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(RESEARCH ARTICLE)

An Experimental Study on the Freeze-Drying Process for *Cordyceps militaris*

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Abstract

This study presents experimental results on the freeze-drying process of *Cordyceps militaris*. Key focus areas included the construction of the freezing curve, product temperature profile, drying curve, and drying rate curve. Freezing experiments showed that reducing the temperature from ambient (20 °C) to −41.2 °C required approximately 4200 s, and the average initial freezing point was determined to be −0.6 °C. Freeze-drying experiments conducted at three heating shelf temperatures (−5, 0, and 5 °C) demonstrated that raising the shelf temperature to 5 °C reduced the total drying time by approximately 16.7% compared to −5 °C. Analysis of the drying and drying rate curves revealed that the sublimation rate is strongly influenced by both the drying temperature and the mass transfer resistance of the dried layer. Furthermore, sensory evaluation showed that the dried *Cordyceps militaris* retained the color, shape, and volume of the fresh material. These findings provide a valuable basis for optimizing freeze-drying conditions to preserve both product quality and bioactive efficacy.

Keywords: Freezing curve; Drying rate curve**;** *Cordyceps militaris*; Primary Drying Stage; Freeze-drying

1. Introduction

Cordyceps militaris is a medicinal fungus widely recognized for its rich profile of bioactive compounds. It has been extensively applied in traditional medicine and the functional food industry due to its therapeutic properties [1–5]. After harvesting, drying is a critical step to enhance product stability, extend shelf life, and facilitate transportation and handling.

An effective drying process must preserve the valuable bioactive substances, retain the original shape and color, and maintain the physical integrity of the product with minimal alterations, ensuring a high rehydration capacity [6–8]. Among various techniques, freeze-drying is considered the most effective due to its superior ability to retain key bioactives – such as cordycepin, adenosine, polysaccharides, vitamins, and amino acids – while preserving the material's color and structural integrity [6, 8–11]. Despite these advantages, freeze-drying is a complex and energy-intensive process. The drying efficiency and final product quality are highly sensitive to operational parameters [6, 8, 9, 12, 13]. Inappropriate drying conditions can lead to extended drying times, increased energy consumption, and reduced product quality [13–17]. Among these parameters, drying temperature plays a pivotal role in determining the sublimation rate and maintaining structural stability, as it directly influences the glass transition behavior and the risk of structural collapse [13, 18–23].

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Nowak and Jakubczyk [13] provided a comprehensive review of the freeze-drying process, outlining its three main stages: freezing, primary drying, and secondary drying. Their work analyzed how key parameters—such as pressure, temperature, and freezing rate—affect the physical properties of food, including texture, color, and porosity. Similarly, Welti-Chanes et al. [14] highlighted emerging technological trends in lyophilization and its potential in preserving food quality, while proposing directions for future research. Silva-Espinoza et al. [16] evaluated the impact of freeze-drying conditions (pressure, shelf temperature) on the physicochemical properties and bioactive compounds of orange purée. Their results showed significant effects on brightness, color saturation, and retention of vitamin C and β-carotene. In another study, Hammami and René [17] used response surface methodology to optimize freeze-drying variables for strawberries, focusing on the effects of pressure and temperature on drying time and quality indicators such as color and rehydration capacity.

The freeze-drying regime is characterized by parameters including freezing temperature, heating shelf temperature, vapor condenser surface temperature, and chamber pressure. These parameters significantly affect both the efficiency and quality of the process. In the freezing stage, both the temperature and freezing rate influence ice crystal size, sublimation rate, and product porosity. Therefore, selecting an appropriate freezing protocol is essential [13, 14]. According to Tse-Chao Hua [24], suitable freezing temperatures for food products range from −43 °C to −14 °C, depending on the material. Many studies recommend freezing to −40 °C to ensure complete moisture solidification [25, 26]. Shelf temperature during primary drying is also critical. According to Hua [24], most food freeze-drying studies maintain shelf temperatures below 70 °C, typically between −30 °C and 20 °C, to support sublimation without exceeding the material's collapse temperature. In addition, the chamber pressure directly affects the drying rate and product quality. Lower pressure increases the driving force for mass transfer but requires more advanced vacuum systems, leading to higher costs. Typical chamber pressures for food freeze-drying range from 4 Pa to 130 Pa [13, 27]. The remaining parameter, the condenser surface temperature, also contributes to the sublimation process. Lower condenser temperatures improve vapor capture but require more powerful cooling systems. Studies often use condenser temperatures from −40 °C to −30 °C [13, 24].

Although extensive research exists on freeze-drying of pharmaceutical and food materials, studies focusing specifically on *Cordyceps militaris* remain limited. Notably, Xiao-Fei Wu et al. [10, 11] applied freeze-drying to *Cordyceps militaris* under the following conditions: freezing at −40 °C, maximum shelf temperature of 50 °C, chamber pressure of 80 Pa, and condenser temperature of −40 °C. However, the study has not yet proposed an optimal drying regime. Therefore, optimizing the drying temperature remains essential to achieving a balance between energy efficiency and product quality. Drying time reflects process performance and cost-effectiveness, while product quality can be evaluated through indicators such as color retention, water activity, rehydration capacity, shrinkage, and nutrient preservation.

