



Microwave freeze drying of sea cucumber (*Stichopus japonicus*)

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ABSTRACT

Freeze drying (FD) yields the best quality of dried sea cucumber but at the cost of long drying time and also the overall cost. Air drying (AD) gives an unacceptably poor quality product. To achieve faster drying along with a high quality product a microwave freeze drying (MFD) technique was developed to dry sea cucumbers. The relationship between corona discharge and microwave power at various pressures and initial moisture content conditions was studied to avoid the possibility of corona discharge during MFD. According to the drying characteristics of MFD, a control strategy for the MFD process was also developed. MFD reduced the drying time by about half of conventional FD process and provided a similar good product quality.

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1. Introduction

The sea cucumber (*Stichopus japonicus*) is an invertebrate animal living in sea water belonging to the phylum Echinodermata. Sea cucumbers are widely consumed extensively in China and Japan. The major edible part of the sea cucumber is the body wall consisting mainly of collagen and mucopolysaccharides which have active functions for nutrition (Cui et al., 2007; Liao, 2001). For example, there have been many reports about such valuable active components as collagen, acid mucopolysaccharide and triterpene glycoside (Vladimir, 2005; Yutaka et al., 1997).

Since sea cucumbers can autolyze after they are taken out of sea water, it is difficult to preserve and transport them. As a result, more than 80% of fresh sea cucumbers harvested all over the world are processed to produce a dehydrated product. Most of the sea cucumbers are dehydrated by traditional techniques, which involve salting, repeated boiling and expose to solar radiation for 2–3 days (Duan et al., 2007). This process is very long, making many active components lost. Furthermore, the rehydration time for the traditional solar-dried sea cucumber can be up to 3 days prior to cooking due to its extremely poor rehydration property.

In order to yield better quality dehydrated sea cucumbers, new drying technology for sea cucumber is needed. Hot air drying (AD) is the simplest and popular drying method, but it leads to a large deformation of the products and also thermally-induced deterioration (Ratti, 2001; Mujumdar, 2004). There are only limited reports in the literature on sea cucumber dehydration. Li et al. (2004) and Yun et al. (2006) have reported that freeze drying (FD) can be used to dry sea cucumbers. However, their FD process requires more than 20 h, resulting in high energy and capital costs. It is widely known that although FD is an excellent drying method from quality standpoint, its energy consumption is excessive compared with other methods of drying (Ratti, 2001). Microwave energy has been used successfully as a heat source in the food industry, because it provides fast and internal heating through dipole rotation and ionic conductance in the materials (Wang, 2000; Wang and Xi, 2005). Used as a heat source in FD, microwave field can heat the material volumetrically and it can be applied successfully in a vacuum environment under appropriate conditions (Wang et al., 2004; Zhang et al., 2007), thus greatly improving the rate of FD; this process is commonly called microwave freeze drying (MFD).

Although MFD can significantly improve the drying rate, there are some many problems to be resolved with this technology. One of the key problems is that the inherently non-uniform distribution of the microwave field leads to an uneven temperature distribution in the drying material (Dolan and Scott, 1994; Zhang et al., 2006). As a result, overheating and quality deterioration can take place. Another problem is the possibility of corona or

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plasma discharge under high vacuum and subsequent melting of the ice in the product (Lombrana et al., 2001). Copson (1958) carried out experiments on microwave freeze drying in early 1958. In the past several decades the technique has been developed only in the laboratory and not applied in industry. The few reports about MFD are mainly about heat and mass transfer modeling (Ma and Peltre, 1975; Wang et al., 1998; Tao et al., 2005).

To reduce the drying time and energy consumption in drying sea cucumbers, MFD can be used to replace the traditional FD method. In our study the microwave resonant cavity was designed as an effective multimode resonant cavity, which makes the electric field distribution uniform. As with most products uniform drying yields improved quality of dried sea cucumbers. Furthermore, cyclic on-off microwave field strategy with simultaneous cyclic applications of pressure can be used for regulating the microwave power and the chamber pressure to avoid overheating and corona discharge, respectively (Lombrana et al., 2001). There is also a relationship between the critical microwave power density for corona discharge and pressure (Mujumdar, 2004), so a suitable microwave power loading scheme must be designed and applied during MFD processes.

