Effects of pulsation ratio on center temperature and drying characteristics of pineapple slices with pulsed vacuum drying

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Abstract: This research explored the application of pulsed vacuum technology on the drying (PVD) of pineapple slices. Influences of drying temperature and pulsed vacuum ratio (PVR) on drying characteristics and pineapple quality (color, rehydration characteristics, microstructure, and texture) were analyzed. As expected, increasing the drying temperature resulted in a higher drying rate and effective moisture diffusivity. The optimal PVR of 5:5 was beneficial in accelerating the drying rate of pineapple slices and the corresponding effective moisture diffusion coefficient (8.9601×10^{-10}) was higher than other PVR conditions based on material center temperature. The material temperature increased during the normal pressure period and decreased rapidly when the pressure dropped to the vacuum condition, which indirectly reflected the moisture transfer that occurred during the vacuum holding period, while moisture diffusion happened during the atmospheric pressure holding period. The optimal pulsed vacuum drying process (PVR of 5:5) could expand air and water vapor and create a looser structure so as to obtain better rehydration performance (rehydration ratio (RR) was 5.43). High drying temperature led to the decrease of L^* value, the increase of ΔE value, and even the formation of surface scorch at 80℃. At the same drying temperature, the color quality depended on the drying time, and the color difference increased with the extension of the drying time. The chewiness and hardness of pineapple slices dried by PVD were significantly higher than those of fresh samples, which was conducive to the chewing taste.

Keywords: pulsed vacuum drying, pineapple slices, color, rehydration ratio, texture, center temperature
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1 Introduction

Pineapple (*Ananas comosus* L.) is an important commercial fruit in Thailand, Philippines, Indonesia, Brazil, and China where it is exported fresh and as a dry product[1]. It is popular with people for its golden color, rich aroma, and nutrients including fructose and vitamin C[2]. But it cannot be stored for a long time, and it was susceptible to the action of enzymes and microorganisms and decays under normal atmospheric temperature conditions. The black heart was one of the most common diseases of pineapple. Drying is one of the most frequently used methods for pineapple preservation as reduced moisture content can hinder the growth and reproduction of microorganisms, and minimize many moisture-mediated deteriorative reactions[3]. In the drying process, a series of physical and chemical reactions take place in the material, resulting in some changes in the organizational structure and nutritional composition of pineapple. Deng et al.[4] also reported the quality attributes of products in terms of color, rehydration capacity, and appearance were affected by pretreatment, drying technology as well processing conditions employed.

Open sun-drying due to its economical, simple, and easy to operate and other characteristics was the traditional dehydration method of pineapple. However, it was easily affected by the weather and environment and the long drying cycle (at least one week). Hot-air drying method was widely applied in industrialized production of agricultural materials due to the simple equipment, diversified form of energy utilization, mass production, and so on[5,6]. However, hot air drying was easy to cause surface hardening, hinder water migration, surface browning, and nutrient loss, resulting in poor quality of final drying products[7]. With the improvement in living standards, people's pursuit of high-quality dried fruit was increasing, and high-quality products were more easily favored by consumers[8]. Therefore, in order to improve the drying process to minimize color and nutrient loss, the traditional open sun-drying and hot air drying methods may be replaced by more efficient and advanced drying technologies.

A large number of scholars at home and abroad have done a lot of research on pineapple slice drying technology. Ramallo et al.[9] found that the hot air temperature had no significant effect on the color and mechanical properties of pineapple, and the rehydration ability and nutrient content were higher at 45℃. Ponkham et

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al.[10] used far-infrared radiation combined with hot-air drying technology to dry pineapple slices in a single layer and achieved good drying results. Results showed that the moisture diffusion coefficient of pineapple was significantly affected by far-infrared radiation intensity and hot air temperature. Through the experiment of pineapple puffing drying with variable temperature and pressure difference, the optimal process range was obtained as follows: puffing temperature 115℃-123℃, puffing pressure 0.04-0.08 MPa, and evacuation time 2-3 h. Zhu et al.[11] studied the microwave vacuum drying characteristics of pineapple and determined the optimal process parameters, that is, the microwave power at 1.4 W/g, the slice thickness at 4.06 mm, and the drying chamber pressure at 60.9 Pa. These findings can shorten drying time and improve the quality of pineapple slices compared with the traditional open sun drying method. However, how to protect the product's color was still a challenge as browning reactions often occur during the drying process due to oxygen. But thus far hot air drying was still the main drying method employed in the pineapple processing industry in producing dehydrated pineapple slices.

Pulsed vacuum drying (PVD) is a recently developed drying technique that can increase the drying rate and improve dried product quality compared to traditional hot air drying[12,13]. Figure 1 was a schematic representation of the PVD operation. During PVD processing, the pulsed vacuum environment created an oxygen-deficient environment, which can reduce adverse biochemical reactions, such as oxidation deterioration, and browning reactions, and thereby improve the quality attributes of dried products[14,15]. Additionally, pressure pulsation resulted in a tunneling effect to enlarge and interconnect the micropores in the products[16] and periodic pressure change can generate porous and fissured structures in the peel, which enhances the mass transfer through them[17]. With so many advantages, PVD has been employed in the drying of wolfberry[15], rhizoma dioscoreae[18], and seedless grapes [14].

Texture, color, and RR are frequently used to evaluate the quality of dried products. During the drying volume of the sample decreases, the surface area also simultaneously shrinks, which significantly influences the drying process and the product's quality attributes such as rehydration capability and texture due to moisture loss[19]. Therefore, the relationship between texture and drying conditions deserves more attention. Color is one of the most important quality attributes as the first quality judgment made by a consumer is by the product's color and it influences consumer's food choices, perceptions, and purchase behavior[20]. Color is also an indicator of thermal processing severity and it can be used to predict the corresponding quality deterioration caused by heat exposure[21]. Dried pineapple slices are usually used to make soup or stir fry vegetables, which are prepared or produced via a rehydration process. It is a complex process composed of two simultaneous processes: the absorption of water by dried product and the diffusion of soluble[22]. RR is one of the important quality attributes for dried products as it could indicate the physico-chemical changes such as cellular structure and water holding capacity[23]. Rapid and complete rehydration is a desired property of dried products. However, to the best of our knowledge, no reports have been found detailing the effect of pulsed vacuum drying on the drying kinetics, texture, color, and RR of pineapple slices.

Thus, the main goals of the current work were to investigate: 1) the effect of PVD on drying characteristics of pineapple slices under different drying temperatures (65℃, 70℃, 75℃, 80℃) and PVR of vacuum time to atmospheric pressure time (3:5, 5:5, 7:5, 9:5, 5:3, 5:7; 5:9 min:min); 2) the changes of product color attributes in terms of a^*, b^*, L^*, the total color difference (ΔE) ; 3) the changes of texture and RR of pineapple slices under different PVD drying conditions, as well as the microstructure observation; 4) the relationships between drying performance and cyclic pressure and pineapple slices center temperature changes.

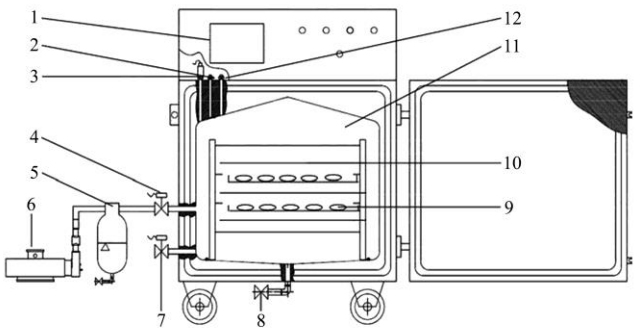
2 Materials and methods

2.1 Material

Fresh pineapples (*Ananas comosus* L.) were produced in Hainan and purchased from Qinghe wholesale market in Beijing. All samples were stored in a refrigerator at 4℃ and 90% relative humidity prior to experiments. To ensure uniformity and accuracy of the experiments, pineapples with the same characteristic (average sugar content and weight were (10.9±0.5) °Brix and (900±50) g, respectively) were selected. The initial moisture content (89.6%±0.5%, w.b.) of samples was determined at a constant 70℃ for 48 h in a drying oven[24]. Prior to the experiment, the fresh pineapples were taken out from the refrigerator and left for about 4 h to reach the room temperature of 20℃. Then samples were peeled, washed, cored, and cut into thin circular slices of 6 mm in thickness.

