# Atmospheric freeze drying of garlic slices based on freezing point depression

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**Abstract:** Garlic is difficult to dry due to browning reaction and degradation of heat-sensitive component. Freeze drying (FD) can keep good shape, color and nutrition of dried garlic, but it consumes long drying time and high energy. To shorten drying time and obtain high quality products, atmospheric freeze drying (AFD) was developed for drying garlic slices. The effects of different inlet air temperature programs on product quality and drying process were studied during AFD process. It was found that freeze point depression could lead to part of ice thawing resulting in product quality deterioration when the air temperature was not low enough. Based on this, a step-down-up temperature program was developed to reduce the drying time and provide good product quality.

**Keywords:** garlic, freeze drying, atmospheric freeze drying

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## 1 Introduction

Garlic (*Allium sativum*) has been cultivated for centuries all over the world due to its culinary and potential medical properties. Garlic is also cultivated widely in China and consumed as fresh, frozen and dried condiments. Garlic is dehydrated into different products such as powders, flakes and slices. Dehydrated garlic has a great commercial value and is used as a spice or standard ingredient in prepared foods and formulations. However, nearly 30% of the harvested garlic is wasted during storage due to respiration, microbial spoilage[1].

The potential medicinal properties of garlic are attributed to allicin and its degradation products[2]. Allicin does not exist in intact garlic, but is rapidly produced when raw garlic is crushed or chopped. After crushing, the allinase will contact with alliin and then convert alliin into allicin and pyruvic acid. Allicin is unstable in the hot place or organic solvents. It can degrade readily to a variety of degradation products, such as allylsulfides, vinyldithins and ajoenes. Therefore, allicin is greatly affected by drying methods and drying conditions[3].

Air drying (AD) is the most commonly used technique to dry garlic slices. Nevertheless, long drying time, high temperatures and high velocities of drying air flow are serious disadvantages for this method[4], which leads to degradation of biological components and undesirable changes in the quality of the products[5,6]. It is well known that freeze drying (FD) can provide products with excellent quality. Ratti et al.[7] reported that freeze dried garlic slices had the same allicin content as fresh garlic, however, this drying method is relatively expensive.

In fact, it was found that the diffusion of water vapor

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from the drying boundary through the dried shell was occurred by vapor pressure gradient, rather than by the absolute pressure on the system[8]. Hence, it is possible to freeze-dry at atmospheric pressure if the partial pressure of water vapor in the drying medium is kept low enough, which can provide a mass transfer driving force for water vapor transfer from the frozen sample. This drying method can be called atmospheric freeze drying (AFD). The use of heat-pump dryers (HPD) operated at atmospheric pressure and freeze-drying mode can fulfill such requirements. AFD could combine the advantages of both freeze drying (high product quality) and convective heat pump drying (low costs of the process)[9]. The main advantages of using heat pump technology are the potential energy-saving and the ability to control drying temperature and air humidity. Claussen et al.[8] reported that typical specific moisture extraction rate (SMER) values for atmospheric freeze drying with heat pumps were in the range of 1.5-4.6 kg of water per kW·h. However, the SMER of industrial vacuum freeze drying was in the range of 0.4 or even lower.

Despite the promises of low energy consumption and better product quality, certain problems still exist in the AFD process, which has limited its practical implementation. In AFD process, drying periods were very long, ice thawing and product shrinkage, affecting the drying rate and the diffusion of water, leads to poor quality of the end product[10]. As a result, the temperature of samples should be controlled to keep relatively high drying rate and good product quality without causing ice melting.

A wet material is constituted by solute and water that interacts and changes progressively during the drying process[8]. Because the freezing point depression resulted in moisture content decrease, the drying process has to be designed individually for each product. Therefore, it becomes vital to control AFD temperature according to freezing point of the material depression during drying.

There is no report about processing of garlic by AFD so far. In order to get good dried garlic whose quality is close to FD products with low energy consumption, a step-up and step-down temperature programs have been introduced to reduce drying time. The first step of the AFD preserve the product structure with minimal shrinkage, after removing the loosely bound water, the temperature was raised to 10-40°C to dry more bound water.

