

Freeze and microwave vacuum combination drying technique for sea cucumber

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Abstract: To improve the quality and efficiency of sea cucumber drying, an orthogonal experiment of freeze and microwave vacuum combination drying technique was carried out. Factors and levels were FD time of conversion point (8 h, 16 h and 24 h), initial microwave power density (1.7 W/g, 2.0 W/g and 2.3 W/g) and microwave intermittent ratio (12s-on/18s-off, 14s-on/16s-off and 20s-on/10s-off). Results showed that the optimal parameters of FD-MVD were 8 h FD conversion point, 1.7 W/g initial microwave power density, 12 s-on/18 s-off microwave intermittent ratio, and the maximum rehydration rate of 280.71%, the lowest shrinkage rate of 14.48%, the least drying time of 9.25 h and energy consumption of 77 316 kJ could be obtained.

Keywords: sea cucumber, freeze drying, microwave vacuum, drying, combination

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1 Introduction

Sea cucumber (*Stichopus japonicus*) is an invertebrate animal belonging to the phylum Echinodermata. The major edible part of sea cucumber is the body wall which consists of a high quantity of collagen, low cholesterol and abundant physiological active substances for nutrition and health functions, especially the triterpene glycosides^[1] and acid mucopolysaccharides^[2] which have inhibiting effects on the growth and metastasis process of tumour

cells.

In China, more than 5000 tons of dehydrated sea cucumbers are consumed every year, but domestic throughput of dried sea cucumber is only about 3000 tons and about 2000 tons of dried sea cucumbers are imported every year^[3]. Currently, freeze drying (FD) and traditional solar radiation drying are regarded as the main methods of drying sea cucumber^[4]. Solar drying is economical but time-consuming and only makes inferior product with uncontrollable sanitary conditions and high salt content^[5]. To date FD yields the best quality of dried sea cucumber which is convenient for storage and transportation, delicate and smooth after rehydrated; and it preserves the most heat-sensitive nutrient components, while substantial investment in equipments, long drying time and high energy consumption are unavoidable. Therefore, it is essential to find a new technology to obtain good quality products at lower cost.

Microwave vacuum drying (MVD) has the mass and heat transfer in the same direction in vacuum, which not only accelerates the drying rate significantly, but also allows the material dried at a lower temperature, with retaining more heat-sensitive nutrients and improving the

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quality, therefore increasing the productivity as a result^[6].

The FD-MVD, which takes advantages of MVD and FD, was introduced for sea cucumber as a new efficient drying technique^[3], while the specified technique parameters have not been reported. The objectives were to study the FD-MVD of sea cucumber by orthogonal experiment, and research the effects of different drying parameters involving conversion point, initial microwave power density and intermittent ratio on the rehydration rate, shrinkage rate, drying time, energy consumption and chroma changes (ΔL^* , Δa^* , Δb^* , ΔE^*).

2 Materials and methods

2.1 Materials and pre-treatment

Fresh sea cucumbers were purchased from Heishijiao, Dalian, China. The guts were removed and the body wall was cleaned up. Then the materials were boiled at 100°C for 30 min with the salinity of 8%. Free water on the surface was removed with a filter paper and only the ones which were 11-15 g in weight and 5-8 cm in length were selected for experiments.

2.2 Equipments

The equipments used in this study include a freeze dryer (LGJ-10D, Beijing Sihuan Instrument Company, Beijing, China), a microwave vacuum dryer (MZ08S-1, Nanjing Huiyan Microwave Equipment Co. Ltd., Nanjing, China), a hot air dryer (101A-3, Shanghai Experimental Instrument Co. Ltd., Shanghai, China), a MSEZ portable colorimeter (4500S, Hunter Lab Company, the USA), an electronic balance (MP1100B, Shanghai Henping Science Instrument Co. Ltd., Shanghai, China), and a multimeter (UT53, Dongguan Youlide Technology Co. Ltd., Dongguan, China).