The freeze-drying process generally consists of three stages: (1) the freezing stage, where water is converted to ice under atmospheric pressure; (2) the primary drying stage (sublimation), in which ice is removed under reduced pressure; and (3) the secondary drying stage (desorption), which eliminates bound moisture to achieve the desired residual water content. Among these, the primary drying stage has the most significant impact on process cost and product quality [13]. Monitoring the temperature and humidity of the material during drying is crucial for process control and product quality. For instance, Gaidhani et al. [28] emphasized the influence of drying regimes on product quality, while Jubilant Hollister Stier [29] reviewed freeze-drying in pharmaceutical applications, highlighting the parameters that affect efficiency and stability. Similarly, regulatory bodies such as the FDA have stressed the need to control key parameters to ensure process consistency [30]. Accurate temperature measurement is also essential. Pentronic [31, 32] reported that insufficient immersion depth of probes can cause significant errors due to stem conduction. An immersion depth of at least 10–15 times the probe diameter is recommended to minimize this effect [31–35]. Moreover, measuring residual moisture after each drying stage is highly valuable for evaluating process efficiency and ensuring product quality [13]. However, moisture measurement during freeze-drying is technically challenging. Under vacuum conditions, mass sensors are affected by factors such as temperature gradients, pressure fluctuations, vibrations, sample geometry, and vapor sorption phenomena, all of which may compromise accuracy—unless advanced, high-cost equipment is used [13, 36, 37]. To overcome these limitations, a practical approach widely used in freeze-drying research involves dividing the batch into subsamples and measuring residual moisture at selected time intervals. This method allows the construction of drying curves that reflect moisture loss over time [38, 39].

In this study, experimental investigations were conducted to evaluate the effects of drying temperature on drying kinetics and the final quality of *Cordyceps militaris* during the primary drying stage. Based on the results, a suitable freeze-drying protocol is proposed to ensure both energy efficiency and quality retention.

2. Materials and Research methods

2.1. Research material

The *Cordyceps militaris* samples used in this study were artificially cultivated at 4/4/A2 Vu Ngoc Nha Street, Vinh Ngoc Ward, Nha Trang City, Khanh Hoa Province, Vietnam. The harvested fruiting bodies displayed a characteristic orange hue, with lengths ranging from 30 to 80 mm and diameters between 3 and 6 mm. Following harvest, the samples were preserved and transported to the laboratory at Nha Trang University. Upon arrival, the fruiting bodies were carefully detached from the cultivation substrate to prepare for subsequent experimental procedures.

Figure 1 Fresh *Cordyceps militaris*

2.2. Experimental Research Equipment

2.2.1. Freezing Equipment

In this study, *Cordyceps militaris* was frozen using a specialized freezing unit located at the Thermal Engineering Laboratory of Nha Trang University, Vietnam. The equipment is capable of delivering a cooling capacity of 1 kW and features a chamber with a volume of 50 liters (dimensions: 430 mm × 280 mm × 420 mm). It can achieve temperatures as low as –50 °C, with an adjustable internal air velocity of up to 5 m/s [40].

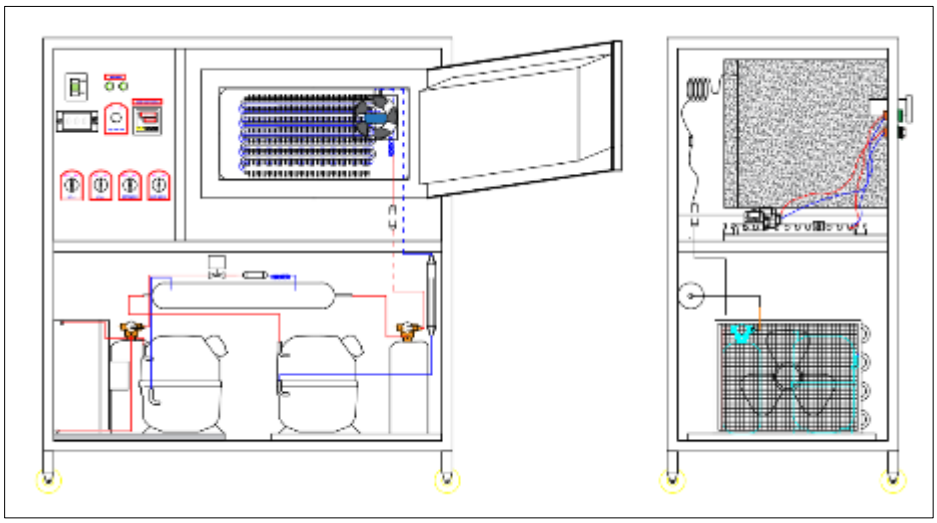

Figure 2 The freezing system [40]

2.2.2. Freeze-Drying system

The freeze-drying process in this study was carried out using a laboratory-scale unit located at the Thermal Engineering Laboratory of Nha Trang University. The system operates with a power consumption of 2 kW and is equipped with a cylindrical sublimation chamber (diameter: 310 mm, length: 500 mm) with a total volume of 38 liters [41]. Ethanol (86%) is used as the cooling medium for the refrigeration system. The heating system consists of a 500 W resistance element, while the condenser plate is capable of reaching temperatures as low as –35 °C. During operation, the chamber pressure is reduced and maintained at approximately 20 Pa to facilitate sublimation.