2.2 Experimental equipment and design

A pulsed vacuum dryer was used in the present study. A schematic diagram of the PVD equipment is shown in Figure 1. The PVD equipment mainly consists of heating, cooling, control, and vacuum systems, which have been described in detail by Xie et al.[15] A vacuum pump (2BV4-2060, Bo-Tong, Shanghai, China) was used to regulate the pressure within the drying chamber. The pressure was measured with CYYB-110 Pressure Transmitter (Shida Chuangye, Beijing, China) in the drying chamber. The thermocouple signals were collected by SHT75 sensors (Sensirion, Shenzhen, China). A proportional-integral-derivative controller (Omron, model E5CN, Tokyo, Japan) with an accuracy of ±0.1℃ was used to control the temperature of water tank and pressure in the drying chamber. An electromagnetic valve isolated the pressure chamber from the ambient air and adjusts the air flow rate from the ambient back into the chamber. Both pressure and thermocouple signals were shown in real-time on the touch screen (Weinview, Shenzhen, China). The minimum pressure level that the system can produce is 8.0 kPa (0.08 bar) and the time taken for the system to reach this minimum pressure from atmospheric pressure is approximately 40 s.



1. Touch screen control panel 2. Material temperature sensor 3. Pressure sensor 4. Vacuum valve 5. Condenser 6. Vacuum pump 7. Air solenoid valve 8. Drain solenoid valve 9. Sample, 10. Far-infrared radiation heating element 11. Drying chamber 12. Infrared-board temperature sensor

Figure 1 Schematic diagram equipment used for pulsed vacuum drying

The drying chamber pressure change kinetics during PVD is shown in Figure 2. During drying, the air was expelled from the drying chamber to a preset vacuum state and maintained for a predetermined time. This step was followed by a pressure recovery step then it was held for the specified duration. This procedure varied according to the properties of the treated products and the operating conditions (e.g., drying temperature, vacuum amplitude, intermittent time). P_A and P_V indicate the highest and lowest pressure in drying chamber. The t_{AP} and t_{VP} are the duration at the atmosphere and the lowest pressure, respectively. The t_s and t_d are the time required during the alternation of P_V and P_A, respectively.

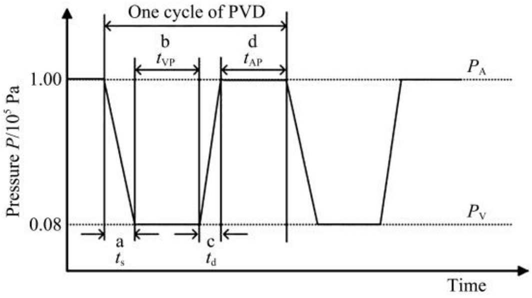


Figure 2 Schematic diagram of drying chamber pressure change kinetics during pulsed vacuum drying

The detailed experimental arrangement is listed in Table 1. For each sub-sample, 20 pineapple slices (the weight was in the range of 60-80 g) were selected randomly from the diameter between 68 and 72 mm. The radiation temperature of 65 ℃, 70 ℃, 75 ℃, and 80 ℃ and PVR (3:5, 5:5, 7:5, 9:5, 5:3, 5:7; 5 min:9 min) were selected as the parameters of experiment. The distance from the radiation heating element to the material tray was 30 mm. The drying system was run for about 20 min to obtain steady conditions and then about 380 g of pineapple slices were spread on the stainless steel tray (45 cm×30 cm) with a thin coat of adherent. The sample tray was taken out at 30 min intervals in the early drying period and was rapidly weighted on a digital balance with an accuracy of ±0.01 g (SP402, Ohaus Co., New Jersey, USA) and then was put back into the chamber. The weight for each group of samples was finished in 30 s. Drying was continued until the weight change of samples within 0.1 g at two successive weight checking. Each experiment was performed in triplicate.

Table 1 Experimental design for PVD of pineapples slices

Experiment number	Drying process	T/℃	PVR	t_T/h
1	Control (raw, non-treated)	-	-	
2	PVD	65	5/5	19.0
3	PVD	70	5/5	14.0
4	PVD	75	5/5	12.5
5	PVD	80	5/5	9.7
6	PVD	75	3/5	15.8
7	PVD	75	7/5	18.0
8	PVD	75	9/5	20.0
9	PVD	75	5/3	15.0
10	PVD	75	5/7	14.0
11	PVD	75	5/9	20.0

Note: PVD is pulsed vacuum drying; T is temperature; t_T is drying time; PVR is pulsed vacuum ratio.

2.3 Drying characteristics

2.3.1 Moisture ratio

The moisture ratio (MR) of the pineapples slices during the drying experiments was calculated using the following Equation (1)[3]:

$$MR = \frac{M_t - M_e}{M_0 - M_e} \tag{1}$$

where, M_t is the moisture content at drying time t, g H_2O/g d.b.; M_0 is the initial moisture content, g H_2O/g d.b.; M_e is the equilibrium moisture content, g H_2O/g d.b. The equilibrium moisture content M_e is relatively small compared to M_t or M_0. Thus, Equation (1) can be written in a simplified form as Equation (2)[3]:

$$MR = \frac{M_t}{M_0} \tag{2}$$

The drying rate of the pineapples slices under various drying conditions was calculated according to Equation (3)[25,26]:

$$DR = \frac{M_{t1} - M_{t2}}{t_2 - t_1} \tag{3}$$

where, M_{t1} and M_{t2} indicate the moisture contents, g H_2O/g d.b. of pineapples slices at time t_1 and t_2, respectively; t_1 and t_2 indicate drying times, h.

2.3.2 Effective moisture diffusivity (D_{eff})

Fick's Second Law is widely used to calculate the effective moisture diffusivity (D_{eff})[3]:

$$MR = \frac{M_t}{M_0} \approx \frac{8}{\pi^2}\exp\left(-\frac{\pi^2 D_{eff}}{L^2}t\right) \tag{4}$$

where, D_{eff} is the effective moisture diffusivity coefficient, m^2/s; L is the thickness of the pineapples slices with 0.006 m as its value; t is drying time, s.

2.3.3 Activation energy

The activation energy for drying was the energy required to initiate mass diffusion from a wet food material during drying. The dependence of the effective moisture diffusivity on temperature is adequately described by the Arrhenius equation[27].

$$D_{eff} = D_0 \exp\left[\frac{E_a}{R(T+273.15)}\right] \tag{5}$$

$$\ln D_{eff} = \ln D_0 - \frac{E_a}{R}\frac{1}{(T+273.15)} \tag{6}$$

where, D_0 is the effective moisture diffusivity at 273.15 K (m^2/s); E_a is the active energy (kJ/mol); R is the universal gas constant 8.32 kJ/mol·K; T is the drying temperature (℃).

The activation energy (E_a) can be determined from the slope of In(D_{eff}) versus the reciprocal of temperature $1/(T+273.15)$ using Equation (6).

2.4 Color

CIE Lab color parameters (L^*, a^*, b^*) were used to quantitatively describe the surface color of the fresh samples and the PVD-dried samples. L^* represents the light-dark spectrum with a range from 0 (black) to 100 (white), while a^* was the red-green spectrum with a range from −60 (green) to +60 (red), and b^* indicates the yellow-blue spectrum with a range from −60 (blue) to +60 (yellow). The color was measured using a colorimeter (Shengmingyang Co., Beijing, China) as described by Wang et al.[32]. The total color difference (ΔE) was calculated according to Equation(7)[21]:

$$\Delta E = \sqrt{(L^* - L_0^*)^2 + (a^* - a_0^*)^2 + (b^* - b_0^*)^2} \tag{7}$$

where, L_0^*, a_0^*, and b_0^* indicate the cross section color parameters of raw sample; L^*, a^*, and b^* indicate the surface color parameters of PVD samples. The results were averaged over 6 measurements.