As magnetrons have become cheaper and better in performance over the past two decades, the cost of MFD equipment is not much higher compared with traditional FD. MFD processed sea cucumbers may provide premium market value as well. The objectives of this research were: (1) to determine relationship between the critical discharge microwave power and drying chamber pressure under four selected initial moisture contents to avoid corona discharge, and (2) to compare the performance of air drying, freeze drying and microwave freeze drying. The parameters examined were: the drying time, rehydration ratio, energy consumption, microstructure and product quality.

2. Materials and methods

2.1. Equipment

Fig. 1 shows the equipment used in our experiments. This equipment consists of two drying cavities where FD and MFD tests can be carried out. Materials dried in the FD cavity are heated by ohmic heating of the shelf. Samples dried in the MFD cavity are

subjected to a microwave field at 2450 MHz. Both vacuum and atmospheric pressure microwave drying can be conducted in the MFD cavity. During drying, vacuum is maintained by a vacuum pump; the temperature of the cold trap is sufficient to condense all vapor generated. To avoid non-uniform distribution of the microwave field, three magnetrons are placed at different angles. The power of the magnetrons could be adjusted continuously. The temperature of the drying samples is monitored using a model PI 1 fiber-optic probe (0.4 mm, Probing Technologies Inc., Shengzhen, China) designed for use in a microwave field. In the FD cavity, a thermocouple (2 mm) is used to monitor the temperature of samples.

2.2. Materials

Fresh sea cucumbers (*S. japonicus*) were purchased in local market and stored at -25°C before use in the experiment. Before other pretreatments, the gut and five pieces of tendons on the body wall of sea cucumbers were removed and the body walls were washed clean. The free water on the surface of sea cucumbers was removed with an absorbent filter paper. The sea cucumber body wall samples had $100(\pm 12.5)\text{g}$ in weight, $12(\pm 3.5)\text{cm}$ in length, and $5(\pm 1.5)\text{mm}$ in thickness.

2.3. Pretreatment

Considering the traditional manufacturing method and the final taste criteria, the sea cucumbers were boiled at 100°C for 20 min. Then the free water on the surface of the samples was removed with a filter paper. The samples subjected to freeze drying were loaded onto a material tray followed by freezing at -25°C for at least 8 h, which could ensure the free water of the samples frozen. Although the freezing temperature used in the present study is not necessarily the ideal freezing temperature for freeze drying, experiments showed satisfactory results.

2.4. Corona discharge experiments

The cause of corona discharge is that air ionization produces plasma – a process which takes place readily at low pressures. Such discharge is undesirable as it consumes excessive microwave

