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Study on Microwave Freeze-Drying of Krill

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Abstract: Antarctic krill (*Euphausua superba*) need to undergo freeze-drying to facilitate lipid extraction, but freeze-drying is time-consuming and energy-intensive, resulting in high processing costs. Microwave heating technology can reduce freeze-drying time and lower energy consumption costs. The objective of this study was to establish a drying kinetic model to help the microwave freeze-drying process by predicting krill drying time and evaluating the impact of the drying process on krill quality. The results showed that changing the microwave power did not alter the total energy requirement to complete drying when the sample weight was fixed. The total energy requirement for microwave drying increases with the sample weight. Comparing the three methods of freeze-drying (FD), microwave freeze-drying (MWFD), and hot-air drying at 55 °C (HAD) showed that they took 18, 0.67, and 16 h, respectively, to reach the drying endpoint for krill. Overall, HAD resulted in browning, shrinkage, and quality degradation of krill due to its high temperature and long duration. While the appearance and active ingredient contents of FD krill are slightly better than those of MWFD krill, FD requires a longer process and more energy. MWFD can reduce drying time by 20 times and energy consumption by 95% compared to FD.

Keywords: krill; freeze-drying; microwave; energy consumption; quality

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1. Introduction

Antarctic krill, a marine planktonic crustacean, is renowned for its high nutritional value, being rich in protein, lipids, minerals, and chitin. Krill oil is unique due to its diverse lipid composition, including a significant amount of phospholipids (PLs) combined with eicosapentaenoic acid (EPA) and docosahexaenoic acid (DHA) [1]. Additionally, krill oil contains various minor bioactive components, such as astaxanthin, sterols, tocopherols, vitamin A, flavonoids, and minerals. Research has highlighted the health benefits of krill oil, which include anti-inflammatory effects, cardiovascular disease (CVD) prevention, neuroprotection, and anticancer activities [2]. As a result, there is a substantial demand for krill oil in the functional oil market after fish oil.

Krill have very active digestive enzymes, such as proteases [3] and lipase [4], which not only affect their nutritional value but also have an impact on later storage, transportation, and processing [5]. Krill must first be dried to remove excess moisture, reducing the interference of water with organic solvents to facilitate lipid extraction. Furthermore, lowering the moisture content reduces the weight load during transportation, and they can be stored at normal temperatures instead of refrigerated. This is consistent with current energy-saving, carbon reduction, and sustainable environmental protection aims [6]. If hot-air drying is used, the drying temperature will accelerate the activity of krill's digestive enzymes; therefore, freeze-drying should be used to maintain the quality of dried krill [7].

During the sublimation process of freeze-drying, the frozen parts of food provide structural rigidity, preventing shrinkage and collapse of tissues during drying. This helps food maintain its original shape and preserves the structural integrity of its pores, giving freeze-dried products excellent rehydration properties. Additionally, the drying process

involves low temperatures and a vacuum, which reduces most chemical deterioration reactions, thus preserving the nutritional value of food during drying [8]. Freeze-drying can even retain probiotic strains in fermented products [9], maintain the gel-forming properties of proteins [10], and preserve the activity of antibodies [11]. However, traditional freeze-drying involves heat transfer barriers, making ice crystal sublimation difficult, which results in lengthy drying times and significant energy consumption, leading to high processing costs. Therefore, freeze-drying is often only applied to more expensive or sensitive raw materials [12].

Using the dielectric heating characteristics of microwaves as a heat source to accelerate ice crystal sublimation can enhance freeze-drying efficiency. This technique is called microwave-assisted freeze-drying. Microwaves do not need to transfer energy through solid, liquid, or gas mediums via temperature gradients. Instead, when acted upon by a microwave electric field, a dielectric material undergoes molecular movements, such as rotation, oscillation, and rearrangement. This friction with food tissues generates heat energy, which provides energy for ice crystal sublimation. Microwaves can directly penetrate food, heating the dielectric material throughout, resulting in better heat transfer efficiency. This shortens the freeze-drying time, reduces energy consumption costs, and maintains the excellent quality of dried products [13–15].

Over the past 30 years, several studies have investigated microwave-assisted freeze-drying of food products, pharmaceuticals, and antibodies. However, most of these studies were conducted using laboratory-scale setups. The primary goal of these studies was to identify appropriate microwave freeze-drying conditions or pretreatment methods to reduce drying time while maintaining good product quality. Examples include studies on strawberries [16] and pineapples [17]. Furthermore, there have been studies on the changes in the structure and properties of food tissues during the microwave freeze-drying process [18–20]. Effective moisture diffusivity coefficients have been calculated in microwave freeze-drying or conventional freeze-drying, following Fick's second law of diffusion [21,22]. Wang and Shi [23] introduced a numerical study on mass and heat transfer during the microwave freeze-drying process. Despite these studies, the use of drying kinetics to explore microwave freeze-drying processes has rarely been mentioned.

Although microwave freeze-drying can save time and energy while maintaining good quality, user-friendly industrial microwave freeze-drying equipment has yet to be made available. This is because "corona discharge" has long been a limiting factor in the development of microwave freeze-drying technology. The primary reason is that, in a high-vacuum environment, gas molecules in the microwave electric field are accelerated by the electric field, resulting in ionization. The ions then produce plasma, also called glow discharge. Corona discharge is an unnecessary waste of energy in the microwave freeze-drying process, causing a rapid increase in product temperature and the possibility of combustion. Furthermore, this phenomenon affects the uniformity of the electromagnetic field distribution in the microwave resonant cavity, resulting in powerful electromagnetic field echoes, which can severely damage the microwave magnetron. Within the vacuum range commonly used for microwave freeze-drying (0.06–4.59 mmHg), the critical electric field strength is higher at 2450 MHz than at 915 MHz. This indicates that gas is more stable and that there is less risk of dielectric breakdown at the high electric field strength of 2450 MHz. Avoiding the pressure range in which the lowest critical electric field appears helps increase gas stability in a strong electric field, minimizing dielectric breakdown [24]. It is crucial to manage both microwave power density and vacuum pressure to avoid plasma discharge or corona discharge during microwave freeze-drying [15].

The objectives of this study were to investigate the drying kinetics of microwave freeze-dried krill using two parameters: microwave power (W) and sample loading (g), to avoid thawing and corona discharge. Furthermore, the energy consumption and product quality (appearance, color, DHA, phospholipids, and total carotenoid content) of krill dried by three different drying methods (hot-air drying, freeze-drying, and microwave freeze-drying) were compared.

2. Materials and Methods

2.1. Raw Materials

Frozen fresh Antarctic krill blocks were provided by Chemsphere Technology, Ltd., Taipei, Taiwan. After thawing and filtering out external seawater, the krill were divided into 50, 100, 150, and 200 g samples, then stored in a freezer at −18 °C for future use. The initial moisture content of the krill was 90.31 ± 0.03%.