2.5 Rehydration ratio

RR determination was carried out in a water bath at 40 ℃. Three slices of dried pineapples as a subsample were kept in 120 mL distilled water in a 150 mL glass beaker. Samples were weighed at 5 min intervals during the first 20 minutes and then change to an interval of 10 min. The adhering water was carefully absorbed with filter paper before each weighing. After weighing, samples were immediately returned to the same soaking baker. This procedure was repeated until a constant weight was obtained in two consecutive weighings. All the experiments were performed in triplicate. RR of each subsample was calculated according to Equation (8)[25]:

$$RR = \frac{W_t}{W_0} \tag{8}$$

where, W_t and W_0 are the weight of rehydrated sample at time t and the weight of the dried sample, respectively.

2.6 Microstructure

The microstructure of the vertical cross-section of dehydrated product was observed using a scanning electron microscope (SEM) (S-3500, Hitachi, Tokyo, Japan) at an accelerating voltage of 20 kV[30,31]. The samples were sputter-coated (SC7640, Quorum Technologies Ltd., Newhaven, UK) with an additional thin layer (~10 nm) of gold[28]. The images of representative areas were saved for further analysis.

2.7 Texture

The texture properties of pineapple slices were measured at room temperature (25 ℃) by a texture analyzer (TAXT plus, Stable Micro Systems, UK) with the procedure described by Wang et al.[29] and Deng et al.[30] with some slight modifications. Texture profile analysis (TPA) parameters including hardness (N), springiness (mm), cohesiveness, resilience, and chewiness (mJ) were determined for pineapple slices from PVD and HAD three times. The squeeze tests were carried out, and the small part of the pineapple slices was compressed along the middle axis of the fruit using a 36 mm diameter probe, the compression ratio of fruit tissue of 30%, and 5 s retention between two bites[31]. Ten pineapple slices were selected randomly for each assay.

2.8 Material center temperature

A 3-way inserted Pt100 temperature sensor (Beijing Kunlun Industrial Control Co., Ltd., precision ±0.2 ℃) was used to monitor the temperature change in the drying process of the material center, and draw the obtained data into a curve for further analysis[32].

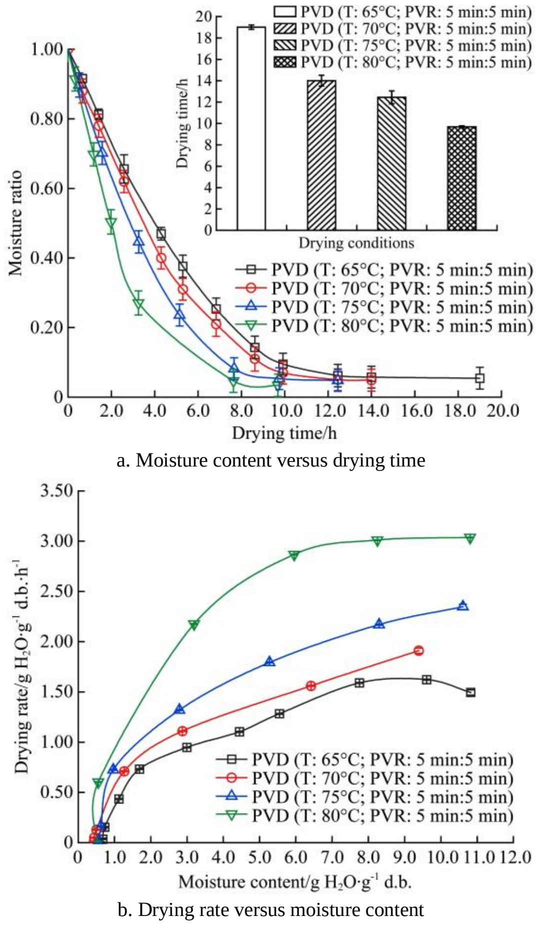
2.9 Statistical analyses

Statistical analyses were performed using SPSS version 13.0 (SPSS Inc., Chicago, IL, USA). One-way analysis of variance (ANOVA) was a method used for testing the differences between samples. Significant differences were determined using Duncan's test at $p<0.05$.

3 Results and discussion

3.1 Effect of drying temperature on drying kinetic curves

As the drying processes, the moisture ratio of pineapple slices decreased until it reached a low point and then became flat. The final moisture ratio of each group was also very close, ranging from 0.03 to 0.05. As expected, the higher the drying temperature, the faster the drying rate. When PVD was conducted at different drying temperatures of 65 ℃, 70 ℃, 75 ℃, and 80 ℃ and constant PVR of 5 min:5 min, the drying times were 19.0 h, 14.0 h, 12.5 h, and 9.7 h, respectively. At 80 ℃, the drying time to reach the final moisture content of 8% (wet basis) was lowered by 48.9%, 30.7%, and 22.4% compared to those of samples dried at temperatures of 65 ℃, 70 ℃, and 75 ℃, respectively. Similar results were reported by Cuccurullo et al.[33] for microwave drying of apple slices at 60 ℃-80 ℃. Significantly, the drying rate curves of pineapple slices were different in the early drying stage under different drying temperatures and constant PVR as shown in Figure 3b. Some of them were falling drying rates, some were constant drying, and others were short-term accelerated drying. Most studies showed that pineapple drying belongs to falling-rate periods of drying. In the study of grape vacuum pulsation drying, the drying rate increased first and then decreased[14]. This also reflects the specificity of pulsed vacuum drying. The material contained a lot of free water, which was easy to remove in the early stage of drying. The drying rate should be fast under the condition of constant temperature drying. However, the PVD of pineapple slices at 65 ℃ showed different characteristics. The reason may be that the temperature of the pineapple itself was low in the early stage of drying, and the heat provided by the radiant plate was mainly used for the rise of material temperature, which made the heat used for water evaporation relatively less, so the drying rate was relatively low; after that, as the material temperature rose to a certain extent, more heat can be used for water evaporation, so the drying rate increased[15, 29].



a. Moisture content versus drying time

b. Drying rate versus moisture content

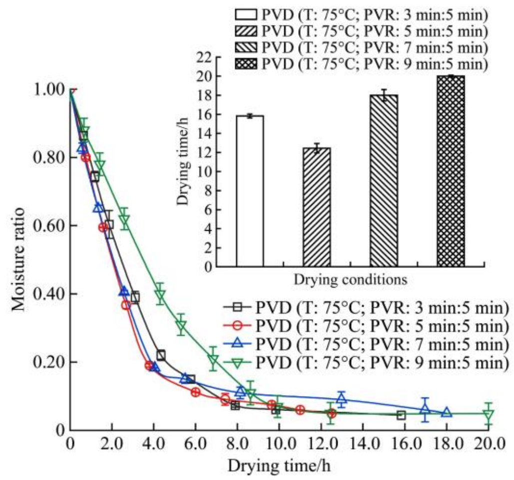
Note: PVD is pulsed vacuum drying; T is temperature, ℃; PVR is pulsed vacuum ratio. The same as below.

Figure 3 Changes in moisture content versus drying time and drying rate versus moisture content during PVD of pineapples slices

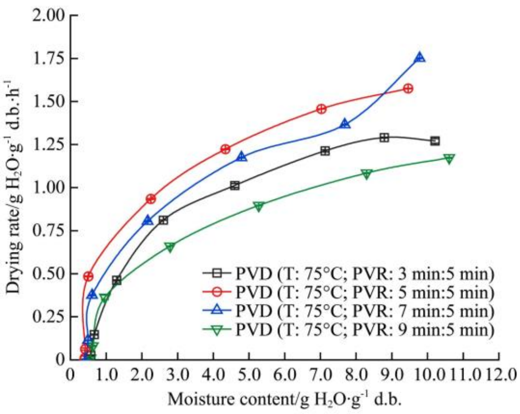
The drying rate of food materials was determined by the relative rate and interaction between the heat transfer inside the food and the mass transfer of water from the food to the external environment[34]. From Figure 3b, the whole drying process was close to occurring in the falling rate period. This indicated that diffusion can be considered the dominant physical mechanism governing moisture transfer during the whole drying process. This result was generally in agreement with that reported on the drying of hawthorn fruit, sour cherries, and sorghum[25,35,36]. This also met the premise of using Fick's second law of diffusion.