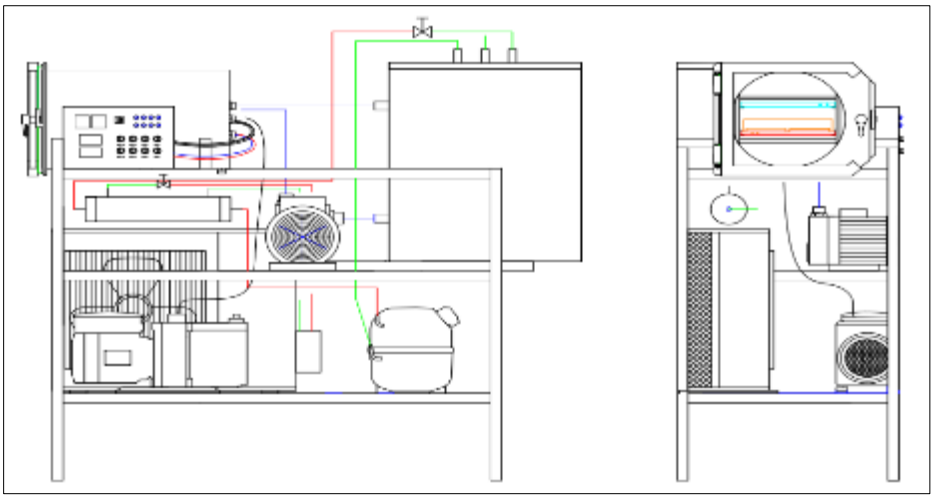

Figure 3 The freeze dryer system [41]

2.2.3. Measuring Devices Used in the Experimental Study

To support measurements and the determination of experimental parameters - including drying conditions, material properties, and product quality - various instruments were employed in this study. The technical specifications of these devices are summarized in Table 1.

Table 1 Technical specifications of measurement devices

Parameter	Device	Accuracy	Resolution	Estimated Uncertainty
Temperature (°C)	Extech TM500	±(0.4% + 1 °C)	0.1 °C	±1.2 °C
Pressure (Pa)	Testo 552	±1.3 Pa	1 micron	±2 Pa
Moisture (%)	Ohaus MB120	±0.1%	0.01%	±0.15%

2.3. Experimental Procedure for Freeze-Drying *Cordyceps militaris*

Freeze-drying is a complex process involving multiple stages, in which drying conditions significantly influence both efficiency and product quality. To minimize experimental errors, a standardized freeze-drying procedure for *Cordyceps militaris* was established, as illustrated in Figure 4.

2.4. Methods for Evaluating the Quality of *Cordyceps militaris*

2.4.1. Method for Determining the surface temperature of Cordyceps militaris

To measure the surface temperature of *Cordyceps militaris* fruiting bodies, a custom T-type thermocouple sensor was fabricated using copper and constantan wires, each with a diameter of 0.03 mm. A medical needle with a diameter of 0.51 mm was used to mount the sensor head.

During measurement, the needle tip with the attached sensor was inserted approximately 15 mm into the sample along its longitudinal axis. The sensor head was then tilted until it made contact with the surface of the fruiting body, ensuring close surface contact. With this setup - including a fruiting body diameter of approximately 5.2 mm, needle tip diameter of 0.51 mm, and a combined sensor wire diameter of 0.06 mm - measurement error of the surface temperature was minimized (see Figure 5) [32-35].