Therefore, the objective of this work was to study the effect of different air temperatures of AFD on the drying characteristics and the qualities of dried garlic in terms of the allicin content, bulk density and color. Based on the measured freezing point of material under different moisture contents, a suitable step-down-up air temperature program was developed for AFD processing of garlic slices.

## 2 Materials and methods

### 2.1 Materials

Fresh garlic bulbs were purchased from a wholesale market in Luoyang, China. Their moisture content was about 67% w.b. The garlic bulbs were manually cracked into cloves. The cloves were manually peeled and then sliced into 5 mm thickness with a slicing machine. A batch of 500 g of sliced garlic was prepared in each experimental. The time required for slicing was about 30 min. During these preparation steps, allicin was produced. Lawson and Wang[11] reported that allicin generation is completed within 6 s after cloves are crushed or chewed. The prepared samples were treated by AD immediately. Other samples which use FD and AFD treatments were frozen at -25°C for at least eight hours.

### 2.2 Atmospheric freeze dryer

The atmospheric freeze dryer is shown in Figure 1 and it has closed circuits for the drying air loop. To avoid heat losses, all the necessary parts of the dryer were properly insulated. The size of cylindrical drying chamber is 0.4 m in diameter and 0.8 m in length. In the drying circuit, the air is cooled in the evaporator and the condensed moisture is drained. Cold air was heated with both condenser and heating coils to secure inlet drying temperature from -15°C to 40°C. The relative humidity (RH) of the inlet air was approximately 30% and the air velocity was about 1.5 m/s. The product slices were placed on perforated shelves in the drying chamber, and a temperature probe was used to detect core temperature of the samples. The weight of samples was continually detected by an electronic-weighing system. Two temperature sensors were placed at inlet and outlet of the drying chamber respectively to monitor and continuous record the inlet-outlet temperature. All drying circuit is made of stainless steel for easy cleaning.

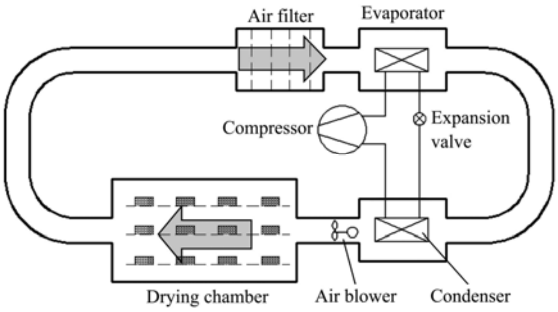



Figure 1 The schematic diagram of atmospheric freeze dryer

## 2.3 Drying experiments

1) FD process

The frozen materials (500 g) were dried by a freeze dryer (LG-0.2, Shenyang Areo Space Xinyang Quick Freezing Equipment Manufacture Co. Ltd., China). Heating shelf temperature and cold trap was set at 40°C and -40°C respectively. The pressure of drying chamber was set at 50 Pa during drying process. The samples were dehydrated until they reached the desired final moisture content (6% w.b.). FD-dehydrated slices were considered as comparative samples for assessment of the quality of AFD dehydrated samples.

2) AD process

The pretreated materials (500 g) 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 and 20% relative humidity. The temperature of the hot air was controlled and held at 40°C. The samples were dehydrated until they reached the required final moisture content (6% w.b.). AD-dehydrated slices were also considered as comparative samples for assessment of the quality of AFD dehydrated samples.

3) AFD process

The frozen samples were dried by different drying programs until the final moisture content reached 6% w.b. The material weight of each drying method was 500 g.

In order to investigate the effect of fixed inlet air temperature on the AFD process, two air temperature levels (-5°C, -10°C) were used respectively during AFD process.

Compared with fixed inlet air temperature, a changing air temperature loading program (0-8 h, -5°C; 8-12 h, -10°C; 12-20 h, -15°C; 20-26 h, 40°C) was performed.