2.3 Drying experiments

2.3.1 Freeze drying

Samples were preliminary frozen at -40°C for 4 h, then put them into drying chamber and vacuumize, maintaining below 10 Pa. The heating board temperature was set at -15°C during the lyophilisation period based on the results of preliminary experiments. The samples were dehydrated until the final moisture content (13% w.b.) and predetermined conversion points were reached, respectively.

2.3.2 Microwave vacuum drying

The freeze dried samples were in succession put tidily in the microwave vacuum chamber which was kept under the absolute pressure of 11 kPa at -40°C and dehydrated until they reached the final moisture content (13% w.b.) under different initial microwave power density levels.

2.3.3 Orthogonal experiment

According to the preliminary drying experiments, $L_9(3^4)$ orthogonal experiment was designed with three factors of conversion point (A), initial microwave power density (B) and intermittent ratio (C), and five indexes of rehydration rate, shrinkage rate, drying time, energy consumption and chroma changes (ΔL^* , Δa^* , Δb^* , ΔE^*). Table 1 shows the variable levels.

Table 1 Factors and levels of $L_9(3^4)$ orthogonal experiment

Factors and levels	1	2	3
FD time of conversion point (A)/h	16	24	8
Initial microwave power density (B)/W · g ⁻¹	1.7	2	2.3
Intermittent ratio (C)/30 s	12s-on/ 18s-off	14s-on/ 16s-off	20s-on/ 10s-off

2.4 Determination of the related parameters

2.4.1 Eutectic and co-melting point

The eutectic and co-melting point were measured by a multimeter according to the change trend of resistance^[7].

2.4.2 Moisture content

The moisture content (wet basis) was measured according to the "National Standard Method for Determination of Moisture in Foods" (GB/T 5009.3-2003).

$$M(\%) = \frac{m_r - m_d}{m_r} \times 100\% \quad (1)$$

where, m_r is real-time mass, g; m_d stands for the mass of dry material, g.

2.4.3 Rehydration rate

The dried samples were immersed in distilled water at 20°C and weighed every four hours with free water on the surface removed, this trial lasted for 24 h.

$$R_f(\%) = \frac{m_r - m_d}{m_d} \times 100\% \quad (2)$$

where, m_r is real-time mass, g; m_d stands for the mass of dried material, g.

2.4.4 Shrinkage rate

The vernier caliper was used to measure sea

cucumber body length before and after drying.

$$r(\%) = \frac{L_b - L_a}{L_b} \times 100\% \quad (3)$$

where, L_b and L_a are the lengths (mm) before and after drying, respectively.

2.4.5 Energy consumption

The energy consumption (kJ) required for drying every group of samples (110±3) g was calculated according to the real-time power and operation time.

2.4.6 Chroma changes (ΔL^* , Δa^* , Δb^* and ΔE^*)

Both dorsal and abdominal chroma changes of sea cucumber caused by drying were accurately measured and recorded by the colorimeter with three chroma parameters and the ΔL^* , Δa^* , Δb^* and ΔE^* were calculated as follows.

$$\Delta L^* = L^*_2 - L^*_1, \Delta a^* = a^*_2 - a^*_1, \Delta b^* = b^*_2 - b^*_1$$

$$\Delta E^* = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \quad (4)$$

where, L^*_1 and L^*_2 represent the degree of brightness (100) and darkness (0) before and after drying, respectively; a^*_1 and a^*_2 represent the degree of redness (+) and greenness (-) before and after drying, respectively; b^*_1 and b^*_2 represent the degree of yellowness (+) and blueness (-) before and after drying, respectively. ΔE^* is the total chroma changes. All of the experiments above were repeated three times and the average value was adopted.