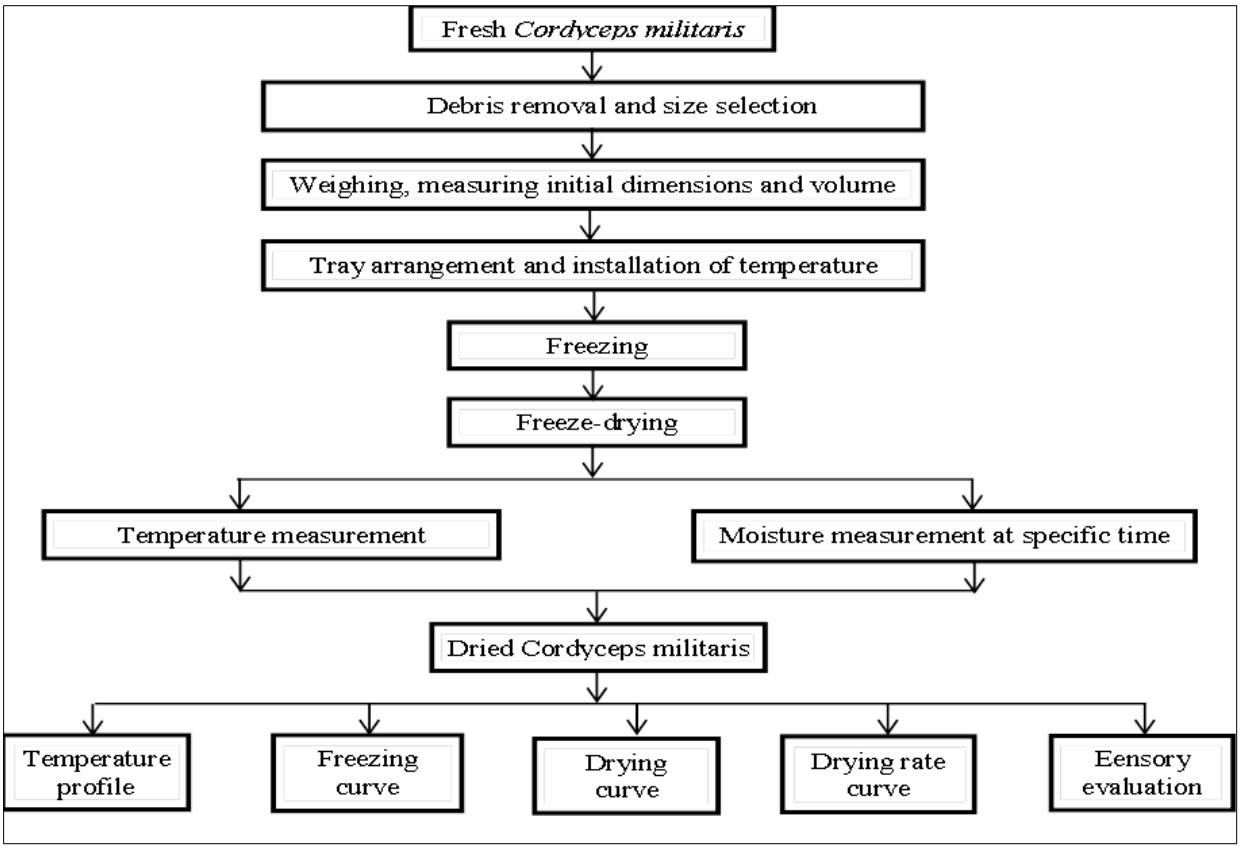

Figure 4 Schematic of the experimental procedure for freeze-drying *Cordyceps militaris*

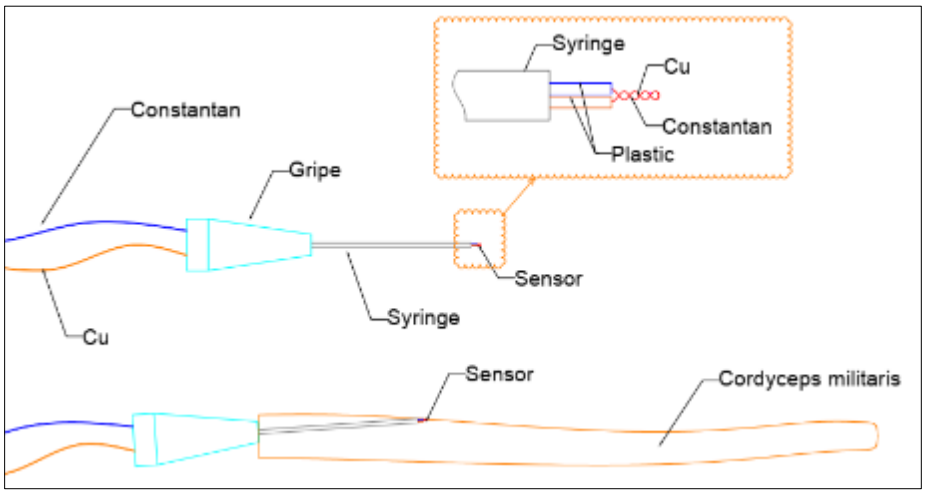

Figure 5 Structure of the Temperature Sensor

2.5. Method for Determining the Drying Curve and Drying Rate Curve

To determine the drying curve of *Cordyceps militaris*, this study employed an iterative approach. Freeze-drying experiments were conducted at multiple time intervals, after which the moisture content of the samples was analyzed to calculate the moisture ratio MR(τ) [38, 39]. By compiling and synthesizing the results obtained at these different time points, both the drying curve and the drying rate curve of *Cordyceps militaris* were constructed.