## 2.5 Analysis of samples

1) Moisture Content

Moisture content was determined by the oven method[12].

2) Bulk Density

Spiked millet substitution method was used to detect the volume of dried samples. The granularity of spiked millet is between 0.9-1.1 mm[13]. The bulk density $\rho$ of the dried material is defined as:

$$\rho=m/V \tag{1}$$

where, $m$ is the mass of dried slices, and $V$ is total volume of dried slices.

3) Analysis of allicin content

The allicin content of garlic was determined according to the method of Lawson et al.[14] with some modifications. Three grams fresh sliced garlic sample or 1.5 g dried garlic powder was put into a centrifuge tube and mixed with 30 mL of distilled deionized water. Then, it was shaken vigorously for 1 min and stood for ten minutes at room temperature. The suspension was centrifuged (TGL-18C, Shanghai Anting Inc., Shanghai, China) at 10 000 r/min (4°C) for 5 min for fresh garlic and 30 min for dried garlic. The centrifuged sample was filtered through a 0.45 $\mu$m syringe filter, and was immediately analyzed by high performance liquid chromatography (HPLC). Allicin content was analyzed by Agilent 1260 HPLC system (Agilent Technologies, USA), coupled with a G1314B UV detector (monitored at 240 nm). The separation was achieved using a 5 $\mu$m C18 column (ZORBAX Extend-C18, Agilent Technologies, USA). The mobile phase used for elution of the allicin was methanol/water (50:50). The flow rate was 1 mL/min. The injection volume of all samples was 25 $\mu$L.

4) Freezing point

For freezing point depression measurements, product samples with different moisture content were put into an expanded polyester box with a temperature sensor placed in the middle of the product. A Hydra logger (2620A, Fluke Corporation, Phoenix, USA) was used to measure the temperature in the sample during freezing in a food deepfreeze fridge (Lab-scale 2, Hengli Refrigeration Equipment Co., Ltd., Zhengzhou, China) and the freezing point of the product was recorded.

5) Color evaluation

Color of fresh and dried garlic samples was evaluated by a colorimeter (Model WSC-S, Shanghai Shenguang Instrument and Meter Co., Ltd., Shanghai, China). The Hunter $L^*$, $a^*$, $b^*$ color scale was used, where $L^*$ represents lightness, $a^*$ represents redness (+) or greenness (-) and $b^*$ represents yellowness (+) or blueness (-). The hue angle (°h) can be calculated from $a^*$ and $b^*$; $°h = \tan^{-1}(b^*/a^*)$ when $a^*>0$ and $b^*>0$ or $°h = 180 + \tan^{-1}(b^*/a^*)$ when $a^*<0$ and $b^*>0$. The colorimeter was calibrated with a standard white plate before each measurement.

6) Microstructure analysis

A small specimen (about 5 mm×5 mm×2 mm) was cut from the dried sample and placed in a fixative containing 3% glutaraldehyde 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%, 90% and 100% ethanol for 15 min in each solution. After critical point drying, the dehydrated samples were sputtered immediately (CPD-030, BAL-TEC Company). Finally, the specimen fragments were mounted on aluminum stubs, coated with gold and photographed using a scanning electron microscope (SEM) (Quanta-200, FEI, Eindhoven, The Netherlands) with an accelerating voltage of 5 kV.

7) Energy consumption

The total energy consumption during drying process 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 Statistical analysis

All the tests were repeated 3 times. Analysis of variance (ANOVA) and the test of mean comparison according to Tukey's honest significant difference (HSD) were conducted at significance level 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 Freezing point depression and the drying profiles under different drying programs

As shown in Figure 2, the freezing point of garlic was decreased with the moisture content dropping, which implied that the freeze point of material would gradually depress during AFD procedure. It is well known that the more water of materials is removed by sublimation, the better product quality can be obtained because the drying operation may be hampered at any stage of drying due to phase change causing structural collapse or increasing stickiness[15]. Therefore, it becomes vital in controlling AFD process according to a reliable change law of the material freezing point depression during drying.