2.2. Analytical Reagents

Ninety-five percent ethanol, KOH, Na_2SO_4, HCl, CH_3COOH, $(NH_4)_6Mo_7O_{24}{\cdot}H_2O$, H_2SO_4, KH_2PO_4, H_2O_2, hydrochloric acid, and HNO_3 were purchased from Kanto Chemical Co., Ltd., Tokyo, Japan. Ricosanoate methyl ester, $C_{24}H_{48}O_2$, CH_3OH, n-hexane, L-ascorbic acid, ethylenediaminetetraacetic acid disodium salt 2-hydrate, and $C_{10}H_{14}N_2$ $Na_2O_8{\cdot}2H_2O$ were purchased from SIGMA-ALDRICH®, St. Louis, MO, USA. Boron trifluoride-methanol complex (20% solution in methanol) (BF_3) for analysis was obtained from Merck, Rahway, NJ, USA.

2.3. Experimental Equipment

The microwave freeze-dryer was assembled in the laboratory, and its appearance is shown in Figure 1. It included a microwave supply oven, a 2 L glass vacuum drying dish, a vacuum pump (0.1–1 torr), a condensation chamber, a compressor (−20 °C), a vacuum monitor, and a controller (for controlling microwave output power and output time). The equipment used and the provenances were as follows: a constant temperature water bath (B201; Firstek, New Taipei, Taiwan), a gas chromatograph (Varian GC-3400, Woonsocket, RI, USA), a vortex mixer (Vortex-Genie® 2; Scientific Industries Inc., New York, USA), a spectrophotometer (CT2700; ChromTech, Tokyo, Japan), a vacuum filtration pump (Rocker 300; Rocker Scientific Co., Ltd., New Taipei City, Taiwan), a homogenizer (SilentCrusher M; Heidolph, Burladingen, Germany), a microwave digestion system (MARS; CEM, Matthews, NC, USA), a heater (PC-420D; Coring Inc, Corning, NY, USA), a colorimeter (Color Flex; Hunter Lab, Reston, VA, USA), and an oven (Channel DCM-45, Sci-Mistry Co., Ltd., Yilan, Taiwan).

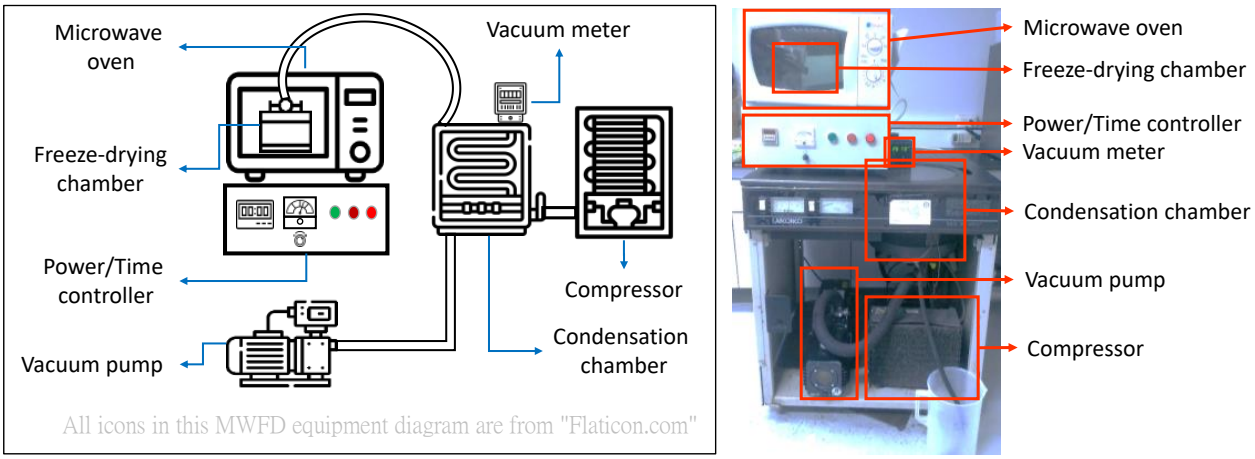

Figure 1. Schematic diagram of microwave freeze-drying equipment.

2.4. Drying Methods

2.4.1. Hot-Air Drying

The hot-air temperature was set at 55 °C. The pre-portioned frozen krill (50 g) was placed in the hot-air oven for drying. Every 2 h, the sample's weight was measured until the dimensionless moisture content (DMC) reached below 0.1; then, a drying curve was plotted for the kinetic analysis of the drying process. The dried krill samples were then collected for further analysis.

2.4.2. Freeze-Drying

First, the refrigeration compressor in the dehydration system was turned on to keep the condensation chamber below −20 °C. Then, the pre-portioned frozen krill (50 g) was placed in the glass drying container, and once the drying container was sealed, the vacuum pump was started to begin drying. Every 2 h, the sample was removed to measure its weight, with the drying process considered complete when the dimensionless moisture content (DMC) reached below 0.1. The DMC was calculated as follows:

$$DMC = \frac{W_t - W_0}{W_i - W_0} \quad (1)$$

where W_t is the weight of the sample measured at different drying times (g), W_i is the initial weight of the sample (g), and W_0 is the dry weight of the sample (g). After drying was complete, a drying curve was plotted for the kinetic analysis of the drying process. The dried krill samples were then collected for further analysis.

2.4.3. Microwave Freeze-Drying

First, the microwave output power (30, 40, 50, 60, 70, 80, 90, and 100 W) and the output time on the controller (with a pause every 10 min to facilitate experimental recording) were set. The compressor was then turned on to maintain the condensation system below −20 °C. The frozen krill (50, 100, 150, and 200 g) was quickly placed in the glass container. Once the container was sealed, the vacuum pump was promptly turned on. When the vacuum level dropped below 4.588 torr, the microwave power was turned on. Every 10 min, the sample was removed to measure its weight and temperature until the DMC reached below 0.1. After drying was complete, a drying curve was plotted for the kinetic analysis of the drying process. The dried krill samples were then collected for further analysis.

2.5. Drying Energy Consumption

During the drying process, an electrical power monitor (SPG-26MS, Son Dar Electronic Technology Co., Ltd., Taoyuan, Taiwan) was installed at the power source to record the power changes of each energy-consuming unit, including the microwave oven, the vacuum pump, and the compressor. The power changes of the vacuum pump and the compressor over time are shown in Figure 2. Since the power of both stabilized within 10 min, the power values used after stabilization were 345 W for the vacuum pump and 320 W for the condenser. Therefore, the total energy consumed could be calculated by multiplying the total drying time for each energy-consuming unit.