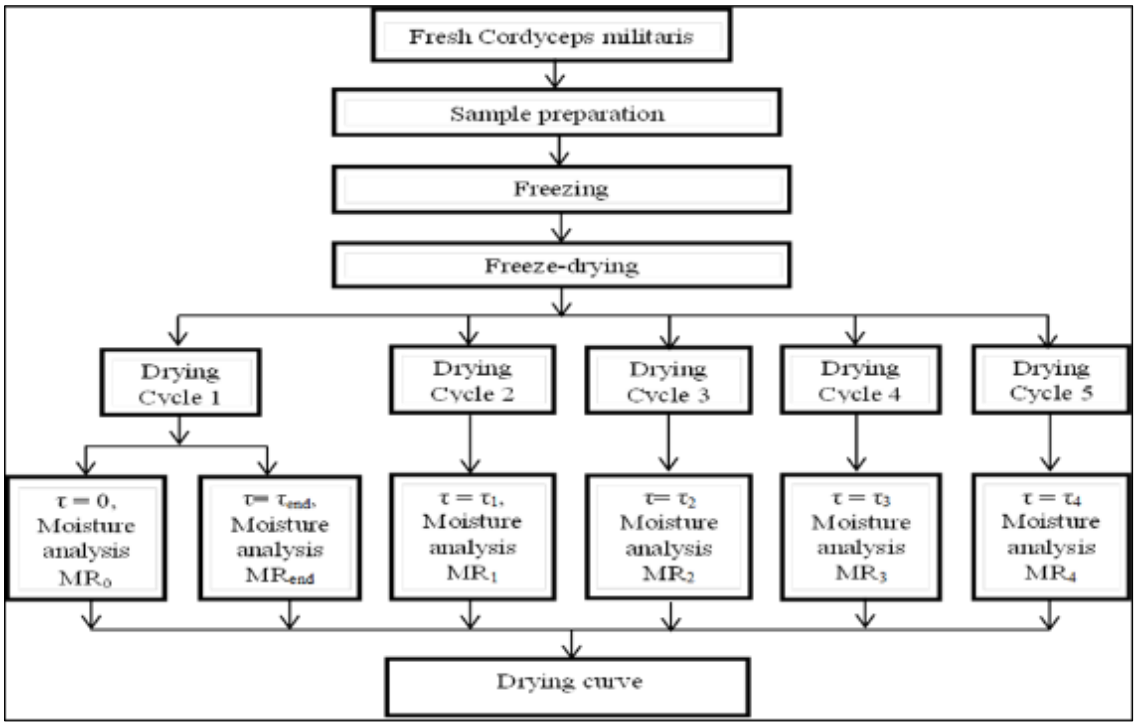

Figure 6 Flowchart of the process of constructing the drying curve of *Cordyceps militaris* during the freeze-drying process

3. Research results and Discussion

3.1. Experimental Results of Freezing *Cordyceps militaris*

As illustrated in the freeze-drying process diagram (Figure 2), fresh *Cordyceps militaris* samples were separated from their substrate, size-selected, and subjected to initial measurements of mass, dimensions, and color before being fitted with temperature sensors for freezing. The freezing process was conducted in a chamber maintained at −50 °C. After approximately 1.42 hours (4200 seconds), the internal temperature of the samples decreased to −40 ± 1 °C. This freezing temperature is in good agreement with prior studies as summarized in [10, 11, 13, 24]. The recorded temperature profile during freezing is presented in Figure 7.

From the experimental results of the freezing process, the freezing curve of *Cordyceps militaris* was established (as shown in Figure 7). Based on freezing curve analysis methods reported in the literature [42-48], the initial freezing point temperature of water in *Cordyceps militaris* was determined to be approximately −0.6 °C. This value falls within the typical freezing point range for water in vegetables and terrestrial animal muscle tissues, which varies from −1.05 °C to −0.5 °C, as reported by A.V. Luikov and D.R. Heldman [49]. Determining the initial freezing point is essential for estimating the proportion of freezable water in the material at that temperature, which subsequently serves as the basis for calculating thermophysical properties throughout the freeze-drying process [50].

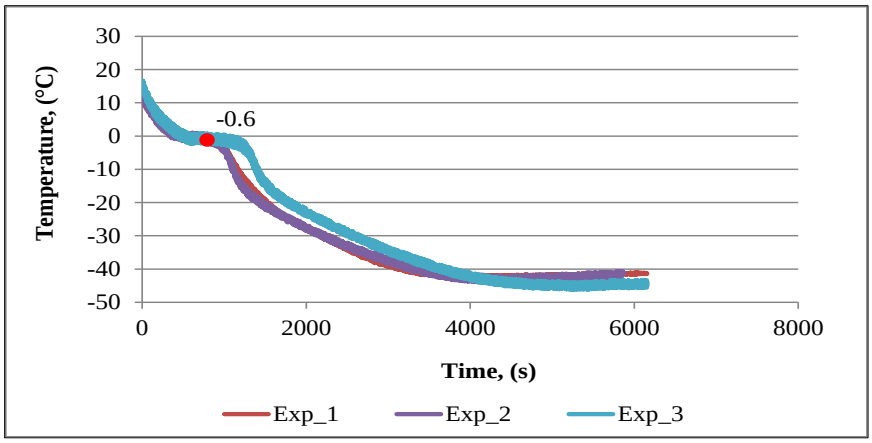

Figure 7 Freezing curve of *Cordyceps militaris* in 3 experiments

3.2. Experimental Results of Freeze-Drying *Cordyceps militaris*

Following the freeze-drying process illustrated in Figure 2, once *Cordyceps militaris* samples reached the target freezing temperature (–40 °C), they were transferred to the sublimation chamber of the freeze dryer to initiate the drying phase. In this chamber, heat was supplied via the heating shelves, while vacuum conditions facilitated the sublimation of ice from the frozen samples. The resulting vapor was condensed on the cold condenser plates, and non-condensable gases were evacuated from the chamber. This study focused on evaluating the influence of the heating shelf temperature on the drying rate and product quality. Three drying modes were investigated, corresponding to shelf temperatures of –5 °C, 0 °C, and 5 °C. During all drying experiments, the condenser plate temperature was maintained at –32 °C, and the sublimation chamber pressure was kept constant at 35 Pa. The recorded changes in temperature and moisture content of *Cordyceps militaris* during drying are presented in Figures 8 to 11.