3 Results and discussion

3.1 FD process control

Eutectic and co-melting point were measured to determine the suitable lyophilisation temperature. Eutectic point is the temperature at which all the material moisture congeals to ice during the freezing process. Lyophilisation temperature is required to be lower than the eutectic point. Co-melting point is the temperature at which material moisture begins to melt from solid to a liquid state in the heating process. A sudden increase and decrease will be seen in the rate of resistance change at the eutectic and co-melting point, respectively.

Resistance curves of the boiled samples are shown in Figures 1 and 2. Eutectic and co-melting point were proved to be -11.5°C and -6.5°C, respectively, so the lyophilisation temperature was determined at -15°C to

guarantee the sublimation drying instead of desorption drying for a relatively higher drying rate.

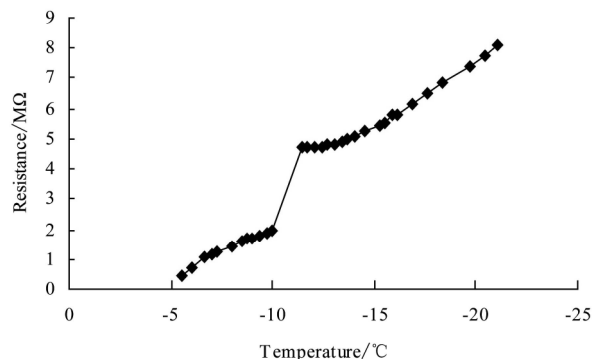


Figure 1 Eutectic point measurement

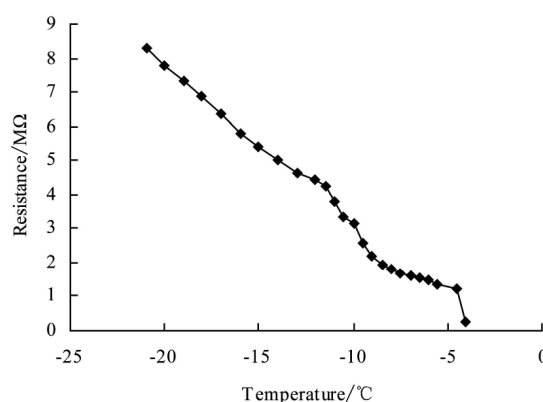


Figure 2 Co-melting point measurement

Figure 3 shows that the conventional FD process took 48 h. Such long drying time was on account of the low thermal conduction rate at low temperature^[8]. For the purposes of shortening drying time, reducing energy consumption and being applicable for practical production operation, the 8 h, 16 h and 24 h which stands for the moisture content of 70.3%, 61.8%, and 52.6%, respectively, were chosen for experiment as conversion point.

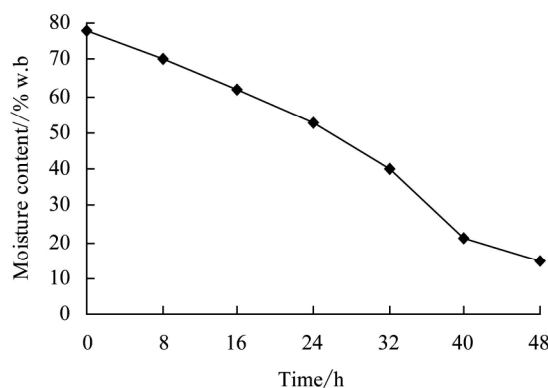


Figure 3 FD drying curve of sea cucumber

3.2 MVD process control

Figure 4 shows the MVD process of sea cucumber in the state of non-pulse continuous microwave. The higher microwave power density, the shorter time was required for drying, and energy consumption was reduced (Figure 5). It is because the microwave supplies the heat for moisture evaporation. A relatively high microwave power density can accelerate the evaporation and increase the drying rate. It took only 100 min with the microwave power density of 2.5 W/g, however, samples got partial charred on the surface by reason of the inherent nonuniform distribution characteristics of microwave heating, and excessive microwave power density.