3.2 Effect of vacuum time on drying kinetic curves

The effect of different vacuum times to constant atmospheric pressure time on the changes in moisture contents (MC) and drying rates (DR) of pineapple slices was evaluated, shown in Figures 4a and 4b, respectively. Generally, the MR of the pineapple slices decreased gradually with the increase in the drying time. A significant effect of PVR on the changes in DR was found during PVD of pineapple slices. PVD at 75℃ and PVR of 3:5, 5:5, 7:5 and 9:5 min:min took 15.8 h, 12.5 h, 18.0 h, and 20.0 h, respectively. The results indicated that the optimum PVR was 5 min:5 min and the drying was reduced by 37.5% compared to the drying time under PVR of 9 min: 5 min. The boiling point of water in vacuum was lower than that under normal pressure. Water evaporated more easily in vacuum environment under the same conditions. Therefore, the dehydration of materials was mainly carried out in vacuum environment. If the vacuum time was too short, it was not conducive to drying, and vice versa. The reason for this phenomenon may be related to two aspects. On the one hand, the material temperature decreased due to the evaporation and heat absorption of water on the material surface in the vacuum stage. On the other hand, the rate of moisture migration from the material to the pore began to slow down, resulting in the decrease of drying rate due to the removal of some water in vacuum. At this time, switching to normal pressure, it was similar to the effect of water homogenization[18,29]. Therefore, it was very important to select the appropriate vacuum holding time and then switch to the normal pressure state for rapid drying.



a. Moisture content versus drying time

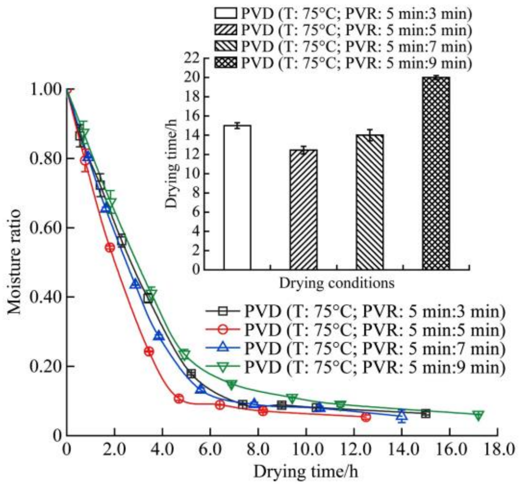


b. Drying rate versus moisture content

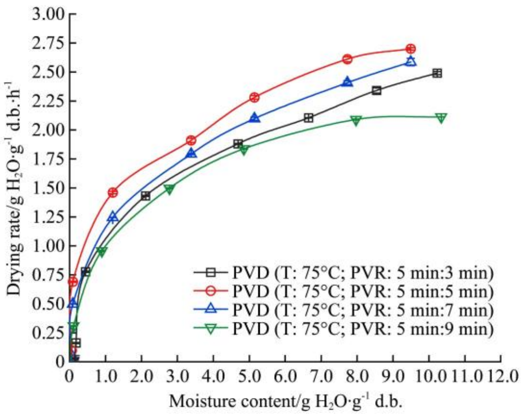
Figure 4 Changes in moisture content versus drying time and drying rate versus moisture content during PVD of pineapples slices at constant drying temperature, atmospheric pressure time, and different vacuum time for pulsed vacuum ratio

3.3 Effect of atmospheric pressure time on drying kinetic curves

As shown in Figure 5a, the drying time of constant drying temperature at 75 ℃, vacuum time at 5 min, and atmospheric pressure time at 5 min to reach the final moisture content of 8% (wet basis) was lowered by 17.0, 11.1, and 37.8% compared to those of samples dried at 3, 7, and 9 min, respectively. From Figure 5b, it was noted that DR decreased continuously as the moisture content decreased, which indicated that the moisture diffusion control over the whole drying process. These phenomena proved that the atmospheric pressure time had a significant effect on the drying rate. The function of atmospheric stage was to make the material absorb enough heat to reach a higher temperature, and provided energy for the rapid evaporation of moisture in the following vacuum stage[15,18]. Therefore, too long or too short atmospheric pressure time was not conducive to drying.



a. Moisture content versus drying time



b. Drying rate versus moisture content

Figure 5 Changes in moisture content versus drying time and drying rate versus moisture content during PVD of pineapples slices at constant drying temperature, vacuum time, and different atmospheric pressure time for pulsed vacuum ratio

3.4 Effective moisture diffusivity

Effective moisture diffusion coefficient reflects the dehydration ability of materials under certain drying conditions and is considered one of the important parameters for designing an optimum drying process[37]. Due to the dehydration of the pineapple slices mainly occurring in the falling drying-rate stage, Fick's second law (Section 2.3.3 Equation (4)) can be applied to describe the water diffusion behavior during the drying process. As shown in Table 2, when PVR was 5 min: 5 min and drying temperature ranged from 65 ℃ to 80 ℃, the $D_{\rm eff}$ values were in the range from 5.9544×10^{-10} to 1.0080×10^{-10} m^2/s. The $D_{\rm eff}$ increased greatly with increasing drying temperature. Since increasing drying temperature could increase moisture diffusion, and thus moisture diffusivity was increased[38]. A similar result was found by Agnihotri et al.[39] for drying of inula racemosa and Bezerra et al.[40] for drying passion fruit peel. For different vacuum times, the $D_{\rm eff}$ ranged from 8.0121×10^{-10} to 8.9601×10^{-10} m^2/s. It was worth noting that the effective moisture diffusion coefficient (8.9601×10^{-10}) at PVR of 5 min: 5 min was higher than other pulsating ratio conditions. This confirmed the conclusion of drying characteristics (Section 3.2–3.3) mentioned above again. The appropriate pulsation ratio can not only promote the material temperature rise but also promote the rapid removal of moisture from the material surface to air. A similar phenomenon occurred in Lou et al.[41] who used a hot air impingement dryer to jujube and Bai et al.[14] who carried out pulsed vacuum drying of grapes. All values obtained from this study were within the general range of reported diffusivities for fruits and vegetables, 10^{-11}-10^{-9} m^2/s[25,42].

Table 2 ln*MR* linear regression formulas and effective moisture diffusion coefficients

Experiment number	Linear regression equation	Coefficient of determination (R^2)	$D_{\rm eff}$/(m^2·s^{-1})
1	--	--	--
2	$\ln MR=-9.1731\times10^{-5}t+0.3965$	0.9540	5.9544×10^{-10}
3	$\ln MR=-1.1612\times10^{-4}t+0.3402$	0.9705	7.5376×10^{-10}
4	$\ln MR=-1.3804\times10^{-4}t+0.3841$	0.9524	8.9601×10^{-10}
5	$\ln MR=-1.5530\times10^{-4}t+0.3315$	0.9584	1.0080×10^{-9}
6	$\ln MR=-1.3155\times10^{-4}t+0.4273$	0.9447	8.5390×10^{-10}
7	$\ln MR=-1.2697\times10^{-4}t+0.3057$	0.9842	8.2421×10^{-10}
8	$\ln MR=-1.0527\times10^{-4}t+0.1580$	0.9817	8.0121×10^{-10}
9	$\ln MR=-1.2284\times10^{-4}t+0.4480$	0.9319	7.9738×10^{-10}
10	$\ln MR=-1.3385\times10^{-4}t+0.4325$	0.9515	8.6887×10^{-10}
11	$\ln MR=-1.0024\times10^{-4}t+0.3241$	0.9614	7.4331×10^{-10}

3.5 Activation energy

Drying activation energy refers to the starting energy required to remove unit moisture from the material during the whole drying process, which can be seen from the drying activation energy, and the energy consumption required for drying can be estimated[43]. The activation energy of pineapple drying can be calculated according to the correlation between drying temperature and effective diffusion coefficient of water, and the activation energy of pineapple drying was 41113 J/mol by using Equation (6), which indicated that the start-up energy required to remove 1 mol of water from pineapple is 41.11 kJ.