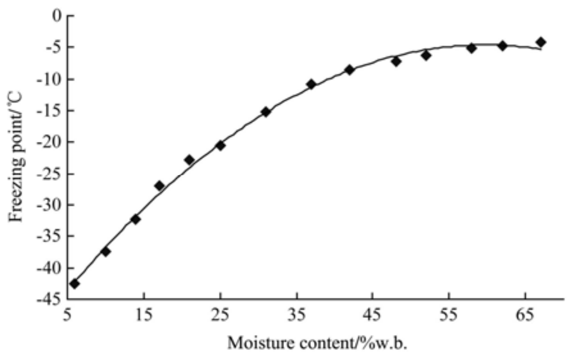



Figure 2 Effects of different moisture contents on the freezing point of garlic slices

Figure 2 shows that the freeze point of garlic slices had a slow decline tendency when its moisture content was above 50% w.b., and the freeze point value is about -5°C. While the moisture content of samples dropped about 40% w.b., the freeze point of garlic slices declined to about -10°C, suggesting a faster depression tendency. In addition, after the moisture content was below 30%w.b., the freeze point depressed faster and reduced to below -15°C. The possible reason was that most water was free water when the moisture content was relatively high, but with the drying proceeding, more and more free water was removed and the bound water gradually occupied more proportion, resulting in freezing point great depression.

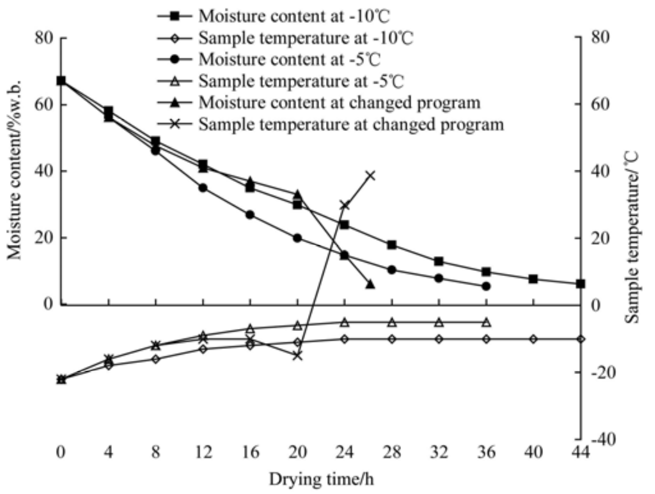



Figure 3 The profiles of product temperature and moisture content under different AFD temperature programs

It was reported that -5°C and -10°C inlet air temperatures were often used in a AFD program[8]. The effect of air temperature on the rate of AFD of cooked beef was investigated by Heldman and Hohner[16]. From Figure 3, it was found that air temperature fixed programs (-5°C, -10°C) all obtained a very low drying rate. According to results of freeze point depression mentioned above, a changing inlet air temperature program should be undertaken to operate as close to the maximum allowable temperature as possible. However, some research have reported that the porous structure of FD products mainly formed when the moisture content was on a relatively high level[17]. Therefore, AFD can be combined with medium temperature to increase drying rate and reduce drying time. This can be done when the moisture or solid fraction level is such that no significant solvent is mobile and no shrinkage occurs since it depletes product quality.

Figure 3 illustrated the profiles of garlic temperature and moisture content under different inlet air temperature programs. Both the two temperature fixed programs obtained long drying time (36-44 h). The difference in drying time was 8 h, comparing drying at -10 and -5°C down to final moisture content. This is because that higher air temperature resulted in a higher ice core temperature, which in turn caused a higher saturated vapor pressure at the ice–vapor interface. The higher vapor pressure at the interface represented an increase in the mass transfer potential and caused more rapid vapor transport across the porous zone. Additionally, due to high freezing point depression of garlic with water content below 40% w.b., some of the water inside the product was unfrozen or semifrozen and so it is easier to remove by drying. As is shown in Figure 3, after the moisture content was reduced below 30% w.b., the drying rate became slower, and this period took up almost half of the total drying time. This suggested that although the inlet temperature was low (-5°C or -10°C), the left water was also removed by evaporation other than sublimation after the moisture content was below 30% w.b. As a result, a higher air temperature was a more reasonable choice after most water had been removed in order to reduce the drying time of AFD process.