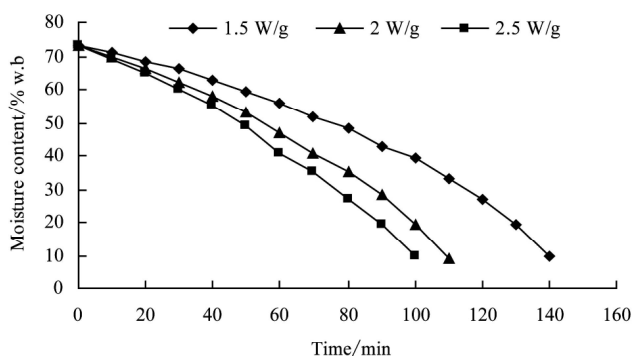


Figure 4 MVD drying curves of sea cucumber under the different initial microwave power densities

Figure 5 shows that the microwave power density exercised influence over rehydration rate, shrinkage rate and energy consumption. As the initial microwave power density increased, rehydration rate was improved, but shrinkage rate was augmented, and the nonuniform shrinkage led to distortion in materials with the initial microwave power density of 2.5 W/g.

Samples dealt with 2 W/g and 1.5 W/g were intact on

the surface with low shrinkage rate in shape. So 1.7 W/g, 2 W/g and 2.3 W/g were chosen for the orthogonal experiment.

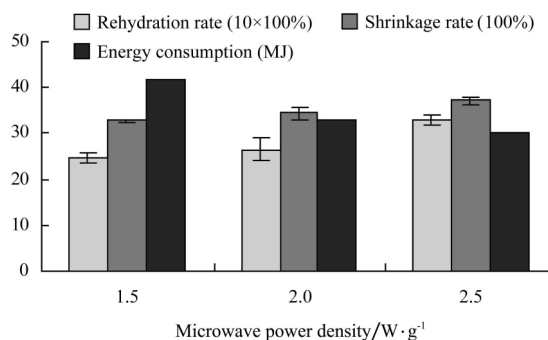


Figure 5 Effects of initial microwave power density

3.3 Effects of the intermittent ratio of the MVD on the rehydration rate

Table 3 shows that the intermittent ratio of the MVD affected the rehydration rate of sea cucumber significantly ($\alpha \leq 0.05$). This is because that during the deceleration period of drying, the moisture content in the material is higher than that of the surface, so the intermittent microwave drying allows the internal moisture to migrate from the interior to the surface under the moisture gradient and evaporate when the microwave is off, which is known as the tempered period^[9]. Tempered phenomenon is propitious for drying in respect that it could not only remove some water without consuming energy, but also balance the water distribution of the material, which results in heating rate slowing down and over-drying avoidance, thus protects the heat-sensitive nutrients, and improves product quality. Table 2 shows that the maximum rehydration rate of 280.71% could be obtained with the intermittent ratio of 12s-on/18s-off.

Table 2 Effects of conversion point, initial microwave power density and intermittent ratio on the rehydration rate, shrinkage rate, drying time and energy consumption

Experiment No.	Variable levels			Experimental results			
	A	B	C	Rehydration rate (y1)/%	Shrinkage rate (y2)/%	Drying time (y3)/h	Energy consumption (y4)/kJ
1	1	1	1	250.89	14.48	16.55	118 872
2	1	2	2	111.02	26.18	16.50	119 112
3	1	3	3	174.50	21.75	16.38	119 512
4	2	1	2	99.84	38.82	24.23	164 968
5	2	2	3	215.04	38.94	24.28	166 408
6	2	3	1	208.28	38.04	24.32	165 288
7	3	1	3	264.64	26.01	9.50	84 816
8	3	2	1	280.71	23.14	9.48	77 496
9	3	3	2	156.96	24.10	9.25	77 316

Table 3 ANOVA showing the effects of variables on rehydration rate, shrinkage rate, drying time and energy consumption