A large number of studies showed that the drying activation energy of most materials was between 12.70 and 110.00 kJ/mol, as shown in Table 3[44]. Different drying technology, drying process, and pretreatment methods can affect the drying activation energy of pineapple, and in fact, the components, tissue structures, and specific surface area of the product have a significant effect on its activation energy. The activation energy of crisp and ripe jujube were 17.19 kJ/mol and 49.00 kJ/mol, respectively[45]. The reason for this phenomenon was related to the maturity of materials, which can be divided into two aspects. On the one hand, the adsorption of water was enhanced with the increase in sugar content. On the other hand, the surface of mature jujube began to shrink, which hindered the moisture migration. The drying activation energy of pineapple was between apple and grape, which indicated that the drying difficulty of pineapple was lower than that of grape but slightly higher than that of apple. Ju et al.[46] also expressed that the products with higher content of sugar or pectin, compact tissue structures, and small specific surface area typically have a higher activation energy than the ones which have a low content of sugar or pectin, porous structures, and large specific surface area.

Table 3 The drying activation energy of pineapple and some common fruits and vegetables under different drying conditions

Material	Drying condition	Ea/kJ mol^{-1}	Reference
Pineapple	Pulsed vacuum drying, (65-85) ℃, 15:3	41.11	This study
Pineapple	hot air drying, (40-70) ℃, 1.5 m/s	36.06	Ramallo et al. (2012)[47]
Pineapple	Hot air drying, (40-70) ℃, 2.5 m/s	41.45	Ramallo et al. (2012)[47]
Pineapple (osmotic treatment)	Hot air drying, (40-70) ℃, 2.5 m/s	39.55	Ramallo et al. (2012)[47]
pineapple	Hot air drying, (50-80) ℃, 0.02 m/s	35.50	Liu et al. (2020)[48]
Apple	Mid-short wave infrared radiation drying, (60-80) ℃	36.58	Ju et al. (2013)[46]
Thompson seedless	Pulsed vacuum drying, (55-70) ℃	56.39	Bai et al. (2014)[14]
Chinese wolfberry	Pulsed vacuum drying, (60-70) ℃	54.30	Xie et al. (2017)[15]

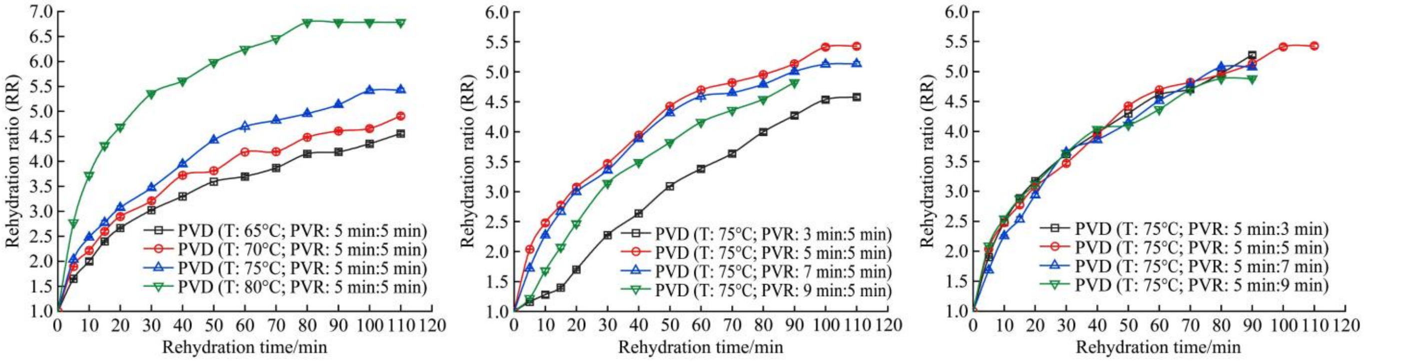
3.6 Rehydration kinetic

Rehydration ability is an important evaluation index of pineapple drying quality. Figure 6a showed the RR dynamic change of dried pineapple slices with rehydration time under the condition of constant PVR at 5 min: 5 min and different drying temperatures at 65 ℃, 70 ℃, 75 ℃, and 80 ℃. Obviously, RR of pineapple increased gradually until it reached saturation and tended to a fixed value as rehydration time increased. In addition, RR just started to be faster, and then gradually decreased. The final RR of pineapple ranged from 4.5 to 7.0, and the time required for RR to reach saturation was within 120 min. Although pineapple continuously absorbed water during rehydration, it could not recover to the fresh level. The ability of rehydration depends on the degree of cell and structure damage, irreversible cell rupture, and translocation, leading to the loss of integrity, cell collapse after dehydration, the structure becomes dense, capillary shrinkage, hydrophilic properties of cells decreased, which reflects that rehydration cannot be completely realized[49].

Different drying temperatures resulted in different rehydration rates and rehydration abilities of pineapple. The maximum RR of pineapple at 80 ℃ was 6.78, while the minimum RR of pineapple at 65 ℃ was 4.55. The rehydration rate and rehydration capacity of pineapple increased with the increase of drying temperature in the range from 65 ℃ to 75 ℃. At 80 ℃, the rehydration ability of pineapple suddenly increases, which may be due to the serious damage to pineapple cell structure caused by too high temperature. Part of the cell components in pineapple flow out from the material during rehydration, which provides more space for water and therefore absorbs more water. Vega et al.[50] also had a similar phenomenon when they studied hot-air drying of red pepper. He thought that too high temperature caused too much cell damage, and some cell solutes were taken away by water migration, leaving more space in the cells for water absorption during rehydration, so the rehydration ability was extremely strong. From the study of the effect of drying temperature on the rehydration ability of pineapple, it can be concluded that an appropriate drying temperature (75 ℃) was conducive to the preservation of the rehydration ability of dried pineapple. Zhao et al.[51] had similar conclusions when they studied the hot air drying of eggplant.

The rehydration ability of pineapple varied with vacuum time. From Figure 6b, the rehydration capacity of pineapple is the highest (5.43) of the vacuum time at 5 min. The results showed that the quality of pineapple is the best when the drying temperature is 75℃, the atmospheric pressure time is 3 min, and the vacuum time is 5 min. Moreover, the RR of vacuum time at 3 min was significantly lower than that of other drying conditions. PVD at a long vacuum time resulted in a tunneling impact that enlarged and interconnected the micropores of pineapple slices. Thus, it resulted in several physical and structural modifications that enhanced moisture transfer during drying. The results were consistent with the finding of previous works of Xie et al.[15] and Wang et al.[52] who discovered that RR of dried samples increased significantly from 2.53 to 2.82 with increasing vacuum pressure duration from 10 to 20 min (p<0.05). This result may be ascribed to the vacuum condition which can contribute to the inducement of greater internal stresses and the creation of more micro-fissure pores during drying.