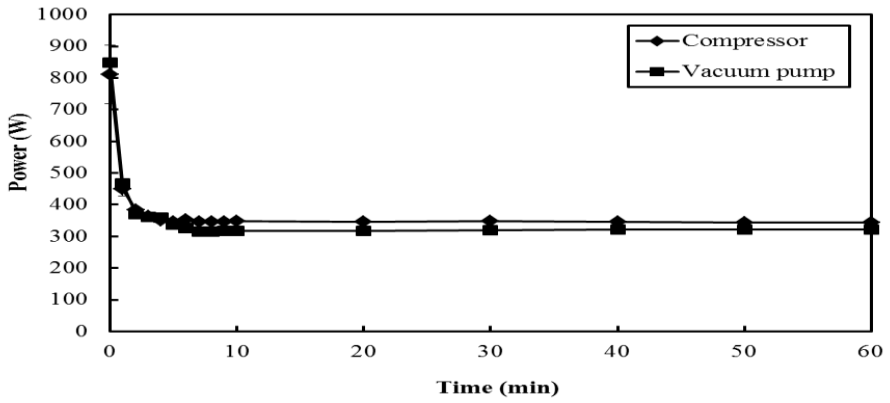

Figure 2. The power changes of the compressor and the vacuum pump during freeze-drying.

2.6. Quality Analysis

2.6.1. Appearance

The appearance of the samples was photographed using a camera (Pentax K-m). Black paper was used as the background to avoid reflections, and a ruler was placed in the image.

2.6.2. Color

The L*, a*, and b* values of the samples obtained by the different drying methods were measured using a colorimeter (Color Flex; Hunter Lab, Virginia, VA, USA) and standardized against a white calibration plate (X = 82.48, Y = 84.23, Z = 99.61, L* = 92.93, a* = −1.26, b* = 1.17). The L* value represents brightness: black (0) to white (100); the a* value represents red (+) to green (−); and the b* value represents yellow (+) to blue (−). The freeze-drying results were used as a reference to calculate the color difference (ΔE*) for microwave freeze-drying and hot-air drying.

The following formula was used:

$$\Delta E = \sqrt{\left(L_2^* - L_1^*\right)^2 + \left(a_2^* - a_1^*\right)^2 + \left(b_2^* - b_1^*\right)^2} \quad (2)$$

2.6.3. Crude Lipid Content

According to the AOCS [25], the finely ground samples had to be dried, and 5 g samples were placed in a cylindrical filter paper. The opening of the filter paper was plugged with defatted cotton to prevent sample loss, and diethyl ether was used as an extract solvent in a Soxhlet extractor at 60–70 °C in a water bath for 8 h. The concentrated lipids were maintained under reduced pressure at 40 °C until dry, and the increase in the flask's weight was measured to determine the crude fat content.

2.6.4. EPA and DHA Content

A quantity of 5 mL of the filtered 95% ethanol extract of krill was taken and placed in a test tube. The solvent was evaporated by passing nitrogen gas over it, and the weight of the crude extract was recorded. Then, 4 mL of 0.5N KOH/methanol solution and 0.1 mL of the internal standard methyl tricosanoate solution (5 mg C23/mL) were added. The test tube was shaken well (having ensured that the test tube mouth was wrapped with sealing tape) and heated in a water bath at 80 °C for a 20 min saponification reaction. After the reaction, 4 mL of 0.7N HCl/methanol solution and 5 mL of 20% BF3/methanol solution were used as a catalyst. The sample was then heated in a water bath at 80 °C for a 20 min methylation reaction. After the reaction, 5 mL of n-hexane was added to extract the fatty acid methyl esters from the methanol phase. The test tube was then left to stand to separate the layers, after which a pipette was used to draw the upper layer and drop it into a test tube containing anhydrous sodium sulfate to absorb moisture. The dehydrated supernatant was placed in a sample vial and sealed for later use.

The prepared fatty acid methyl esters (FAMEs) were analyzed for their fatty acid composition by injecting 3 μL of the sample into a gas chromatograph (Varian GC-3400, Woonsocket, RI, USA) using a micro-sample syringe. A GC capillary column (60 m × 0.32 mm i.d. × 0.25 μm film thickness) (CP7864; Supelco, Inc., Bellefonte, PA, USA) was employed to separate the methyl esters, which were then detected with a flame ionization detector (FID). Nitrogen was employed as the carrier gas, with a flow rate of 1 mL/min and a split ratio of 20:1. The injection port temperature was set to 250 °C, and the detection temperature was set to 280 °C. The initial column temperature was maintained at 150 °C for 0.5 min, then increased at a rate of 10 °C per minute to 180 °C, followed by a further increase at a rate of 1.5 °C per minute to 200 °C. Subsequently, the temperature was raised from 30 °C per minute to 230 °C and held for 5 min, before a final increase at a rate of 10 °C per minute to 250 °C, where it was held for 10 min, totaling 35 min, which procedure was modified from the AOCS [25]. The identification of individual FAMEs from the sample was accomplished by matching the retention times of unknown FAMEs with those of known FAME standard mixtures (Sigma-Aldrich Corp., Saint Louis, MO, USA). The contents of EPA and DHA were quantified by comparing their peak areas to those of a known-concentration internal C23 standard.

2.6.5. Phosphorus Content

Following the method by Tosi et al. [26] with modifications, a phosphorus standard solution (5 mg-P/L) was prepared using a 10% ascorbic acid solution, amine molybdate (60 mg/mL), 51% sulfuric acid, and anhydrous potassium dihydrogen phosphate (KH_2PO_4). Then, water was added to 0, 0.1, 0.3, 0.6, 0.9, 1.2, and 1.5 mL of the phosphorus standard solution to make up 10 mL volumes, to which 0.7 mL of ascorbic acid solution, 1 mL of sulfuric acid, and 1 mL of ammonium molybdate were added. These volumes were then heated in a water bath at 75 °C for 20 min, the absorbance was measured at 650 nm, and standard curves were plotted.

To measure the phosphorus content of krill oil, 0.5 g of dry shrimp powder was weighed and put in a microwave digestion tube. Then, 8.5 mL nitric acid and 1.5 mL hydrogen peroxide were added. The tube was then placed in a microwave digestion oven and reacted at 600 W for 5 min. Then, we waited for it to cool down for 1 h, took the reaction solution, and quantified it to 50 mL. Then, taking 0.25 mL of the quantitative solution, we added distilled water to quantify it to 10 mL, after which we added 0.7 mL of ascorbic acid, 1 mL of sulfuric acid, and 1 mL of ammonium molybdate. The tube was placed in a water bath at 75 °C for 20 min and then cooled to room temperature. We then took the reaction solution and measured the absorbance value at 650 nm. The value was substituted into the standard curve to calculate the phosphorus content (%) of krill.