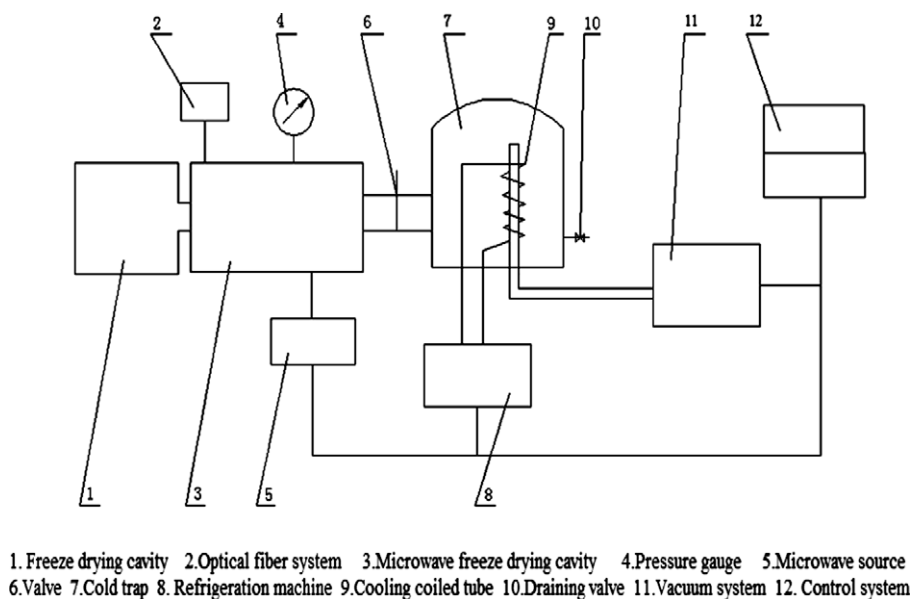


Fig. 1. Schematic diagram of the multifunctional microwave dryer used in this study.

energy and as the plasma can also lead to material burn. Furthermore, it can destroy the uniformity of the electromagnetic field distribution in the resonant cavity and produce strong electromagnetic waves, which can damage the magnetrons as well (Lombrana et al., 2001).

The samples were divided into four groups according to their original moisture contents (15%w.b., 30%w.b., 45%w.b., and 78%w.b.). Each group of samples weighed 300 g and was put into the MFD chamber and heated by microwave at different sub-atmospheric pressures (20, 40, 60, 80, 100, 150, and 300 Pa). When the pressure was stable, microwave energy was applied from the 150 W power source and increased in steps of 50 W until corona discharge took place. This microwave power level was defined as the critical discharge power.

2.5. Drying procedure

2.5.1. Hot air drying (AD)

The pretreated materials were spread uniformly on the bed (mesh) of a tray dryer (SHT, Sanxiong Machinery Manufacture Co. Ltd., Shangyu, China). Hot air flowed through the bed at 1.5 m/s velocity and 20% relative humidity. The temperature of the hot air was controlled and held at 60 °C. The samples were dehydrated until they reached the desired final moisture content (7%w.b.).

2.5.2. Freeze drying (FD)

The frozen materials (300 g) and tray were put into the FD chamber. Heating shelf temperature was set at 60 °C. The pressure of the drying chamber was set at 50 Pa during drying and the cold trap temperature was maintained at –40 °C. The samples were dehydrated until they reached the final moisture content (7%w.b.).

2.5.3. Microwave freeze drying (MFD)

The samples after frozen at –20 °C for at least 8 h were also dried to a final moisture content of 7%w.b. in the MFD chamber. Three microwave power levels (1.6, 2, and 2.3 W/g) were tested under 50 Pa of the absolute pressure and –40 °C of the cold trap temperature.

All the experiments were repeated twice and the average value of three measurements of moisture content for each treatment was used to plot the drying curves. The dehydrated samples were packed immediately into polyethylene bags after drying for further analyses.

2.6. Determination of the related parameters

2.6.1. Moisture content measurement

The sample moisture content was determined by drying in a vacuum oven at 60 °C until constant weight was reached (AOAC, 1980).

2.6.2. Rehydration ratio (RR)

The dried samples were soaked in 25 °C distilled water for 2 h, and then put on the filter paper of a Büchner funnel, which was held on a suction flask evacuated for 30 s to remove free water on the surface. The sample weighing was performed in triplicate. The rehydration ratio (RR) was estimated as follows:

$$RR = \frac{W_r}{W_d} \quad (1)$$

where W_d and W_r were the weights (g) of samples before and after rehydration, respectively.

2.6.3. Texture analysis

The texture characteristics of the sea cucumbers were measured using a Texture Analyzer (TA-XT2, Stable Micro System Ltd., Leicestershire, UK) fitted with a spherical probe (P/0.5). The pre-speed, test-speed, and post-speed were 3.0 mm/s, 1.0 mm/s, and 5.0 mm/s, respectively, and the deformation ratio was 50%. A force–time curve was recorded and analyzed using the software of Texture Exponent 32 (Surrey, UK). To dehydrate sea cucumber, the indices of hardness after rehydration were emphasized. The texture of dried samples was measured after soaked in 25 °C distilled water for 2 h. These tests were performed in triplicate.