The rehydration curves of each group were very close, almost coincident, and the difference of RR at the final equilibrium was very small, which indicated that the normal pressure time had little effect on the rehydration characteristics of pineapple (Figure 6c).



a. PVD at different drying temperatures and constant pulsed vacuum ratio

b. PVD at constant drying temperature, atmospheric pressure time, and different vacuum time for pulsed vacuum ratio

c. PVD at constant drying temperature, vacuum time, and different atmospheric pressure time for pulsed vacuum ratio

Figure 6 Rehydration ratio of PVD pineapple slices

3.7 Color evaluation

L^* is one of the indicators to describe the light and shade of color, ranging from 0 to 100. The values of L^* significantly decreased (p<0.05) as the drying temperature increased from 65℃ to 80℃ and were much smaller than that of fresh samples. The fact was that browning occurred during the drying of *Panax notoginseng* roots as the L^* values decreased. The result was in agreement with the results of Pathare et al.[21], who revealed that when the grape samples were treated with the same condition, the L^* values of the raisins dried at 70℃ were significantly lower than the raisins dried at 55℃ to 65℃. Samples dried by PVD at 80℃ obtained the lowest L^* values. These values were found to be significantly different (p<0.05) than those dried at lower temperatures (Table 4). The results indicated that high temperature accelerated the decomposition of sugars in pineapple slices, and aggravated the occurrence of Maillard reaction. In addition, there was no significant difference in L^* value of the PVR at 5 min: 5 min and the drying temperature at 65℃ and 75℃. This change may be due to the combined effect of drying time and drying temperature[32]. It can be concluded that too high or too low drying temperature will lead to a great change in the L^* value, and only appropriate low temperatures can ensure the minimum change in L^* value. The a^* value represented the value of red and green, ranging from −60 to 60. The larger the value of a^*, the redder the color. The a^* value increased from 0.50±0.09 to 2.70±0.13 as the drying temperature increased from 65 to 80℃, and the maximum a^* value was 2.70 of the drying temperature at 80℃ and constant PVR at 5 min: 5 min, which was much higher than that of fresh samples. This was because when the drying temperature is 80℃, pineapple slices had a large area of coking, which led to red color and confirmed that a^* value can reflect the coking situation of pineapple slices to a certain extent[53]. The b^* value represents the yellow and blue values, ranging from −60 to 60. There was no significant (p>0.05) difference in the b^* value of pineapple slices dried at different temperatures, ranging from 49.15 to 52.77. ΔE value reflects the overall change of color, the smaller ΔE value indicate better color retention. The change of ΔE value was opposite to that of L^* value, that is to say, ΔE value increased significantly as the drying temperature increased from 65 to 80℃. Therefore, it can be determined that proper low temperature can retain the original color quality of pineapple slices to the greatest extent.

Table 4 Color parameters (L^*, a^*, b^*) of raw and dried pineapple slices and total color difference (ΔE) between raw and pulsed vacuum dried (PVD) samples

Experiment	L^*	a^*	b^*	ΔE
Control (raw, non-treated)	66.23 ± 1.47^{a}	-3.54 ± 0.57^{d}	48.15 ± 0.49^{c}	--
PVD 65℃, PVR 5:5	44.90 ± 0.45^{b}	0.50 ± 0.09^{c}	52.77 ± 1.24^{a}	22.62 ± 0.63^{f}
PVD 70℃, PVR 5:5	46.65 ± 0.95^{b}	1.88 ± 0.14^{b}	50.56 ± 0.47^{ab}	20.46 ± 0.92^{g}
PVD 75℃, PVR 5:5	39.16 ± 1.00^{c}	2.35 ± 0.16^{ab}	51.38 ± 0.37^{ab}	27.97 ± 0.99^{e}
PVD 80℃, PVR 5:5	28.85 ± 0.08^{f}	2.70 ± 0.13^{a}	49.15 ± 0.92^{bc}	37.62 ± 0.05^{b}
PVD 75℃, PVR 3:5	31.03 ± 0.25^{e}	2.33 ± 0.30^{ab}	50.97 ± 0.23^{ab}	35.80 ± 0.32^{c}
PVD 75℃, PVR 7:5	35.01 ± 0.19^{d}	2.67 ± 0.16^{a}	51.67 ± 0.76^{a}	32.03 ± 0.30^{d}
PVD 75℃, PVR 9:5	29.95 ± 1.06^{ef}	2.80 ± 0.28^{a}	52.50 ± 1.27^{a}	37.10 ± 0.84^{bc}
PVD 75℃, PVR 5:3	34.97 ± 0.91^{d}	2.47 ± 0.23^{ab}	51.55 ± 1.06^{ab}	32.03 ± 0.73^{d}
PVD 75℃, PVR 5:7	30.58 ± 0.74^{ef}	2.92 ± 0.19^{a}	52.33 ± 1.87^{a}	36.50 ± 0.91^{bc}
PVD 75℃, PVR 5:9	24.99 ± 0.94^{g}	2.86 ± 0.20^{a}	51.84 ± 1.07^{a}	41.91 ± 0.86^{a}

Note: The different superscript letters in the same column indicate significant differences between samples (p<0.05). The same as below.

The maximum L^* value (39.16) occurred when the PVR was 5 min:5 min and the drying temperature was 75℃ compared with other vacuum time conditions. This corresponded to the drying time under different vacuum conditions. In other words, the shorter the drying time, the greater the L^* value. There was no significant effect on a^* and b^* values under different vacuum time. The influence trend of ΔE value in different vacuum time conditions was similar to that of L^* value. Therefore, the effect of vacuum time on pineapple slices drying color change was mainly through affecting the drying time under the same drying temperature. Long drying time may lead to a relatively long time sample in high-temperature environment, which will promote pineapple slices' color change.

The constant vacuum time at 5 min, the drying temperature at 75 ℃, and the L^* values under different atmospheric pressure time (3, 5, 7, and 9 min) were 34.97, 39.16, 30.58, and 24.99, respectively. L^* value first increased and then decreased as the atmospheric pressure time increased from 3 to 9 min. On the contrary, the ΔE value decreased first and then increased. Different atmospheric pressure time had no significant effect on a^* and b^* values. The results showed that the color of pineapple slice was bright and the color difference was small when the drying temperature was 75 ℃ and the PVR was 5 min: 5 min.

3.8 Texture analysis

The texture index can help people to understand the crispness and rejection of fruits and vegetables in numerical terms, and indirectly understand whether the taste is satisfactory, which can be used as an important sensory evaluation basis. The effects of different PVD drying conditions on the texture of pineapple slices as compared with fresh samples are listed in Table 5. The hardness was associated with structural strength under compression[54]. The hardness of pineapple slices increased with the increase in drying temperature, vacuum time, and atmospheric pressure time. The reason for this phenomenon was that the increase in drying temperature, vacuum time, and atmospheric pressure time will result in the increase of drying strength, which indirectly affects the surface hardening and the increase of internal structure strength of the material. As expected, the hardness of pineapple slices under different drying conditions was significantly lower than that of fresh samples of 171.55 g due to the removal of moisture and the microstructure becoming compact. The cohesiveness measured the difficulty in breaking down the internal structure of samples under mechanical action[54]. Drying temperature and atmospheric pressure time had no significant (p>0.05) effect on the cohesiveness of pineapple slices, while the cohesiveness decreased as the vacuum time increased from 3 to 9 min. This was because the longer the vacuum time, the greater the damage degree of the negative pressure state to the material structure. The cohesiveness of raw pineapple slices was significantly (p<0.05) higher than that of the dried sample. These results illustrated it may be more difficult to break down the raw pineapple slices. Pei et al. also found that the cohesiveness of raw garlic cubes was significantly (p<0.05) higher than that of blanched garlic cubes and there was no significant difference among blanched garlic cubes[55]. Springiness was the measurement of breaking down the internal structure through the initial compression[54]. There was no significant difference in the springiness of dried pineapple slices, but it was much smaller than that of fresh sample. This was in line with the expectation that the dried sample will become inelastic due to a lack of water filling. In addition, there was no significant difference in chewiness and gumminess between pineapple slices dried by different methods. It should be noted that the chewiness of fresh samples was significantly smaller than that of dried samples, while the gumminess was significantly bigger than that of dried samples. Based on the results, we can observe that texture profile analysis illustrated that all hardness, cohesiveness, springiness, chewiness, and gumminess were significantly (p<0.05) affected by PVD drying conditions compared to fresh samples. It was easy to understand that the internal pores of pineapple slices change dramatically due to the removal of moisture after drying.