2.6.6. Total Carotenoid Content

To obtain the total carotenoid content, we took 0.2 g of a dry sample, placed it in a centrifuge tube, added 10 mL of 95% ethanol, homogenized it at 12,000 rpm for 5 min, filtered it and collected the filtrate, measured the absorbance value of the filtrate at wavelengths of 665, 649, and 480 nm, and substituted these values into the following formula (modified from Wellburn [27]):

$$Ch_a = 12.19A_{665} - 3.45A_{649}$$

$$Ch_b = 21.99A_{649} - 5.32A_{665}$$

$$\text{Total carotenoid content}(\mu g/gd.b.) = \frac{(1000A_{480} - 2.14Ch_a - 70.16Ch_b)}{220} \quad (3)$$

where Ch_a and Ch_b are the concentrations of chlorophyll a and chlorophyll b in the extraction solution (μg/mL), respectively; A_{665}, A_{649}, and A_{480} are the absorbance values at wavelengths of 665, 649 and 480 nm, respectively; and $V_{ethanol}$ is the volume of the extraction solution (mL). The research extraction solution was ethanol, with a volume of 10 mL. $W_{dry\ krill}$ is the dry sample weight (g).

2.7. Statistical Analysis

Experimental data are presented as means ± standard deviations (SDs). A one-way analysis of variance (ANOVA) was conducted, followed by Duncan's multiple range tests of treatment means using the Statistical Analysis System (SAS 9.4; SAS Institute, Cary, NC, USA). Statistical significance was determined at a level of 0.05 ($p < 0.05$).

3. Results and Discussion

3.1. Effect of Microwave Power Density on Microwave Freeze-Drying of Krill

Figure 3 shows the freeze-drying curves and surface temperature changes of 50 g samples of krill at different microwave power levels. The results indicated that as the microwave power increased from 50 W to 90 W, the time taken for the krill to reach the drying endpoint reduced from 80 min to 40 min. In terms of temperature, during the initial stage of freeze-drying (0–10 min), the surface temperature of the krill samples increased rapidly, with the microwave energy supplying the sensible heat to increase the sample temperature.

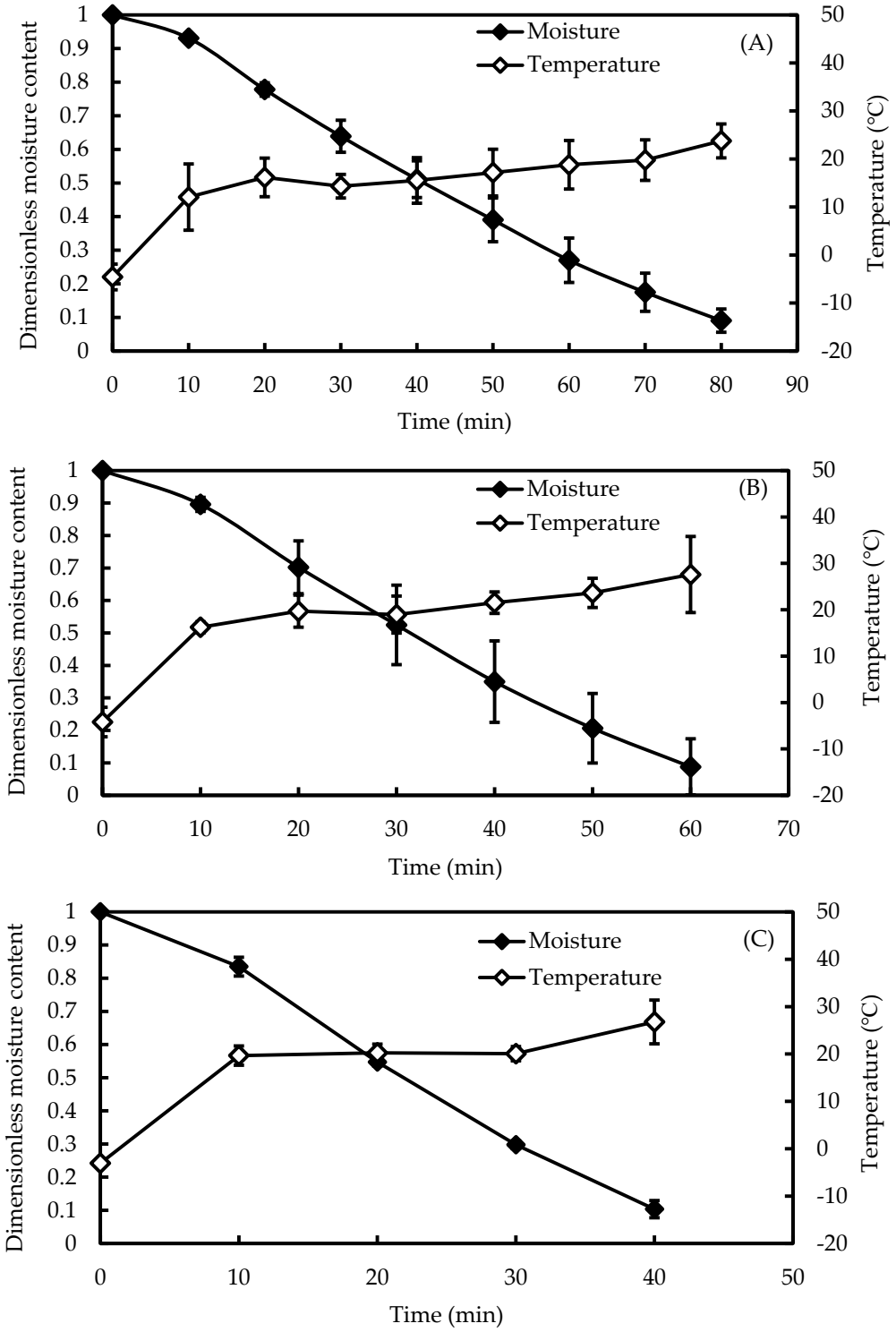

Figure 3. The surface temperature profiles and drying curves during the (**A**) 50 W, (**B**) 70 W, and (**C**) 90 W microwave freeze-drying of 50 g krill.

As the microwave power increased, the initial heating rate of microwave freeze-drying increased from 1.66 °C/min at 50 W to 2.27 °C/min at 90 W. In the middle stage of freeze-drying (after 10 min), the temperature stabilizes to around 25 °C, with less noticeable heating. During this period, the microwave energy mainly supplies the latent heat required for the sublimation of ice crystals. After reaching the drying endpoint (DMC < 0.1), if microwaves continue to supply, "corona discharge" will occur, causing the

surface temperature to rise rapidly, potentially leading to combustion and compromising product quality.

Duan et al. [28] experimented with microwave freeze-drying of 500 g of cabbage and showed that increasing the microwave power from 600 W to 900 W reduced the drying time from 6 h to 4 h. Wang et al. [29] used 450 g of instant vegetable soup as material and found that raising the microwave power from 225 W to 900 W shortened the drying time from 9 h to 3 h. In another study by Wang et al. [30] using potatoes, the microwave power (W) and sample weight (g) were integrated into the microwave power density (W/g). The results indicated that when the microwave power density increased from 1.4 W/g to 2.0 W/g, the drying time reduced from 10 h to 4 h.