2.6.4. Color

The color of dried samples was measured using a spectrophotometer (Model WSC-S, Shanghai Shengguang Instrument and Meter Co. Ltd., Shanghai, China). The results are expressed as Hunter L^* , a^* , b^* , respectively, where L^* is the degree of lightness, a^* the degree of redness (+) and greenness (–), and b^* the degree of yellowness (+) and blueness (–). The Hunter L^* , a^* , b^* values of each treatment were determined in triplicate.

2.6.5. Sensory evaluation

The sensory evaluation of dried samples was carried out by a taste panel of nine untrained judges. The panelists were asked to indicate their preference for each sample, based on the quality attributes of color, appearance, texture, aroma/flavor, and overall acceptability. A balanced 10-point hedonic rating was employed for all the attributes evaluated where 9–10 denoted “like very much”, 7–8 “like”, 5–6 “neutral”, 3–4 “dislike”, and 1–2 indicated “dislike very much”. The judges were asked to give their remarks about each of the samples.

2.6.6. Energy consumption

The total energy consumption during MFD was measured by an ammeter (Le Qing Electrical Energy Instrument Ltd., Shanghai, China), and the energy consumption required to remove 1 kg of water was calculated.

2.6.7. Microstructure examination

Samples from differently treated sea cucumbers (fresh, FD and MFD) were selected for microstructure examination using a scanning electron microscope (SEM). Pieces (2 × 2 × 2 mm) were excised from the dried samples and placed in a fixative containing 2.5% glutaraldehyde/2% paraformaldehyde in a 0.1 mol/L phosphate buffer overnight at 4 °C. The specimens were then rinsed in a phosphate buffer, post-fixed in 1% osmium tetroxide in phosphate buffer and dehydrated in a serial ethanol solution containing 30%, 50%, 70%, 95% and 100% ethanol for 15 min in each solution. The ethanol in the dehydrated samples was removed with two changes of 100% acetone at 10 min for each, then another 10 min with a mixture of acetone and hexamethyldisilazane (HMDS) (1:1) followed by two changes of 100% HMDS. The HMDS was air-dried overnight in a fume hood. The samples were cut along the muscle fibers using a razor blade to produce longitudinal sections. The specimen fragments were then mounted on aluminum stubs, coated with gold and examined and photographed in a SEM (Quanta-200, FEI Company, Eindhoven, The Netherlands) using an accelerating voltage of 10 kV.

2.6.8. Determination of amino acid

The oven-dry and finely ground samples were hydrolyzed for 24 h with 6 mol/L hydrochloric acid under the vacuum condition, then the volumetric flask was filled with distilled water and the determination was carried out using an Amino Acid Analyzer (HP1100, Agilent Technologies Inc., Santa Clara, CA, USA).

2.7. Statistical analysis

Analysis of variance (ANOVA) and the test of mean comparison according to Tukey's honest significant difference (HSD) were conducted at the level of significance of 0.05. The statistical software of SPSS System (version 10.0) for Windows was used for the analysis.

3. Results and discussion

3.1. Relationship between critical discharge microwave power and drying pressure

Fig. 2 shows that a pressure range from 100 to 200 Pa can cause readily corona discharge in MFD. Regardless of the moisture content, the critical discharge microwave power was the lowest when the cavity pressure was about 150 Pa. For freeze drying, although low pressure can improve the drying rate, the practical pressure range can be set at 50–100 Pa. This pressure range ensures that no corona discharge occurs.

The moisture content had a clear effect on the critical discharge microwave power (Fig. 2). Higher moisture content of the samples had a higher critical discharge power at a fixed pressure. Air discharge took place readily with decreasing moisture content. As a result, the microwave power must be controlled precisely during MFD procedure. When most of free water was removed, the microwave power should be reduced.