Table 5 TPA analysis of fresh pineapple slices and the samples treated by different PVD drying conditions

Experiment	Hardness/g	Cohesiveness/%	Springiness	Chewiness/N	Gumminess/N
Control (raw, non-treated)	171.55±60.63^e	0.86±0.04^a	94.81±3.17^a	108.78±3.57^b	5313.69±99.21^a
PVD 65 ℃, PVR 5:5	462.71±152.61^d	0.42±0.13^c	67.27±7.46^b	171.09±6.23^a	300.52±71.40^b
PVD 70 ℃, PVR 5:5	501.88±50.72^d	0.41±0.03^c	69.53±10.52^b	153.77±16.35^{ab}	173.31±40.60^b
PVD 75 ℃, PVR 5:5	782.21±166.29^c	0.32±0.06^c	65.03±2.57^b	163.76±12.08^{ab}	273.93±47.96^b
PVD 80 ℃, PVR 5:5	921.13±60.63^{bc}	0.32±0.09^c	67.81±2.40^b	147.67±41.68^{ab}	291.33±92.69^b
PVD 75 ℃, PVR 3:5	308.24±2.83^{de}	0.63±0.08^b	59.76±11.94^b	149.66±1.87^{ab}	240.08±115.11^b
PVD 75 ℃, PVR 7:5	1107.08±133.55^b	0.24±0.03^{cd}	65.09±2.50^b	177.78±3.59^a	367.47±48.24^b
PVD 75 ℃, PVR 9:5	1450.04±38.05 ^{a}	0.07±0.05^d	65.35±6.89^b	159.79±0.55^{ab}	220.17±8.22^b
PVD 75 ℃, PVR 5:3	483.31±4.26 ^{d}	0.37±0.08^c	64.71±4.33^b	152.61±22.16^{ab}	259.75±77.64^b
PVD 75 ℃, PVR 5:7	800.09±7.85 ^{c}	0.29±0.01^c	60.47±0.93^b	150.11±20.14^{ab}	337.86±54.76^b
PVD 75 ℃, PVR 5:9	925.71±15.76^{bc}	0.32±0.01^c	61.58±4.15^b	171.50±18.77^a	308.13±4.79^b

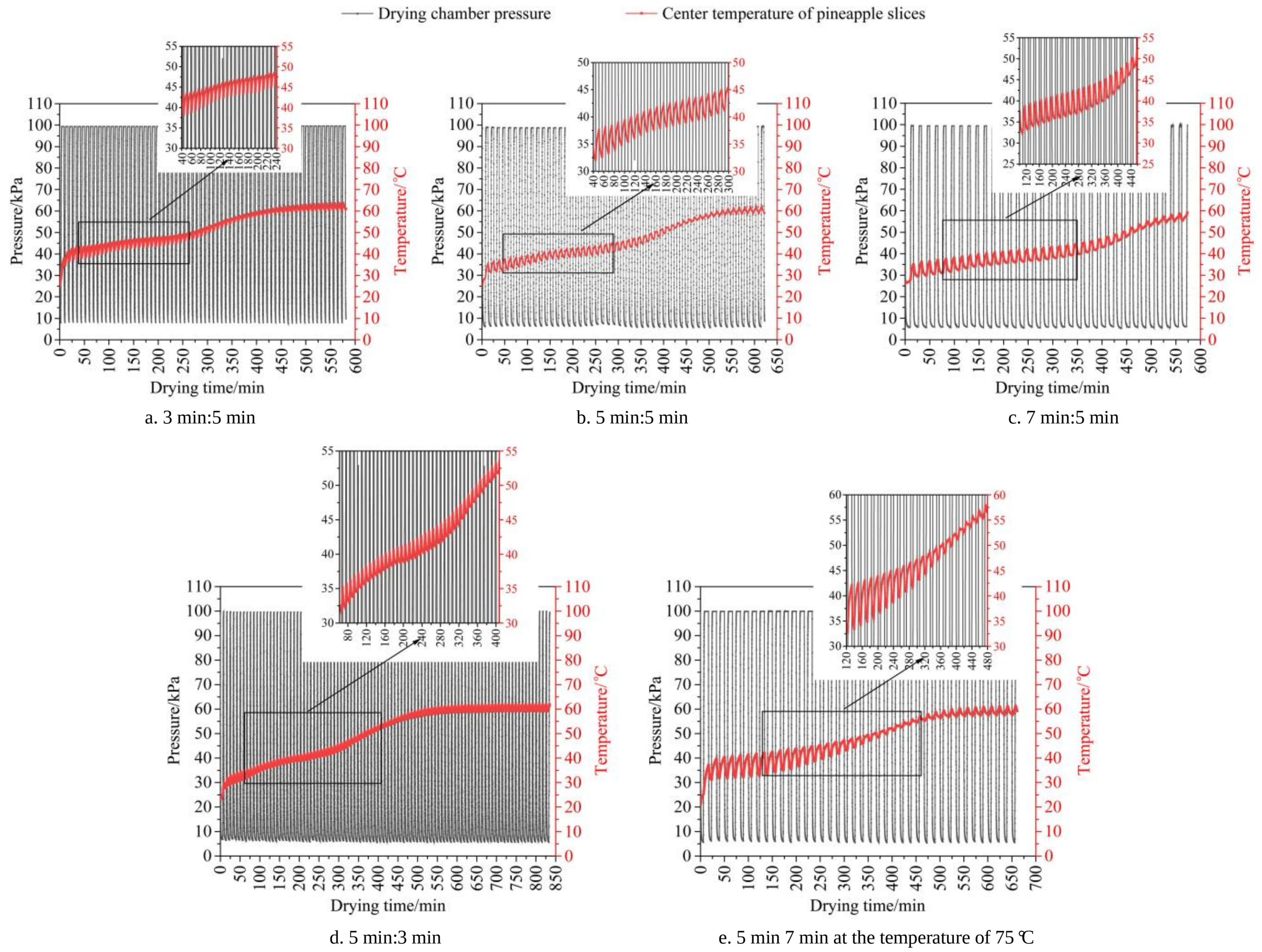
The change in hardness was affected by drying temperature, vacuum time and atmospheric pressure time, which may be related to two aspects. On the one hand, the evaporation rate of water on the material surface was fast, which was easy to cause shrinkage and crusting. On the other hand, the internal structure and pore of the material were irreversibly damaged by the periodic change of pressure. Nathakaranakule et al.[56] also reported that the increasing porosity resulted in the decrease of hardness of longan.

3.9 Effect of pulsing ratio on material temperature

The suitable pulsed vacuum ratio condition based on the material center temperature was good for promoting the moisture removal of the material and accelerating the drying rate. During pulsed vacuum drying, the material temperature and drying chamber pressure histories for pineapple slices with a thickness of 6 mm at a temperature of 75 ℃ and pulsed vacuum ratio of 3:5, 5:5, 7:5, 5:3 and 5:7 were shown in Figure 5, respectively. As observed from Figure 7, the material's internal temperature increased gradually from normal temperature (about 25 ℃) to a certain value (15 ℃ lower than the infrared radiation plate temperature), but there is a pulsating fluctuation during pressure changes: in the atmospheric pressure stage, the material temperature rose to a certain extent, and in the transition from atmospheric pressure to vacuum stage, the temperature dropped suddenly. The possible reason for the fluctuating change of material temperature was that the boiling point of water was higher in the normal pressure stage, so the drying process was difficult to proceed smoothly, the material accumulates a lot of heat energy, and the heat energy consumed by water evaporation was less, which reflects the rise of material temperature; but in the vacuum stage, the boiling point of water decreased, and evaporation absorbed a lot of heat, so the material temperature dropped sharply. Previous researchers had also observed this relationship between material temperature and drying chamber pressure[15,18].