Source of variation	<i>df</i> ^a	Rehydration rate/%		Shrinkage rate/%		Drying time/h		Energy consumption/kJ	
		MS ^b	<i>F</i> -value ^c	MS ^b	<i>F</i> -value ^c	MS ^b	<i>F</i> -value ^c	MS ^b	<i>F</i> -value ^c
A	2	3 322.02	8.60	265.49	19.74*	165.90	29 372.07**	5.52E+09	1 027.96**
B	2	571.44	1.48	6.66	0.50	0.01	2.08	4.19E+06	0.78
C	2	12 654.24	32.76*	17.12	1.27	0.01	1.98	9.43E+06	1.76

Note: ^a*df*, Degree of freedom; ^bMS, mean square; ^c*F*-value, ratio of variance estimate; *Significant at 5%; **Significant at 1%.

FD time of conversion point also impacted rehydration rate, and the rehydration rate of sea cucumber freeze dried for 8 h was much better appreciated than those dried for 16 h and 24 h; initial microwave power density had a little effect on rehydration rate, while 1.7 W/g was better on the whole (Table 2).

3.4 Effects of FD time on the shrinkage rate

Table 3 shows that the FD time of conversion point influenced the shrinkage rate of sea cucumber significantly ($\alpha \leq 0.05$). Although the lowest shrinkage rate was obtained when the FD time of conversion point was 16 h, 8 h had similar effects with 16 h, all remarkably superior to 24 h (Table 2). It indicates that a lengthy FD period in the sea cucumber FD-MVD is detrimental. Considering the drying time and energy consumption, 8 h could be taken as the optimal conversion point.

Effects of initial microwave power density and intermittent ratio on shrinkage rate were not significant and the lower shrinkage rate could be obtained with the initial microwave power density of 1.7 W/g and intermittent ratio of 12s-on/18s-off (Table 2).

Compared to the single MVD, FD-MVD excelled in shrinkage rate apparently since the shrinkage rate of FD-MVD with 8 h and 16 h FD was 14% to 26%, much superior to that of MVD (Figure 5).

3.5 Effects of FD time of conversion point on drying time

Table 3 shows that FD time of conversion point had a significant effect on the drying time ($\alpha \leq 0.01$) which meant the MVD process shortened the drying time remarkably. It is because the paths for water diffusion can be maintained during the MVD process, resulting in high magnetic conductivity^[10]. Consequently, material at a wide range of moisture content can be heated internal and external synchronously, and dried at extremely high mass transfer efficiency.

The total drying time for FD-MVD was 9 h to 25 h, which was 19% to 51% of that for the traditional FD. Especially, it took less than 10 h of processing time when the FD time of conversion point was 8 h, which was only 57% and 39% of that for 16 h and 24 h, respectively. It is because the temperature of MVD (30°C to 40°C) was much higher than that of FD (-15°C) despite the higher vacuum degree of FD, more heat was supplied for moisture evaporation by MVD, significantly speeding the drying rate.

3.6 Effects of FD time of conversion point on energy consumption

Table 3 shows that FD time of conversion point had a significant effect on the total energy consumption ($\alpha \leq 0.01$). When the FD time of conversion point was 8 h, energy consumption was lowest, only 67% of that for 16 h and 48% for 24 h, reducing cost substantially. Reason for this is the efficient MVD with fast dehydration rate and incredible short drying time; meanwhile, FD is a much higher energy consumption process.

3.7 Effects of experimental factors on chroma changes (ΔL^* , Δa^* , Δb^* , ΔE^*)

Though none of the three experimental factors showed the significant effect on chroma changes, Table 5 indicates that the relationship between FD time of conversion point and dorsal and abdominal darkness changes was inerratic. From Table 4, the longer the FD period lasted, the less chroma changed, namely, 24 h of FD was the best in chroma retention. Reason for this phenomenon is that during FD procedure, the moisture content is dehydrated by sublimation of ice crystals in situ, leaving physical form, chemical and biological properties basically unchanged, thus avoiding the chroma changes^[11]. In the process of MVD, the chroma changes were related to material characteristics, drying time and

the temperature, in general it increased with the moisture content^[12].
temperature and drying time in the material of medium