3.2. Characteristics of MFD

Fig. 3 showed that the traditional FD process needs the longest drying time (18 h). This is because FD, under vacuum conditions, supplies the sublimation heat by conduction or radiation (Ratti, 2001; Matteo et al., 2003). The rate of heat transfer is slow and thus drying takes a long time since formation of liquid water during drying must be avoided. AD had the fastest drying rate due to the high convection heat transfer coefficient in the hot air dryer. The AD process needs only about 8 h. MFD takes 12 h of processing time, which is about 40% less than that for conventional FD. Overheat may lead to rise of internal steam pressure, which will result in partial melting of the ice crystals, and then result in decline of product quality. As a result, even if the microwave can heat samples efficiently in the vacuum environment, the rate of heat transfer must still be controlled to avoid melting during the entire process.

Table 1 shows that air-dried product has poorest rehydration properties and maximum hardness. The main reason for this

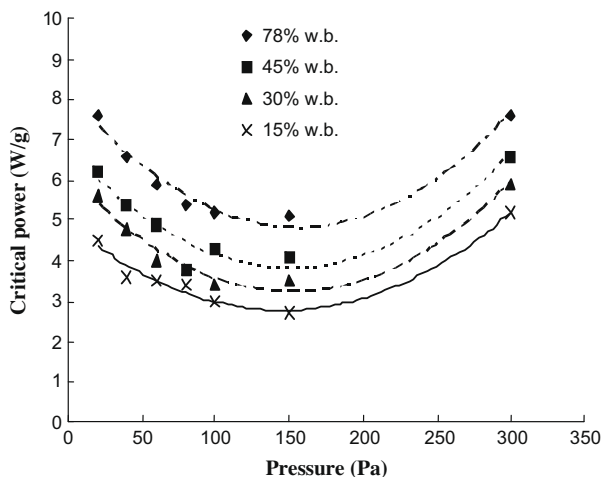


Fig. 2. Curves of critical discharge microwave power versus pressure.

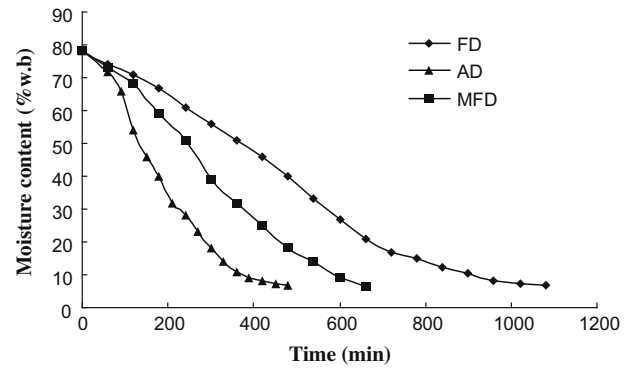


Fig. 3. Drying curves of sea cucumbers under different drying methods.

Table 1

Effect of drying method on energy consumption and product quality.

Drying method	Hardness (g)	Rehydration ratio	Energy consumption (kJ/kg H ₂ O)
AD	146.56 ± 2.62 ^{a*}	1.89 ± 0.32 ^b	8864.8 ± 73.2 ^c
FD	90.34 ± 1.83 ^b	3.85 ± 0.48 ^a	72628.6 ± 168.8 ^a
MFD	100.46 ± 2.02 ^b	3.16 ± 0.43 ^a	49566.8 ± 105.6 ^b

* Different letters indicate a significant difference ($P \leq 0.05$) in a column.

Table 2

The comparison of the amino acid content of sea cucumber processed with different treatments (g/100 g dry basis).