Theoretically, the dehydration of materials was mainly carried out in the vacuum stage, and there was almost no moisture evaporation in the normal pressure stage. This is due to the boiling point of water in the normal pressure stage being much higher than that in the vacuum environment. The heat absorbed by materials was mainly used to raise the material temperature, so as to store energy for dehydration in the vacuum stage and reflect the significance of pulsation. Based on this comprehensive material internal temperature analysis of vacuum holding time and atmospheric holding time on pineapple slice drying by PVD has a sufficient basis. Figures 7a-7c showed the material temperature and pressure curves under the conditions of constant atmospheric pressure holding time of 5 min, drying temperature of 75℃, and different vacuum holding times of 3 min, 5 min, and 7 min, respectively. It can be seen that the fluctuation range of material temperature of vacuum holding time at 3 min is less than that at 5 min, and the fluctuation range of vacuum holding time at 5 min and 7 min was basically the same, but the material temperature of vacuum holding time at 7 min showed a stable and slightly rising trend in the vacuum stage. This showed that the vacuum holding time had a significant effect on the drying process. When the vacuum stage time was too short, the material moisture cannot be fully evaporated, which led to less heat absorption, that is, the material temperature decreased less. When the vacuum stage time was too long, the internal moisture migration of the material was slow, which limited the surface moisture evaporation rate, that is, the material temperature will be stable or slightly increased. In conclusion, too long or too short vacuum time will lead to an increase in drying time. This also confirmed the difference in drying rate with different vacuum holding time in Section 3.2. Similarly, Figures 7b, 7d, and 7e illustrate the curves of material temperature and chamber pressure of constant vacuum holding time at 5 min, drying temperature at 75℃, and different atmospheric holding times at 3 min, 5 min, and 7 min, respectively. The material temperature rose less, which led to insufficient heat accumulation of the atmospheric pressure time at 3 min, while the material temperature rose to a certain degree and tended to be stable of the atmospheric pressure time at 7 min. The comparison between the two results showed that the atmospheric pressure time of 5 min was a proper time node to switch to the vacuum stage, which provides enough heat for moisture removal. This confirmed the result of Section 3.3 again, that is, the drying time of constant drying temperature at 75℃, vacuum time at 5 min, and atmospheric pressure time at 5 min to reach the final moisture content of 8% (wet basis) was lowered by 17.0% and 11.1% compared to those of samples dried at 3 min and 7 min, respectively.

Significantly, keeping the pulsation ratio constant during the drying process was not appropriate. The pulsation ratio of 5 min:5 min showed good adaptability in the early and middle stages of drying, but the material temperature was abnormally stable in the atmospheric or vacuum stages with the drying process. Therefore, it was necessary to further explore the drying process of pineapple slices with variable pulsation ratio in the future.



a. 3 min:5 min

b. 5 min:5 min

c. 7 min:5 min

d. 5 min:3 min

e. 5 min 7 min at the temperature of 75℃

Figure 7 Temperature curves of different pulsed vacuum ratios (PVR)

3.10 Microstructure analysis

The characteristics of microstructure reflect the macro phenomenon, which helps us to explain the essence of the phenomenon more deeply. SEM observations indicated that pulsed vacuum dried pineapple slices were composed of typical polyhedric shapes of phloem parenchyma cells with smooth cell walls similar to that of fresh carrots[57]. The material surface structure collapses, the cells deform compactly, capillary contraction and the pores were less due to the unsuitable pulsed vacuum ratio (Figures 8c, 8d). As compared with sample dried at PVR of 9:5 (Figures 8c, 8d) which had the longest vacuum pressure holding time, surface bulges of sample dried at PVR of 5:5 were arranged looser (Figure 8a), with more even and consistent cell size and shape, more voids (Figure 8b). The reason was that the appropriate PVR, i.e. switching between vacuum and atmospheric pressure at the right time node, can affect the material structure and promote moisture transfer. That was why a higher RR was obtained at that condition (PVR of 5:5).

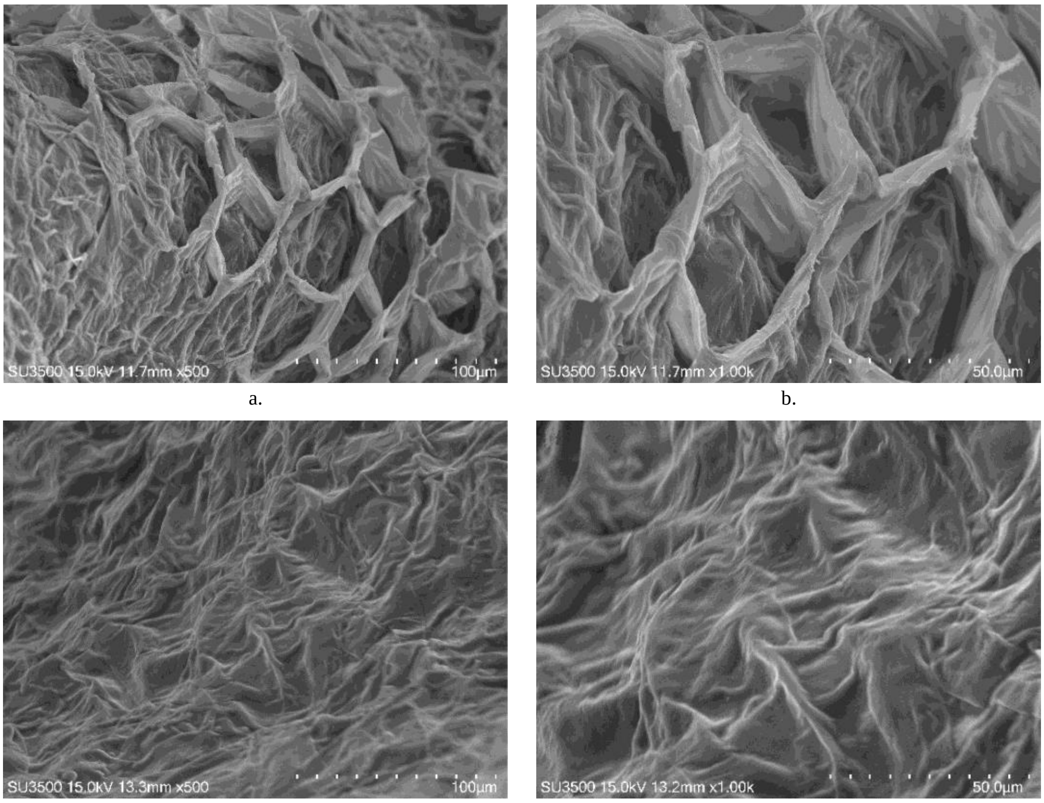


Figure 8 SEM images of pulsed vacuum dried pineapple slices surface in different magnifications (a, b: pulsed vacuum ratio of 5:5 in different magnifications; c, d: pulsed vacuum ratio of 9:5 in different magnifications)

The above findings indicated that appropriate PVR was helpful for the formation of a looser structure, similar to the conclusion of Wang et al.[52] and Xie et al.[18] that optimal pulsed vacuum drying process could expand air and water vapor and create a frothy and puffed structure[58] so as to obtain better rehydration performance.

4 Conclusions

Taking the material center temperature as the reference, the optimal PVR of 5:5 was beneficial in accelerating the drying rate of pineapple slices. The effective moisture diffusion coefficient (8.9601×10^{-10}) at PVR of 5:5 was higher than other pulsating ratio conditions. The material temperature increased during the normal pressure period, and decreased rapidly when the pressure dropped to the vacuum condition, which indirectly reflected the moisture transfer occurred during vacuum holding period, while moisture diffusion happened during atmospheric pressure holding period. Optimal pulsed vacuum drying process (PVR of 5:5) could expand air and water vapor and create a looser structure so as to obtain better rehydration performance (about 5.43). High drying temperature will lead to the decrease of L^* value, the increase of ΔE value, and even the formation of surface scorch at 80 ℃. Under the same drying temperature, the color quality depends on the drying time, and the longer the drying time, the bigger the color difference. The chewiness and hardness of pineapple slices dried by PVD were significantly higher than those of fresh samples, which was conducive to the chewing taste. Further, more studies focused on exploring the drying process of pineapple slices with variable pulsation ratios in the future and extending to the drying of various food products with different structures.

Acknowledgements

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