Table 4 Effects on chroma changes (ΔL^* , Δa^* , Δb^* and ΔE^*)

Experiment No.	Experiment results							
	Dorsal color changes				Abdominal color changes			
	$\Delta L^*(y5)$	$\Delta a^*(y6)$	$\Delta b^*(y7)$	$\Delta E^*(y8)$	$\Delta L^*(y9)$	$\Delta a^*(y10)$	$\Delta b^*(y11)$	$\Delta E^*(y12)$
1	0.96	1.55	2.32	2.95	3.29	0.98	1.34	3.69
2	-3.42	0.39	-0.38	3.46	1.62	0.82	-0.04	1.82
3	-0.51	0.88	0.71	1.24	7.61	2.94	2.54	8.54
4	-1.77	-0.46	-3.51	3.96	2.96	0.99	0.71	3.20
5	0.10	-0.01	-1.61	1.61	6.09	1.53	2.42	6.73
6	-1.62	-0.31	-0.76	1.82	1.93	0.58	0.40	2.05
7	0.77	0.58	-0.03	0.96	6.16	2.06	2.37	6.91
8	1.12	1.09	0.63	1.69	8.71	2.06	3.06	9.46
9	4.61	2.62	4.03	6.66	8.97	3.27	2.89	9.98

Table 5 ANOVA showing the effects of the variables on chroma changes (ΔL^* , Δa^* , Δb^* and ΔE^*)

Source of variation	df ^a	Dorsal color changes															
		$\Delta L^*(y5)$		$\Delta a^*(y6)$		$\Delta b^*(y7)$		$\Delta E^*(y8)$		$\Delta L^*(y9)$		$\Delta a^*(y10)$		$\Delta b^*(y11)$		$\Delta E^*(y12)$	
		MS ^b	F-value ^c	MS ^b	F-value ^c	MS ^b	F-value ^c	MS ^b	F-value ^c	MS ^b	F-value ^c	MS ^b	F-value ^c	MS ^b	F-value ^c	MS ^b	F-value ^c
A	2	10.31	1.20	2.28	2.05	10.4	2.24	0.36	0.15	140.91	1.77	16.07	2.40	16.07	1.52	20.11	1.80
B	2	1.83	0.21	0.29	0.26	3.09	0.66	0.74	0.30	127.68	0.34	13.85	1.15	13.85	0.11	3.9	0.35
C	2	0.11	0.01	0.11	0.10	0.83	0.18	9.47	3.87	128.66	0.45	14.90	1.08	14.90	0.78	5.59	0.50

Note:^adf, Degree of freedom; ^bMS, mean square; ^cF-value, ratio of variance estimate.

4 Conclusions

High-quality dried sea cucumber with higher rehydration rate and lower shrinkage rate on the premise of short production time and low energy consumption could be produced by FD-MVD.

Effects of microwave intermittent ratio on the rehydration rate and the FD time of conversion point on the shrinkage rate and energy consumption were significant; the maximum rehydration rate of 280.71% was obtained with the intermittent ratio of 12s-on/18s-off; both a better shrinkage rate of 24.10% and the least energy consumption of 77 316 kJ could be obtained when the conversion point was 8 h FD. The initial microwave power density influenced notably the rehydration and shrinkage rate; the optimal level was 1.7 W/g, though resulting in a little higher energy consumption than that of 2.3 W/g. The conversion point of 24 h FD performed best in chroma retaining, but not obviously.

To reduce the drying time and energy consumption in the drying sea cucumbers, FD-MVD can be used to replace the traditional FD method.

Although MVD has been the subject of substantial research over the last decade, further investigation should be undertaken on modelling of MVD and optimization of FD-MVD method, especially based on nutrition retaining.

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