Amino acid	Sea cucumbers after different treatments			
	Fresh	AD	FD	MFD
Asp	7.63 ^{a*}	6.99 ^b	6.73 ^b	6.54 ^b
Glu	11.92 ^a	11.90 ^a	11.0 ^a	11.12 ^a
Ser	3.97 ^b	4.04 ^a	3.94 ^b	3.91 ^b
His	0.67 ^a	0.48 ^b	0.50 ^b	0.48 ^b
Gly	13.10 ^b	15.01 ^a	13.18 ^b	13.20 ^b
Thr	3.91 ^a	3.65 ^b	3.81 ^a	3.84 ^a
Arg	6.22 ^a	6.39 ^a	6.17 ^a	6.13 ^a
Ala	5.58 ^b	5.99 ^a	5.59 ^b	5.55 ^b
Tyr	1.81 ^a	1.51 ^c	1.76 ^b	1.74 ^b
Cys-s	0.32 ^a	0.26 ^b	0.25 ^b	0.26 ^b
Val	2.74 ^a	2.35 ^c	2.45 ^b	2.41 ^b
Met	1.15 ^a	1.04 ^b	0.99 ^b	1.02 ^b
Phe	1.57 ^a	1.27 ^b	1.54 ^a	1.58 ^a
Ile	2.20 ^a	1.81 ^c	1.97 ^b	1.91 ^b
Leu	3.20 ^a	2.72 ^b	2.65 ^b	2.61 ^b
Lys	2.09 ^a	1.71 ^b	1.68 ^b	1.61 ^b
Pro	6.72 ^c	9.88 ^a	9.03 ^b	9.12 ^b
Summation	74.80 ^b	77.00 ^a	73.24 ^b	73.03 ^b

* Different letters indicate a significant difference ($P \leq 0.05$) in a row.

phenomenon is that AD can lead to fast water evaporation of surface moisture, and at the same time a number of inorganic salts migrate to the evaporation surface with the water; this results in surface hardening of the sea cucumber. As a consequence, rehydration of air-dried (AD) sea cucumber is difficult. However, AD had the lowest energy consumption. For products subjected to FD and MFD, the rehydration ratio and hardness showed no significant difference ($P > 0.05$). This implies that MFD also can give the same product quality as FD does. Both FD and MFD consumed more energy than AD because of the need to maintain a very low temperature ($-40\text{ }^{\circ}\text{C}$) as well as a high vacuum environment. However, MFD was found to consume 32% less energy compared to the conventional FD because of the greatly reduced drying time.

Table 2 lists 17 different amino acids detected in the dried sea cucumbers except for tryptophan that is destroyed by hydrolysis.

Table 3
Effect of different drying methods on color and sensory value of sea cucumbers.

Drying method	L^*	a^*	b^*	Sensory value
AD	38.66 ± 0.03 ^b	2.6 ± 1.70 ^b	2.54 ± 0.04 ^b	3.25 ± 0.28 ^b
FD	64.81 ± 0.02 ^{a,*}	12.24 ± 0.03 ^a	20.30 ± 0.01 ^a	9.42 ± 0.32 ^a
MFD	66.09 ± 0.04 ^a	15.13 ± 0.04 ^a	19.88 ± 0.07 ^a	9.50 ± 0.41 ^a

* Different letters indicated a significant difference ($P \leq 0.05$) in a column.

Eight of them are essential amino acids, which cannot be synthesized by the human body. It was observed that there was no significant difference ($P > 0.05$) between FD and MFD products in terms of the total contents of the amino acids.

It is known that amino acids contributing to the flavor of sea cucumbers include Asp, Glu, Gly, Ala, Ser and Pro (Konosu, 1973; Chiou and Lai, 2002). It should be noted that Ser, Gly, Ala and Pro increased significantly during AD ($P < 0.05$), causing the product to develop a strong odor. This is why people prefer to eat traditional dehydrated sea cucumbers obtained by AD. The increase of these four particular amino acids during MFD had no significant difference with that during FD ($P > 0.05$), suggesting that the flavor of MFD products was close to that of the FD ones.

As for color of the dried sea cucumber, L^* expresses the brightness of sample, high L^* means brighter color. Table 3 shows that L^* of AD sea cucumbers is much lower than the others. This means that AD samples possess a much darker color than other two methods, and their sensory value is also low because AD processing leads to major shrinkage. There was no significant difference between MFD and FD samples in terms of both color and sensory

values ($P > 0.05$). Thus, MFD ensures good appearance quality of the product.