As also shown in Figure 3, the changing inlet air temperature based on the freeze point depression made a better drying efficiency, and the drying time was reduced to 26 h. From the sample temperature profiles, it could be found that when the moisture content was above 40% w.b., the material temperature was sustained under -10°C, and also kept a decreasing tendency with the moisture content dropping. Before the moisture content dropped to 30% w.b., the air temperature was increased to 40°C, and consequently the sample temperature gradually increased until the drying was finished. As a result, before the moisture content dropped to 30% w.b., the sample temperature was sustained below the freeze point. As mentioned above and compared with the fixed temperature programs, more water was removed by sublimation under the temperature changing program.

### 3.2 Effects of different temperature programs on product quality and energy consumption of drying

Table 1 shows the color of garlic with different drying process in terms of $L^*$, $a^*$ and $b^*$ values. The hue angle (°h) values, which were calculated from $a^*$ and $b^*$ values, can be used as an index of browning beside the $L^*$ value. A smaller hue angle value implies more change from yellow to red. It can be clearly seen from Table 1 that FD and AFD of temperature changing program provided garlic with the brightest color as manifested by the highest values of $L^*$ and °h. This implied that the air temperature program based on the freezing point depression could obtain the product quality same as that of FD treatment. On the other hand, the two temperature fixed programs of AFD got a relatively high browning effect. As mentioned above, both the two programs underwent more ice thawing than the temperature changing program during the drying, which worsened the browning effect. From the results of color measurement, it was also found that obvious browning would not take place although the air temperature was increased a higher level when most of water of the samples was removed. This agreed with some reports about improving drying temperature at the end of drying stage[17].

**Table 1 Effects of different drying conditions on the dried garlic color**

| Drying conditions | $L$*-value | $a$*-value | $B$*-value | Hue angle/°h |
|---|---|---|---|---|
| AD | 75.38±0.46$^d$ | 3.20±0.08$^a$ | 20.16±0.22$^a$ | 81.02±0.44$^d$ |
| FD | 88.26±0.58$^a$ | 0.58±0.12$^d$ | 10.42±0.26$^d$ | 86.86±0.32$^a$ |
| AFD (-5℃) | 79.46±0.32$^c$ | 2.23±0.12$^b$ | 16.25±0.32$^b$ | 82.23±0.36$^c$ |
| AFD (-10℃) | 80.46±0.35$^b$ | 1.02±0.08$^c$ | 14.36±0.82$^c$ | 85.98±0.28$^b$ |
| AFD (tem. changing prg.) | 87.16±0.22$^a$ | 0.60±0.12$^d$ | 10.88±0.48$^d$ | 86.89±0.42$^a$ |

Note: a, b, c, d, e indicated a significant difference ($p$<0.05). Drying conditions can be described as follows: AD (Air drying), FD (Freeze drying) and AFD (Atmospheric freeze drying). The same below.

Table 2 showed the allicin contents in different treated garlic. Both two temperature fixed programs had similar high level of allicin contents, and there was no significant difference between FD and temperature changing program in terms of allicin content. Even AD also could obtain an allicin content closed to FD. This indicated that drying temperature was the dominant factor to affect allicin content of garlic. This result also agreed with the fact that allicin was heat sensitive[7].

