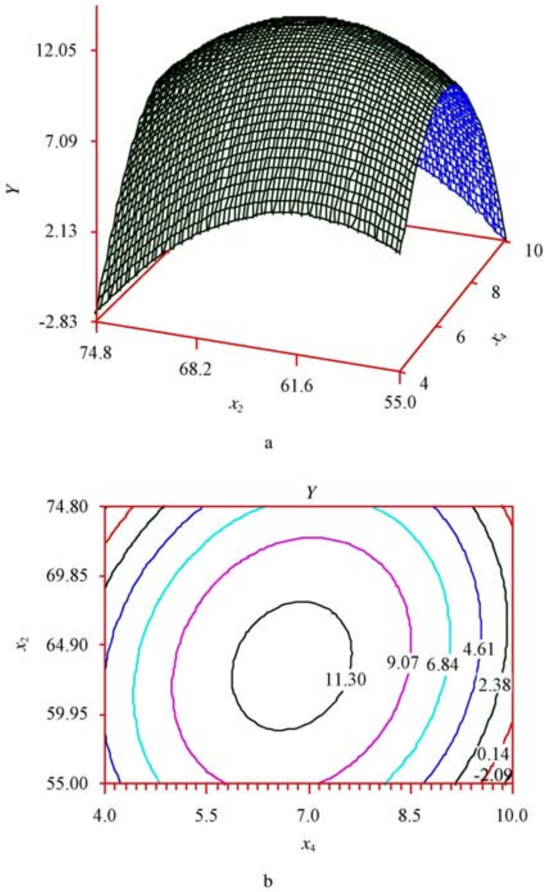

Figure 5 (a) Response surface and (b) contour lines of drying time (Y) as a function of heating plate temperature (x_2) and material thickness (x_4)

3.3.6 Interaction analysis of cooling rate and material thickness

Figure 6 shows response surface and contour lines of drying time as a function of cooling rate and material thickness. When cooling rate is 1.00–2.50℃/min and material thickness is 4.0–7.0 mm, drying time increases with an increase in cooling rate and material thickness. This shows that there is a synergistic effect between cooling rate and material thickness. However, when cooling rate is 2.50–4.00℃/min and material thickness is 7.0–10.0 mm, drying time decreases with an increase in cooling rate and material thickness, and thus, there is a strong opposition between cooling rate and material thickness. Moreover, when cooling rate is 2.50℃/min and material thickness is 7.0 mm, the interaction between cooling rate and material thickness is obvious and reaches the maximum value.

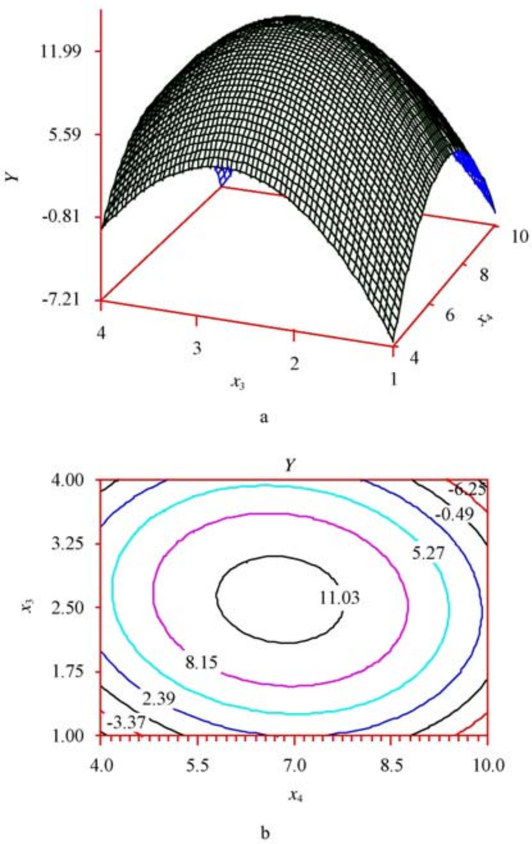

Figure 6 (a) Response surface and (b) contour lines of drying time (Y) as a function of cooling rate (x_3) and material thickness (x_4)

3.4 Optimization and analysis of process parameters

Table 4 shows optimization of process parameters for freeze-drying of *Letinus edodes* and the optimum results. Regression models verified by F test showed that significant level of regression equation reached more than 90%. In order to get optimal parameters for freeze drying of *Letinus edodes*, the regression model was further analyzed using SAS software.

The most optimal parameters are as follows: drying pressure of 61.8 Pa, heating plate temperature of 61.7℃, freezing rate of 2.58℃/min^{-1}, material thickness of 6.78 mm, and drying time of 7.27 h. As a result, mean error of experimental and theoretical values was small, indicating that regression model was a good fit.

Table 4 Optimization of process parameters of freeze-dried *Letinus edodes* and verification of results

Factors	Standardization	Non-standardized	Production rate
1	-0.320587	61.794131	
2	-0.332108	61.678922	7.265152
3	0.276329	2.576329	
4	-0.126195	6.747610	

4 Conclusions

1) Freeze-drying process and mechanism for *Letinus edodes* were analyzed through the theory of heat and mass transfer. Moreover, drying time was found to be an indicator of freeze-drying rate and quality of *Letinus edodes*. The range of main factors affecting the freeze-drying process was determined through single factor experiment.

2) Effect of each factor on drying time was measured through four factors and five levels of quadratic regression with rotatable orthogonal experiment. The degree of effect of various factors on the drying time was as follows: freezing rate > drying pressure > material thickness > heating plate temperature.

3) To ensure the quality of *Letinus edodes*, vacuum freeze drying process parameters for *Letinus edodes* were determined using the regression model established with the experimental factors and indicators, and using SAS analysis of the interaction of various factors. The most optimal freeze drying parameters were: drying pressure of 61.8 Pa, heating plate temperature of 61.7℃, freezing rate of 2.58℃/min^{-1}, material thickness of 6.78 mm, and drying time of 7.27 h.

4) The optimal process parameters were determined through the research of freeze-drying of *Letinus edodes* with the use of new equipment. This research would offer an effective theoretical foundation for industrial application of freeze-drying of *Letinus edodes*.

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