Fig. 4 shows the SEM images of dried sea cucumbers obtained using different drying methods. From Fig. 4A, the muscles and collagen fibers of the fresh sea cucumber were observed to be slender and arranged in a specific direction with a large gap in between. From Fig. 4B, C and D, it can be seen that the muscles and collagen fibers in the sea cucumber did not possess specific directions. This implies that after the loss of water in the sea cucumber, the fiber arrangement was altered. Most of the collagen fibers were broken, forming a reticulation structure, which made the dried sea cucumbers a unique texture. On the other hand, the AD sea cucumbers had clearly lower porosity than the FD and MFD sea cucumbers, thus resulting in poorer rehydration ability. There was no obvious difference between the FD and MFD sea cucumbers in terms of their microstructure. This implies that MFD not only shortened the processing time of FD, but also produced products with similar micro-structural characteristics to the FD sea cucumbers.

3.3. MFD process control

Temperature–time history in the freeze drying process is important as it reflects the general drying performance. From Fig. 5, the MFD process could be divided three phases: the phase of temperature fall, the sublimation phase and the final desorption phase. Unlike in the traditional FD, the sublimation phase in the MFD process is relatively shorter when the product temperature rises quickly. The temperature in the desorption phase rises more quickly than that in the sublimation phase. At the beginning of 0.5 h, the vacuum pump was on but the magnetron was off. The temperature began to drop because water in the material sublimed

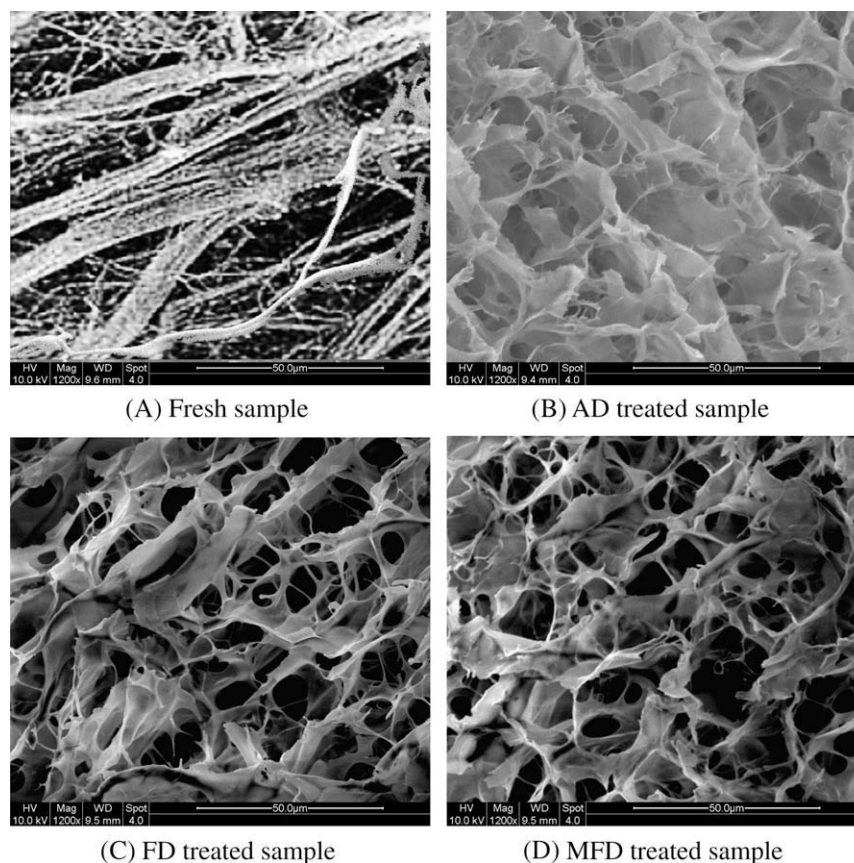


Fig. 4. SEM images of sea cucumbers under different drying methods.