Whole strawberries (125 g) were dried using MFD until the moisture content dropped below 10%. The process included two stages: sublimation and desorption. During the sublimation stage, the strawberries were heated at a constant rate of 0.035 °C/min until they reached 0 °C. In the desorption stage, the temperature increased at a constant rate of 0.2 °C/min until it ranged between 0 °C and 50 °C. The primary drying phase lasted 7 h, followed by a secondary drying phase of 4 h. The controlled heating rates ensured uniform drying, preventing issues such as burning and collapse, while also avoiding corona or plasma discharge [16].

According to a study on microwave freeze-drying of starch model foods [13], the surface temperature of the sample was consistently higher than the central temperature throughout the drying process. This is because, after the sublimation of ice crystals on the surface of food, the tissue continues to absorb microwave energy and keeps heating up, while sublimation continues at the center, maintaining a constant temperature.

Setting the microwave power output too high can cause ice crystals to melt or lead to corona discharge, resulting in a temperature rise. Solid ice absorbs excessive microwave energy and melts into liquid water. In a 2450 MHz microwave field, the dielectric loss factor of liquid water is 12.48, which is significantly higher than that of solid water (ice) at 0.0029. Therefore, liquid water converts microwave energy into heat much more efficiently than solid ice, causing microwave energy to be more readily absorbed in the melting ice regions, leading to localized high temperatures.

A comparison of results reported in the literature showed them to be consistent with the trend of this experiment. Increasing the microwave power significantly shortens the drying time. However, excessive temperatures can undermine the structural support provided by ice in the frozen state, causing tissue collapse, leading to surface burning or charring of a sample and the degradation of heat-sensitive active ingredients. Thus, increasing the power density can accelerate the initial heating rate and shorten the phase transition time from ice to gas, promoting the sublimation of ice crystals in krill and significantly enhancing drying efficiency. However, it is crucial to control the microwave power within an appropriate range to avoid the adverse effects of excessive microwave energy, such as ice crystal melting and corona discharge.

In this experiment, the microwave frequency was 2450 MHz, and the chamber pressure was maintained at 0.1–0.2 mmHg (13.33–26.66 Pa). According to the research by Gould and Kenyon [24], 2450 MHz has a higher dielectric breakdown critical discharge power than 915 MHz. The pressure range for the minimum power is around 1 torr (approximately 133.32 Pa) at 2450 MHz. The pressure range used in this experiment fell within the higher critical power region, which helped prevent the occurrence of corona discharge. The experimental process also revealed that as the microwave power density increased, the improvement in drying efficiency gradually slowed down; when the power density exceeded 2 W/g, ice melting tended to occur. Therefore, the microwave power density was kept below 2 W/g throughout the drying process to prevent the ice crystals in the samples from melting. Furthermore, when the samples reached the drying endpoint (DMC < 0.1), the microwave power source was turned off to prevent corona discharge and protect the product.

The power density must be carefully controlled to prevent the excessive acceleration of gas molecules, which leads to ionization. Lower power densities are typically more favorable, as they reduce the risk of plasma formation while still providing sufficient energy for effective drying. Moreover, maintaining an appropriate vacuum pressure is essential. High vacuum levels reduce the number of gas molecules present, thus minimizing the likelihood of ionization. However, the vacuum should not be so high that it impedes the drying process. Therefore, it is important to balance these parameters to optimize the drying process while avoiding the complications associated with plasma discharge [15].

Table 1 shows the effect of microwave power on the moisture loss kinetics during microwave freeze-drying of 50 g krill. The results showed that the krill weight (y) and the drying time (x) had a linear relationship, indicating a zero-order reaction. The relationships between microwave power levels of 50 W, 70 W, and 90 W and corresponding power densities of 1, 1.4, and 1.8 W/g were described by equations $y = -0.4278x + 50.507$, $y = -0.6200x + 50.886$, and $y = -0.9287x + 50.960$, respectively, with R^2 values of 0.995, 0.995, and 0.994. Based on these equations, the estimates drying times required to reach the drying endpoint (DMC < 0.1) at different microwave power levels were 76, 58, and 44 min, respectively. However, the equation for freeze-drying without microwave power was $y = -0.0421x + 47.475$, with an R^2 of 0.979, and the predicted drying time was 868 min. This is because typical freeze-drying relies on external air convection and conduction for heat transfer, particularly in the later phase, when heat transfer resistance increases, leading to a falling drying rate stage. Therefore, the use of microwave assistance can decrease the freeze-drying time compared to traditional methods by a factor of 10 to 20.

Table 1. Effect of microwave power on the drying rate of 50 g krill by microwave freeze-drying

Power (W)	Linear Regression Equation	R^2	Drying Rate (g/h)	* Drying Time (min)
50	$y = -0.4728x + 50.507$	0.995	28.37	76
70	$y = -0.6200x + 50.886$	0.995	37.20	57
90	$y= -0.9287x + 50.960$	0.994	55.72	40
FD	$y = -0.0421x + 47.475$	0.979	2.53	868

Where y is the weight (g) and x is the drying time (min). * Estimate of the drying time (min) required for DMC to drop to 0.1.

Microwave freeze-drying was performed on 50 g of krill, with the microwave power gradually increasing, resulting in a microwave power density ranging from 0.5 to 2 W/g. The predicted drying time and rate were plotted for analysis (Figure 4). The distribution of the power density (x, (W/g)) and the drying time (y, (min)) approached a hyperbola, with the relationship represented as $y= 82.71x^{-1.063}$, $R^2 = 0.9869$. This result showed that when the power density was increased to a particular level (2 W/g), the effect of microwave power on drying time gradually decreased. This showed that the power density may be the critical load power for this weight and that exceeding it leads to inefficient microwave energy consumption, potentially causing ice crystal melting or corona discharge. When the microwave power density exceeded 2 W/g, the samples exhibited evidence of ice melting. The power density should be below 2 W/g for optimal microwave energy usage.

Furthermore, the drying rate increased with the rise in microwave power, from 0.0118 min^{-1} at 1 W/g to 0.0225 min^{-1} at 1.8 W/g, nearly doubling the drying efficiency. A comparison was made between the microwave power density and the drying rate (Figure 4). The results showed a linear relationship between microwave power density (x, (W/g)) and drying rate (y, (min^{-1})), represented as $y = 0.0128x - 0.0018$, $R^2 = 0.9872$. Using these two sets of equations, the drying rate and drying time at various microwave output powers may be calculated for a given krill sample load weight.