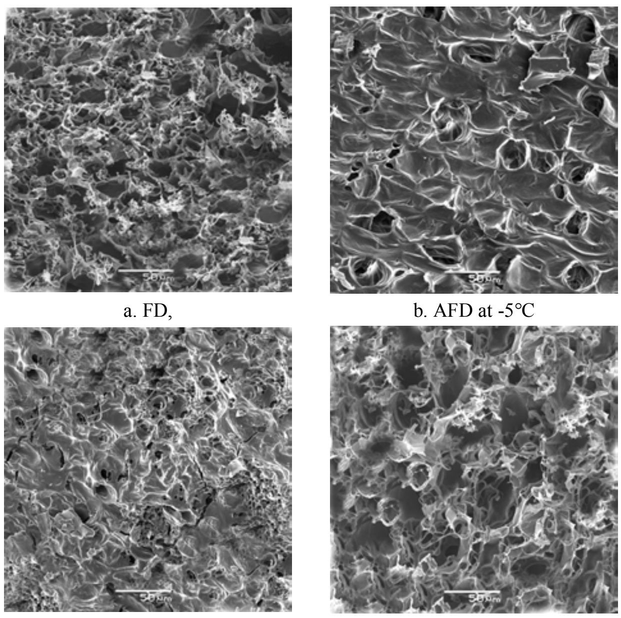
**Table 2 Effects of different drying conditions on bulk density, allicin content and energy consumption of dried garlic slices**

| Drying conditions | Bulk density /kg·m$^{-3}$ | Allicin content /$\mu$g·(g dry mass)$^{-1}$ | Energy consumption /kJ·(kg $H_2O$)$^{-1}$ |
|---|---|---|---|
| AD | 668±88$^a$ | 10954.6±120.2$^c$ | 17306.7±896.5$^e$ |
| FD | 478±76$^e$ | 11150.5±201.8$^b$ | 55842.6±1428.4$^a$ |
| AFD (-5℃) | 530±63$^b$ | 12022.6±80.2$^a$ | 36908.2±1657.2$^c$ |
| AFD (-10℃) | 506±55$^c$ | 12042.8±113.8$^a$ | 41342.8±936.6$^d$ |
| AFD (tem. changing prg.) | 495±48$^d$ | 11238.4±96.2$^b$ | 35088.2±1008.4$^b$ |

It is well known that sublimation drying could lead to porous structure resulting in a low bulk density of products. It can be seen that the bulk density of FD samples was the lowest, and followed by that of temperature changing program product. This suggested that using temperature changing program could also get good porous structure, which could be attributed to less ice thawing compared with other two temperature fixed programs. It is likely that the temperature fixed program treatment leads to more ice thawing, which removed by evaporation rather than sublimation, resulting in the porous structure destroyed. As a result, the temperature changing program based on the freezing point depression can help getting porous structure which is similar to FD treatment.

As shown in Table 2, AD performed the lowest energy consumption, and the energy consumption of FD was the highest. This result also agreed with many reports[18]. On the other hand, as a sublimation drying method, AFD yielded less energy consumption compared with FD. This is because that AFD is carried out at atmospheric condition and the vacuum pump and cold trap is removed. In addition, heat pump can make full use of latent and sensible heat from moisture air. Compared with temperature fixed program, the temperature changing program needed a less energy consumption. This can be attributed to a higher inlet air temperature, i.e. a higher evaporator temperature of heat pump.

Figure 4 shows micrographs of garlic slices obtained by different drying conditions.



a. FD, b. AFD at -5℃ c. AFD at -10℃ d. AFD at temp. changing prg.

Figure 4 Effects of different drying conditions on the microstructure of dried products

It was evident that FD sample exhibited the best porous structure. The samples undergoing temperature changing program also revealed a clear porous structure but there was some collapse parts. In addition, there was no clear porous structure in the samples under -5°C air temperature drying. Both two fixed temperature drying samples indicated cracking on the surface. This would happen during freeze drying because ice thawing raised internal gas pressure.

## 4 Conclusions

During the drying process, the freeze point of garlic depressed gradually. When the moisture content becomes low, the water in the samples is difficult to freeze. The inlet air temperature has a significant effect on product quality and drying efficiency during AFD process. Fixed air temperature (-5°C, -10°C) drying could not ensure good product quality and high drying rate. Based on the freeze point depression, a step-down-up air temperature program is potentially a better choice to obtain good product quality and low energy consumption.

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