Results showed that reducing the initial moisture content of 80.6 ± 1.4% (wet basis) to a final value of 5.42 ± 0.08% (equivalent to 0.057 ± 0.001 kg/kg dry basis) required approximately 18000 to 21600 seconds, depending on the drying mode. A clear correlation was observed between the shelf temperature and both the sample temperature and drying time: as the shelf temperature increased, the sample temperature rose and the drying duration decreased (as show on the Figure 8). Specifically, at a shelf temperature of 5 °C, the required drying time was approximately 18000 seconds (equivalent to 5 hours) - representing a 16.7% reduction compared to the case of –5 °C. For comparison, Xiao-Fei Wu et al. [10] reported drying times ranging from 6 to 9.3 hours when drying *Cordyceps militaris* at chamber temperatures of 40 °C to 70 °C under 80 Pa pressure, with the sublimation stage lasting from 3 to 5.83 hours. Despite the lower drying temperatures used in the present study, the overall drying time was shorter or comparable. This can be attributed to the lower chamber pressure applied, which enhances the sublimation rate.

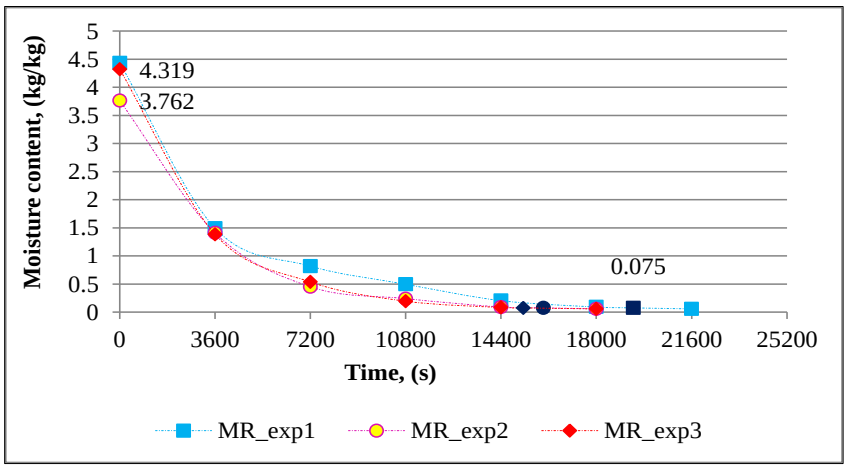

Figure 8 Moisture content profile of *Cordyceps militaris* during the freeze-drying process

As shown in Figure 9, a characteristic of the freeze-drying process is the absence of a constant-rate drying stage, which is typically observed in convection or radiation drying methods. The sublimation rate reaches its peak during the early

phase and then gradually decreases over time. This reduction is attributed to the increasing resistance to both mass and heat transfer as drying progresses. Initially, sublimation occurs at the surface layers of the *Cordyceps militaris* fiber and gradually extends toward the center. As a result, the dried layer thickens, leading to higher thermal and mass transfer resistance. Toward the end of the sublimation stage, the drying rate becomes negligible. To continue moisture removal, the remaining unfrozen moisture in the material must be removed, which requires increasing the product temperature while maintaining a low or reduced chamber pressure [13, 24, 28-30].

The temperature evolution of the material during the freeze-drying process is illustrated in Figure 10, which presents the surface temperature profile of *Cordyceps militaris*. Overall, the surface temperature increased throughout the drying process. In this study, the samples were arranged in a single layer on a drying tray mounted directly onto a heating shelf with electric resistance applied to its bottom, enabling the shelf temperature to represent the heating source. During drying, the heat supplied by the heating shelf is partially used for sublimating ice at the sublimation front, partially stored as sensible heat in the dry zone (raising its temperature), and partially lost to the environment, mainly through radiation to the vapor condenser. Once a dry layer forms on the surface - devoid of ice - the supplied heat can no longer be used for phase change and instead raises the surface temperature.

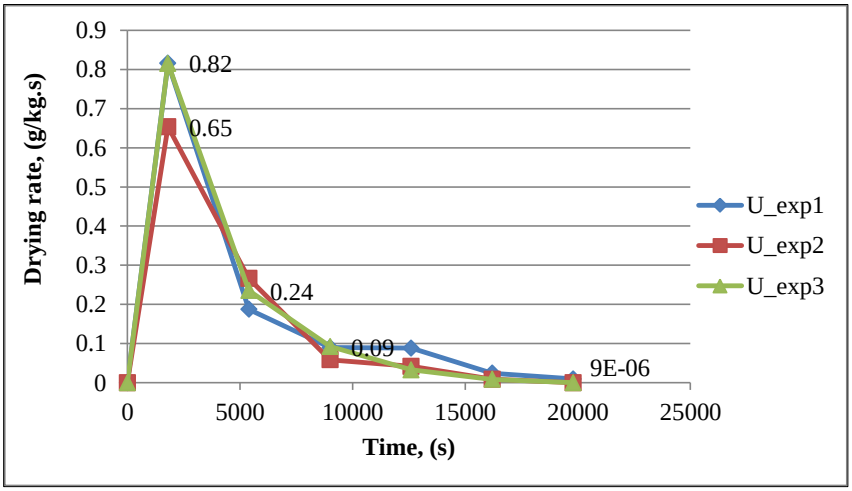

Figure 9 Drying rate curve of *Cordyceps militaris* in 3 experiments

As the dry layer thickens, its thermal resistance increases, impeding heat transfer to the frozen core and causing more heat to accumulate in the dry region. Toward the end of the drying process, when all ice has sublimated, the dry zone encompasses the entire volume of the sample. At this stage, most of the supplied heat is absorbed as sensible heat, leading to a rapid rise in the material's temperature, which approaches the heating shelf temperature. This temperature rise is a key indicator marking the end of the primary drying stage.