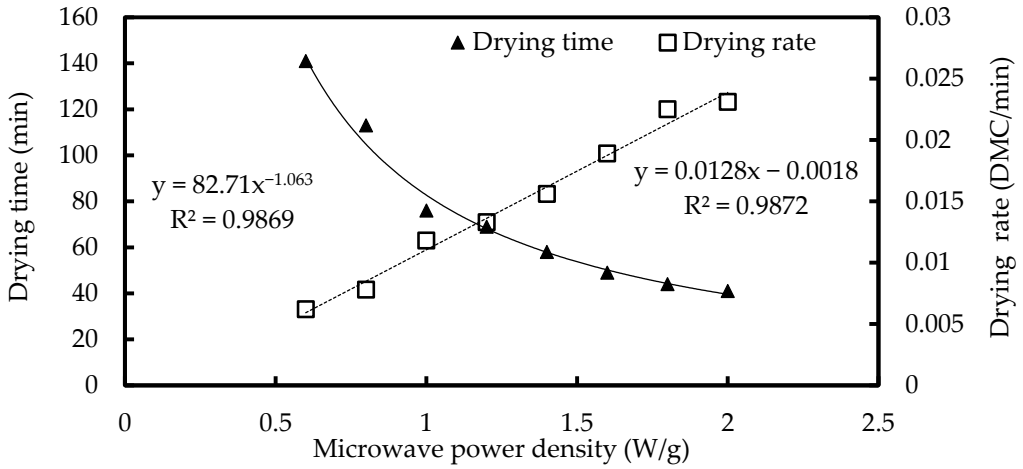

Figure 4. The relationship between microwave power vs. drying time and drying rate during microwave freeze-drying of 50 g krill.

The drying time estimated using the formula can be used to calculate the energy loss of each component during the microwave freeze-drying of krill, as shown in Figure 5. The energy-consuming components include the compressor, the vacuum pump, and the microwave oven. As power density increased, drying time and energy loss decreased. The compressor and vacuum pump accounted for most of the overall energy loss (approximately 85–95%), while the microwave oven accounted for only 4–13%. Therefore, reducing the operation time of the compressor and the vacuum pump will significantly reduce the energy loss throughout the freeze-drying process. Experiments also showed that increasing the power density from 1.0 W/g to 1.8 W/g reduced the total energy loss from 3746 kJ to 1995 kJ. This significant reduction in energy loss was due to the decreased operation time of the compressor and the vacuum pump. However, due to the long drying period and poor heat transfer efficiency in traditional freeze-drying, the constant operation of the compressor and the vacuum pump results in an energy loss of about 6000 kJ. With the same weight (50 g), adjusting the microwave power density resulted in a nearly identical energy loss for the microwave oven during the freeze-drying process.

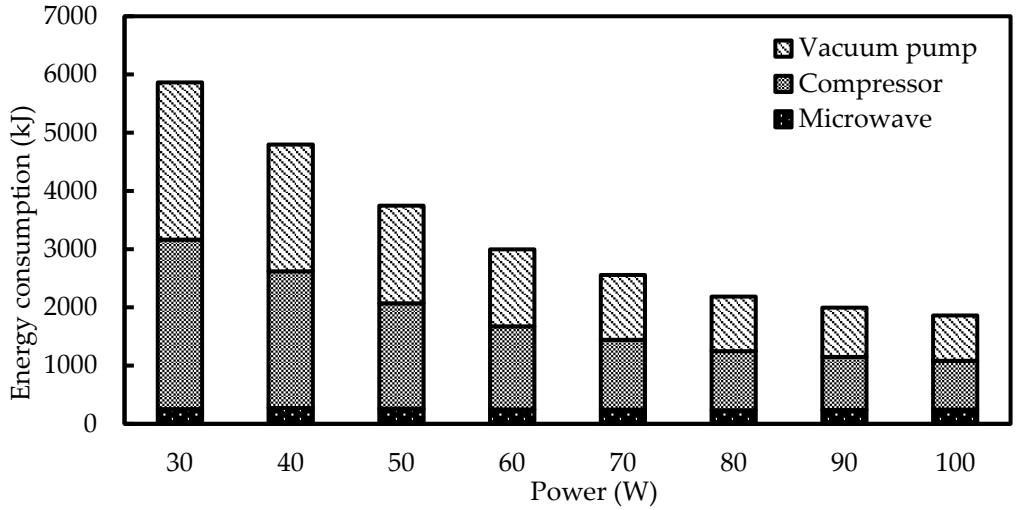

Figure 5. Effect of microwave power on energy consumption during microwave freeze-drying of 50 g krill.

Although increasing power increases energy consumption, it also reduces drying time, accelerating the freeze-drying process. This is because, given the same weight, the sample moisture content is nearly constant (90.31%), implying that the sensible heat to raise the temperature of the sample and the latent heat for ice sublimation will be the

same during the dehydration process. Using a microwave as a heat source, increasing its output power just accelerates moisture removal without affecting the total energy demand. Therefore, making power adjustments has little effect on the energy consumption of a microwave oven.

3.2. Effect of Loading on Drying Kinetics and Energy Consumption during Microwave Freeze-Drying of Krill

Figure 6 shows the effect of sample weight on the microwave freeze-drying trend of krill at a constant microwave power (50 W). The results showed that as the weight of krill increased (50–200 g), the time required to reach the drying endpoint also increased (83–250 min). This result corresponds with the findings of Wang et al. [29] for microwave freeze-drying of instant vegetable soup (150–600 g). Under a fixed microwave power (450 W), when the weight of the vegetable soup increased, the drying time also extended from 3.5 h to 6.5 h. This is because, as the sample load weight increased while the microwave power remained constant, the microwave absorption power per unit weight decreased from 1 W/g to 0.25 W/g.

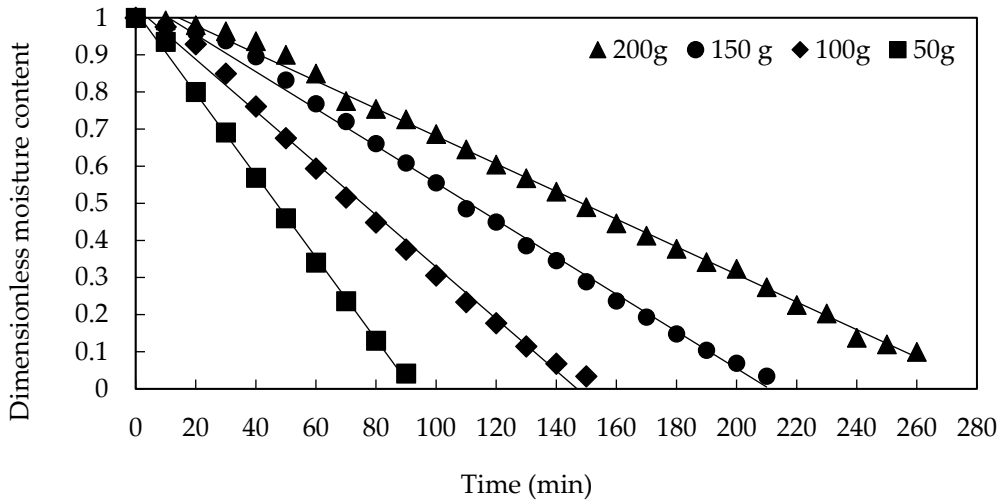

Figure 6. Effect of sample weight on drying curve of krill by microwave freeze-drying.