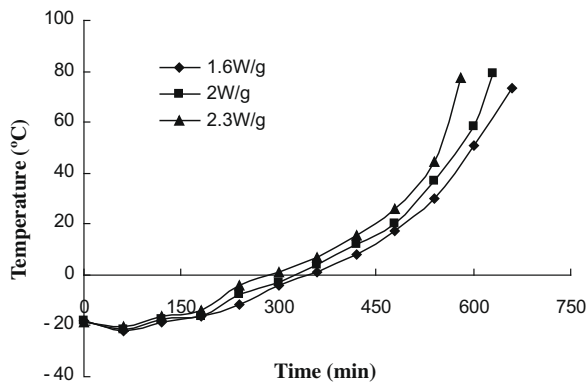


Fig. 5. Temperature curves of sea cucumbers in MFD under different microwave powers.

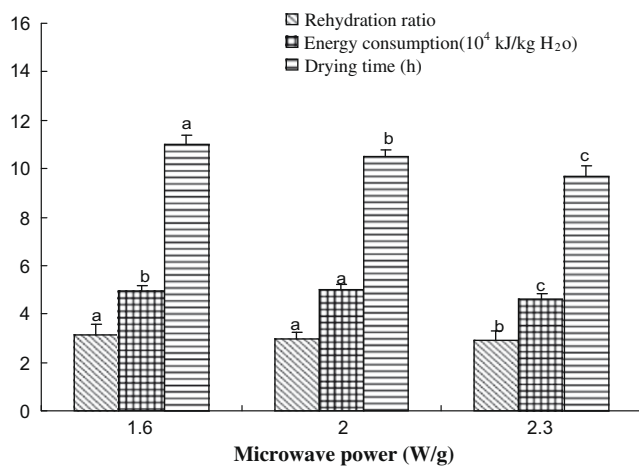


Fig. 6. Effect of microwave power on drying time, energy consumption and rehydration ratio using MFD process. Different letters indicate a significant difference ($P \leq 0.05$).

by absorbing heat in a vacuum environment. After the magnetron was turned on, the sublimation rate increased as the water in sea cucumbers absorbed microwave energy. The sublimation phase lasted about 5 h, removing most of the free water. In order to ensure that the water was removed by sublimation, the temperature in the sublimation phase should be lower than the co-melting temperature. It was found that the temperature changed insignificantly in the sublimation phase under different microwave power levels (Fig. 5). As a result, higher microwave power could be applied in this phase to improve the drying rate. In the desorption phase, the product temperature increased greatly with increasing microwave power (Fig. 5).

Compared to traditional FD, the duration of the desorption phase is reduced greatly in MFD. The total drying time for MFD was from 9 to 11 h, which was nearly half of that for the traditional FD. In fact, in the desorption phase, as most of the free water had been removed, a small energy input could make the temperature rise quickly. This characteristic also could be looked at as the indicator of the end of drying. Thus, to save energy and obtain better quality, higher microwave power should be applied in the sublimation phase, but a lower microwave power should be applied in the desorption phase.

Fig. 6 shows that excessively high microwave power (2.3 W/g) makes the rehydration ratio to decrease, which could be because the high microwave power level might lead to hardening of sea

cucumber body wall as well as possible melting of the ice to water phase causing shrinkage. The drying time decreases significantly with increasing microwave power ($P < 0.05$). The effect of microwave power on energy consumption is different from that on the drying time. The energy consumption at 2 W/g is the largest, followed by 1.6 and 2.3 W/g. Better product quality is obtained by setting the microwave power level at 2 W/g although the least energy consumption was achieved at microwave power of 2.3 W/g due to shorter processing time.

4. Conclusions

Freeze drying maintains product quality of sea cucumbers but takes a long processing time and needs high energy consumption. Air drying results in poor product quality although it consumes much less energy. Microwave freeze drying can replace conventional freeze drying because it greatly reduces the drying time and energy consumption while producing the same product quality as that of conventional freeze drying. In order to avoid corona discharge during microwave freeze drying, the cavity pressure should be applied within the range 50–100 Pa. The microwave power at different phases of the MFD process would be optimized based on energy efficacy and product quality.

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