Figure 10 also confirms that the heating shelf temperature significantly influences the surface temperature of the samples. Specifically, at a shelf temperature of –5 °C, the maximum surface temperature reached only –8.9 °C, lower than the cases at 0°C and 5 °C. The highest surface temperature, –5.5 °C, was observed at a heating shelf temperature of 5 °C.

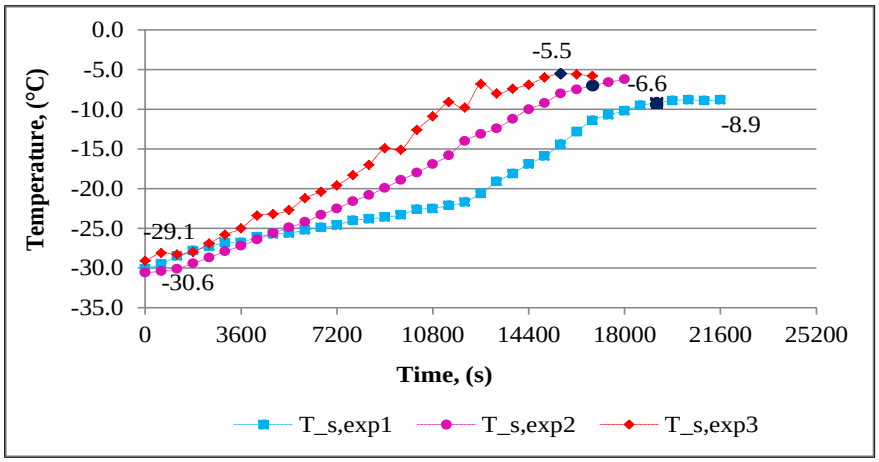

Figure 10 Temperature profile of *Cordyceps militaris* during the freeze-drying process

Figure 11 presents the summarized variations in temperature and moisture content of *Cordyceps militaris* during the freeze-drying process.

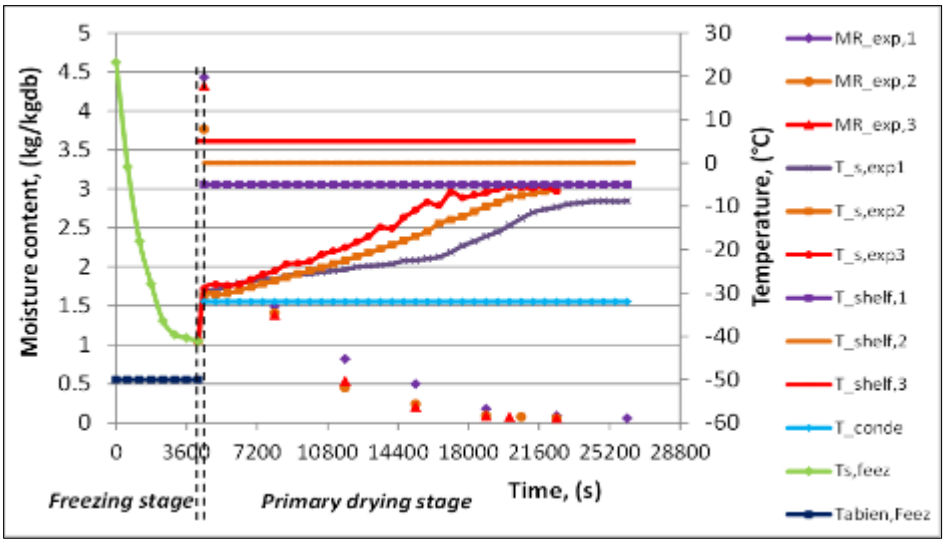

Figure 11 Drying rate curve of *Cordyceps militaris* in 3 experiments

Where: $T_{s,feez}$: Temperature of *Cordyceps militaris* during the freezing process; $T_{abien,feez}$: Temperature of the freezing environment; T_{conde}: Temperature of the vapor condensation plate; $T_{s,exp1}$, $T_{s,exp2}$, $T_{s,exp3}$: Temperature of *Cordyceps militaris* during freeze-drying in experiment mode 1, mode 2 and 3 respectively; $T_{shelf,1}$, $T_{shelf,2}$ and $T_{shelf,3}$: Temperature of the heating shelf in mode 1, mode 2 and 3 respectively; $M_{Rexp,1}$, $MR_{exp,2}$ and $MR_{exp,3}$: Experimental moisture ratio in mode 1, mode 2 and 3 respectively.

3.3. Preliminary Evaluation of the Quality of Dried *Cordyceps militaris*

The freeze-dried *Cordyceps militaris* product is shown in Figure 12. Compared to the fresh samples prior to drying, the freeze-dried Cordyceps retains a similar color. Furthermore, the shape and size of the dried specimens appear to remain nearly unchanged in comparison with the fresh ones. These results indicate that the proposed freeze-drying process and operating conditions do not cause significant alterations in the physical characteristics of *Cordyceps militaris*, including color, shape, and size. This demonstrates the suitability of the applied drying process and conditions for preserving the quality of *Cordyceps militaris* during freeze-drying.