Table 2 shows the effect of sample weight on moisture loss kinetics during microwave freeze-drying of krill at a constant microwave power (50 W). During the drying process, the weight (y, (g)) followed a linear relationship with the drying time (t, (min)). The linear regression equations for 50 g, 100 g, 150 g, and 200 g were $y = -0.4256x + 50.684$, $y = -0.5508x + 102.92$, $y = -0.55765x + 156.96$, and $y = -0.55179x + 208.27$, with R^2 values of 0.998, 0.996, 0.995, and 0.997, respectively. Using these equations, the total drying time required to reach the drying endpoint (DMC < 0.1) for different weights of krill (50, 100, 150, and 200 g) at a microwave power of 50 W was determined to be 83, 133, 191, and 264 min, respectively.

Table 2. Effect of sample weight on the drying kinetics of krill by 50 W microwave freeze-drying

Weight (g)	Linear Regression Equation	R^2	Drying Rate (g/hr)	* Drying Time (min)
50	$y = -0.4256x + 50.684$	0.998	25.54	83
100	$y = -0.5508x + 102.92$	0.996	32.32	133
150	$y = -0.5765x + 156.96$	0.995	34.31	191
200	$y = -0.5179x + 208.27$	0.997	34.10	264

Where y is weight (g) and x is time (min). * Estimate of the drying time (min) required for DMC to drop to 0.1.

The drying time increased with the increase in weight for two reasons. First, the total amount of water in the sample increased with the weight; therefore, the same microwave power took longer to provide enough energy to remove the higher total water content. Second, as the weight increased, the microwave power density decreased, which reduced the drying rate. When the weight of the krill increased from 50 g to 200 g, the microwave power density decreased from 1 W/g to 0.25 W/g.

The microwave freeze-drying time predicted with the formula was used to calculate the energy consumed by krill of various weights at the drying endpoint (DMC < 0.1). Figure 7 shows that as the weight (50–200 g) increased, so did the drying time and the total energy loss (3575–11,356 kJ). The compressor and the vacuum pump were responsible for 48% and 45% of the overall energy consumption, respectively, while the energy consumption of the microwave oven accounted for only 7%.

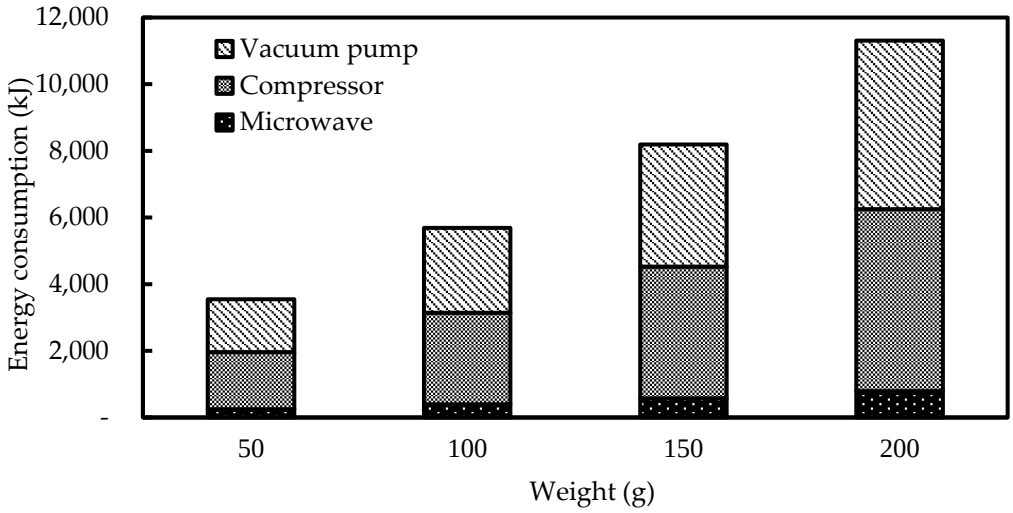

Figure 7. Effect of sample weight on energy consumption during 50 W microwave freeze-drying of krill.

It was shown that for the same microwave power (50 W), when the sample weight increased, the drying time increased linearly. This issue was also related to the moisture content in the sample. With the same moisture content (approximately 80% for frozen krill), increasing the krill sample weight proportionally increased the ice weight. Under continual microwave power, the greater ice weight requiring sublimation caused a longer drying time. The energy consumption of the microwave oven proportionally increased the drying time.

3.3. Effect of Different Drying Methods on the Quality of Krill

Figure 8 shows the drying curves for 50 g of krill using hot-air drying (HAD) at 55 °C, freeze-drying (FD), and microwave freeze-drying (MWFD) at 90 W. Table 3 presents the linear regression equations for the HAD, FD, and MWFD drying processes. According to the linear equations, the drying endpoints (DMC < 0.1) for the three processes were achieved at 18, 16, and 0.67 h, respectively. In comparison, the drying times for HAD and FD were approximately 26 times and 24 times longer than those for MWFD, indicating that using microwaves as a heat source during the drying process can greatly improve drying efficiency and reduce drying time. Table 4 compares the energy consumption units and total energy loss for FD and MWFD when drying 50 g of krill to the endpoint (DMC < 0.1). The results showed that the total energy consumption for FD and 90 W MWFD was 34,651 kJ and 1995 kJ, respectively. Increasing microwave power reduced drying time, resulting in lower energy consumption for overall processing. This indicated that microwaves help lower the drying time of traditional freeze-drying by more than 20 times while saving nearly 90% of the energy consumption.

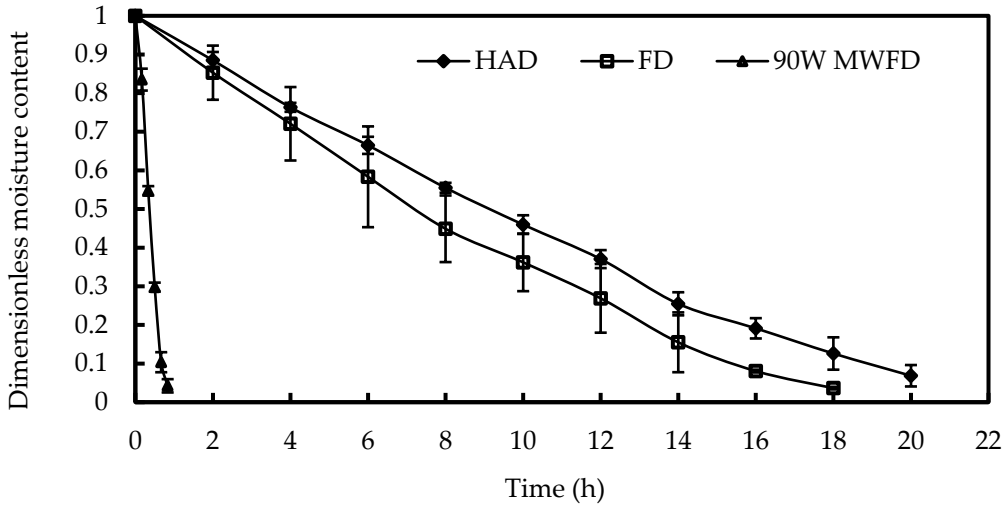

Figure 8. The drying curve during the drying process of 50 g krill by hot-air drying (HAD), freeze-drying (FD), and 90 W microwave drying (MWFD).