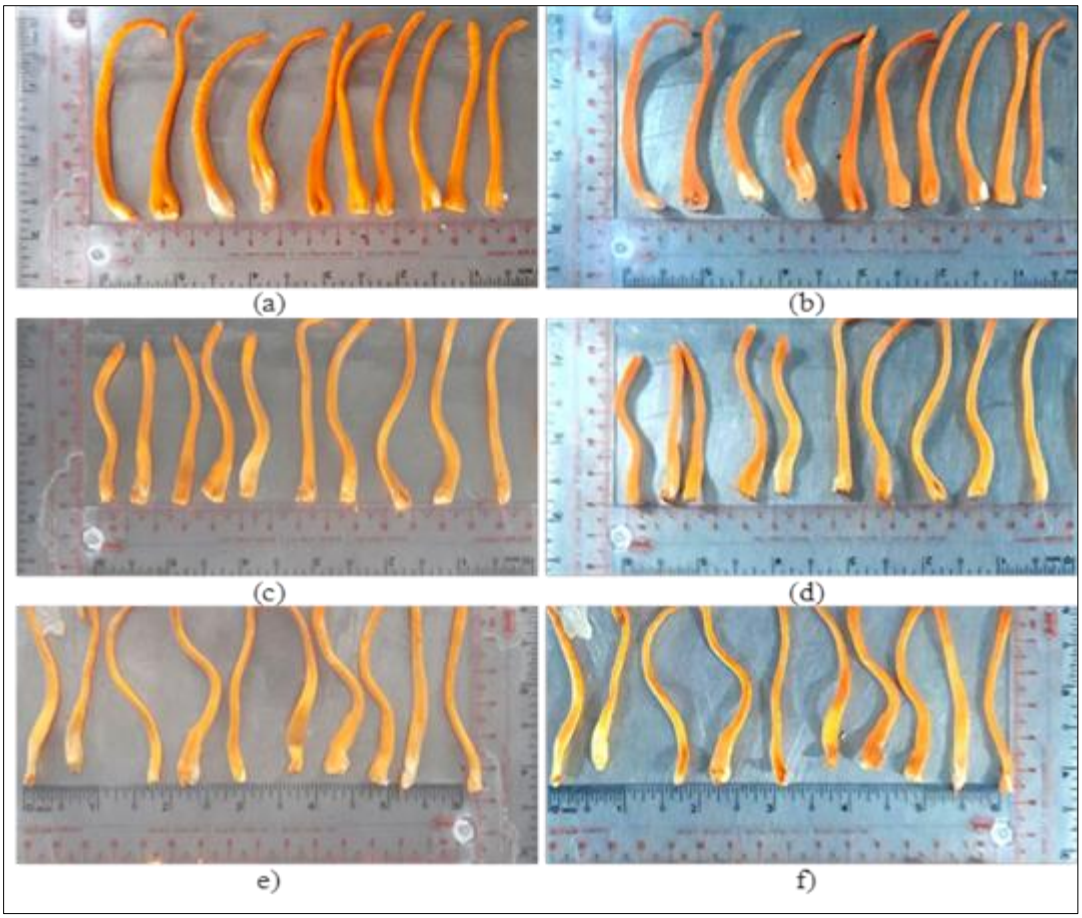

Figure 12 Images of fresh (a, c, e) and dried (b, d, f) *Cordyceps militaris* obtained at different drying modes

4. Conclusion

This study investigated the freeze-drying process of *Cordyceps militaris*, focusing on the changes in temperature, moisture content, and drying rate under varying heating conditions. Experimental results showed that reducing the temperature of *Cordyceps militaris* from ambient (20 °C) to −41.2 °C required approximately 4200 seconds (1.17 hours) under an ambient freezing temperature of −50 °C. Additionally, based on the freezing curve and Raoult's law, the initial freezing point of *Cordyceps militaris* was determined to be approximately −0.6 °C.

The results confirmed that the sublimation-based drying process is strongly influenced by the heating shelf temperature. Within the studied range of −5 °C to 5 °C, higher shelf temperatures led to shorter drying times but also caused an increase in the sample's temperature. Temperature elevation during freeze-drying must be carefully controlled, as excessive heat can result in undesirable changes such as discoloration, shrinkage, structural collapse, reduced porosity, and impaired rehydration capacity. These findings align well with established freeze-drying theory and previous experimental studies.

In addition, a comparison between fresh and freeze-dried *Cordyceps militaris* revealed that the dried samples retained their original color, shape, and size, indicating the appropriateness of the applied freeze-drying process and conditions. The outcomes of this study provide a scientific basis for further research aimed at optimizing the freeze-drying parameters for *Cordyceps militaris* to ensure both energy efficiency and product quality. It is necessary to further investigate the effects of other drying parameters, particularly chamber pressure, on the sublimation process. Moreover, evaluating product quality indicators—such as volume shrinkage, rehydration capacity, color change, water activity, energy consumption, and retention of bioactive compounds—will provide a foundation for determining the optimal sublimation drying conditions for *Cordyceps militaris*.

Compliance with ethical standards

Disclosure of conflict of interest

The authors declare that they have no conflicts of interest to disclose.

Statement of ethical approval

The authors declare that this study complies with all ethical standards applicable to scientific research.

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