Table 3. Effect of different drying methods on the drying rate and drying time of krill

Drying Method	Linear Regression Equation	R^2	Drying Rate (DMC/h)	* Drying Time (h)
Hot-air drying (HAD)	y = −0.050x + 1	0.981	0.050	18
Freeze-drying (FD)	y = −0.056x + 1	0.996	0.056	16
Microwave freeze-drying (90 W MWFD)	y= −1.353x + 1	0.992	1.323	0.67

Where y is DMC and x is time (h). * Estimate of the drying time (h) required for DMC to drop to 0.1.

Table 4. The energy consumption during freeze-drying (FD) and microwave freeze-drying (MWFD) of 50 g krill

MWFD Power (W)	Condenser (kJ)	Vacuum Pump (kJ)	Microwave (kJ)	Total Energy Consumption (kJ)	* Energy Saving (%)
50	1,808	1,677	262	3,746	89.19
70	1,199	1,113	243	2,555	92.63
90	912	846	238	1995	94.24
FD	17,977	16,674	0	34,651	0.00

* $Energy\ saving(\%) = \frac{E_{FD}-E_{MWFD}}{E_{FD}} \times 100\%$.

Using the Lab values of FD as a reference point, the color differences (ΔE) between MWFD and HAD were calculated to be 7.32 and 19.04, respectively (Table 5). This indicated that HAD krill had a more distinct appearance than FD or MWFD krill (Figure 9). The appearance of the samples showed that the hot-air-dried krill underwent severe browning and shrinkage, which was presumed to be due to excessive autolysis and browning caused by extended high-temperature processing during drying. Although the MWFD krill showed slight shrinkage, the overall impact on their appearance was less obvious.

Table 6 shows the content of active components in krill following the three different drying methods. Overall, the active components in dried krill significantly decreased, most likely because drying enhanced the adhesion of active components to the food matrix, making them harder to extract, or because the oxidation process during drying lowered the oxygen-sensitive components. However, drying krill is a necessary procedure before transportation, storage, and later processing applications. Although the active components in FD krill are slightly higher than those in MWFD, the differences are small, with total

carotenoids, phospholipids, and DHA content decreasing by 0.34%, 7.74%, and 11.87%, respectively. Based on the information presented above, microwave freeze-drying is a highly promising emerging alternative technology in krill processing. Freeze-dried krill had the best quality of the three drying methods, followed by microwave freeze-dried krill, with the lowest quality exhibited by hot-air-dried krill. Microwaves can greatly improve the efficiency of the krill freeze-drying process while reducing energy consumption, making it a more economical and environmentally friendly drying process improvement technology.

Table 5. Effect of drying methods on the color of krill

Drying Method	L*	a*	b*	ΔE
FD	45.25 ± 3.92 ^{a}	11.56 ± 0.76 ^{c}	31.23 ± 3.92 ^{a}	0
MWFD	40.04 ± 3.11 ^{b}	15.83 ± 0.98 ^{a}	28.38 ± 1.99 ^{b}	7.32
HAD	30.91 ± 0.46 ^{c}	13.21 ± 0.31 ^{b}	18.82 ± 0.48 ^{c}	19.04

$\Delta E = \sqrt{(L_2^* - L_1^*)^2 + (a_2^* - a_1^*)^2 + (b_2^* - b_1^*)^2}$. Data are expressed as means ± SDs (n = 3). Means with different superscript letters in the same column are significantly different ($p < 0.05$).

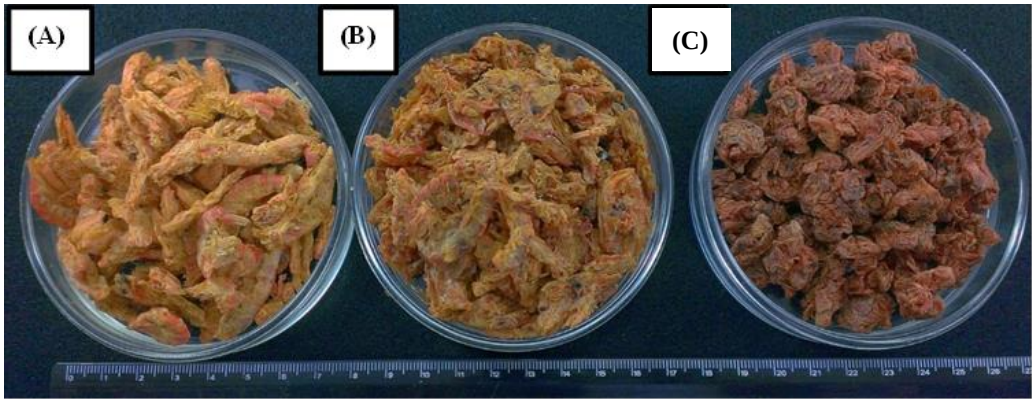

Figure 9. Photos of krill dried by FD (**A**), MWFD (**B**), and HAD (**C**).

Table 6. Effect of drying methods on total carotenoids, phospholipid, and DHA content of krill

Samples	Total Carotenoids (μg/g d. b.)	Phospholipid (%)	DHA (mg/g d. b.)
Fresh	218.41 ± 45.63 ^{a}	16.30 ± 0.34 ^{a}	11.67 ± 0.22 ^{a}
FD	137.29 ± 39.28 ^{b}	5.17 ± 0.023 ^{c}	8.59 ± 0.56 ^{b}
90W MWFD	136.82 ± 17.11 ^{b}	4.77 ± 0.14 ^{d}	7.57 ± 0.10 ^{c}
HAD	120.54 ± 24.52 ^{b}	5.60 ± 0.06 ^{b}	-

Data are expressed as means ± SDs (n = 3). Means with different superscript letters in the same column are significantly different ($p < 0.05$).

4. Conclusions

The microwave freeze-drying process can be scaled up by adjusting the krill weight (50~200 g) and microwave power settings (30~100 W). The drying kinetics model was established through the zero-order reaction. Therefore, this study found through experiments that (1) when the sample weight is fixed and the microwave power changes, the total energy required to complete drying remains constant, and that (2) as the sample weight increases, the total energy consumption needed for microwave drying also rises. When comparing the three drying methods, microwave-assisted heating has the potential to significantly reduce drying time while maintaining high product quality. Compared to traditional freeze-drying, microwave freeze-drying can reduce drying time by more than 20 times and save about 95% of energy consumption.

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