Advance Journal of Food Science and Technology 12(6): 331-336, 2016
DOI:10.19026/ajfst.12.2969
ISSN: 2042-4868; e-ISSN: 2042-4876

Submitted: November 3, 2015 Accepted: January 16, 2016 Published: October 25, 2016

Research Article

Research on Production Technology of Low-temperature Superfine Grinding Jujube

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Abstract: At present, the drying methods for jujube powder consists of powder process after jujube dryness and powder process after concentrating jujube juice. In order to obtain jujube powder with small particle size, good rehydration, higher VC content and better color, this study proposes to adopt different drying methods, namely, first carry out preliminary cooling against the jujube pieces at low temperature before drying and then take the secondary cooling method during the drying process, in order to make jujube pieces brittle and get superfine jujube powder. After comparing the jujube powder prepared through ordinary drying methods and the drying method of low-temperature superfine grinding, results show that in terms of physical properties, the latter may get smaller particle size, lighten color and better tastes of jujube powder particle than the former; in terms of nutrient content, the latter has relatively higher reducing sugar and total sugar contents. Therefore, the second drying method is superior to the first one. In this context, this study analyzes the structure, drying principle and force required by the drying process of drying equipment of low-temperature superfine grinding system. In the design process of grinding system, this study attempts to separate the nucleation and growth process of powder particles into different parts for drying equipment, in order to effectively control the particle size of jujube powder, reduce the particle agglomeration when collecting the jujube powder and improve the quality and uniformity of jujube powder.

Keywords: Brittleness, low temperature, red jujube, superfine crushing

INTRODUCTION

Red jujube is a fruit of endemic to China, has a high nutritional value and medicinal value, and has a reputation of "king of the fruits". China is the country of origin of red jujubes and also is the world's largest producer of red jujubes (Zhang *et al*., 2013). According to statistics, the total of red jujube cultivation in China reached more than 400 million tons with an average annual growth rate of more than 11% and jujube trees have been generally enter full fruit period (Zhou *et al*., 2013). However, at present red jujube selling limited to domestic, products mainly are dried jujubes, some red jujubes are processed into candied jujube, jujube juice and jujube wine, but mostly for primary processing products, form is single and the added value is low (Xu *et al*., 2012; Zhou *et al*., 2013). The deep processing of Jujube products is still in the primary development stage, the overall size is small, the product variety is less and can not form a valid transformation of jujube resources (Kim *et al*., 2012). Therefore, according to the present situation of Chinese jujube industry development, develop the diversified products and make the comprehensive utilization of jujube resources have important scientific significance and application value to the development of red jujube industry. In this study, used liquid nitrogen cooling technology to crush seedless red jujubes under cryogenic vacuum freezing conditions, produced ultra fine particles original jujubes powder without any additives and provide reference basis for the research and industrial production of jujubes powder processing technology.

MATERIALS AND METHODS

Materials: The raw materials are Xinjiang Hetian jujube purchased from Harbin Hada Fruit and Vegetable Market.

Instruments and equipment: FW 100 high-speed universal grinder, Tianjin Taisite Instrument Co., Ltd.; WH-A-12 thermostatic blast oven, Nanjing Wolfowitz Technology Industry Co., Ltd.; QDPH10-1 variable temperature and pressure difference fruit and vegetable puffing dryer; Alphal-4L-plus vacuum freeze dryer, German Christ company; Quanta200ESEM environmental scanning electron microscope, American FEI company; AL204 0.1mg electronic analytic balance, Mettler-Toledo Instruments Shanghai Co., Ltd.; jujube low-temperature grinder is independently designed by the research group.

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Method: Take jujube raw material, remove the damaged jujube and weigh 5kg, clean with flowing water, dry 2min with thermostatic blast oven, increase the permeability of jujube peel, enucleate and cut it into thin pieces with a thickness of 3mm. By pre-drying 3 min at 60℃ hot air, carry out puffing and dryness for 5 min at 90℃ and the puffing temperature of 0.2 MPa; conduct vacuum freezing dryness for 5 min at the cold trap temperature of -45℃ and vacuum degree of 0.1 MPa and prepare the jujube pieces with moisture contents below 5%.

Preparation of jujube powder: Process ordinary coarse powder, put the dried jujube pieces into FW 100 high-speed universal grinder to prepare powder and grind 3 times in total.

Process the low-temperature superfine powder, put the dried jujube pieces into independently designed low-temperature jujube grinder to prepare powder and grind 3 times in total.

Determination of rehydration: Respectively take 1g coarse and fine jujube powder into 50 mL centrifuge tubes, add 20 mL distilled water and quietly place 1h at 25℃. Respectively centrifuge 30min at a speed of 10000r/min and the precipitate quantity is the mass of rehydration powder. Apply Eq. (1) to calculate the rehydration of jujube powder (Costa *et al*., 2004):

$$w = \frac{m_2 - m_1}{m_1} \times 100\% \quad (1)$$

w : Rehydration, (%)
m_2 : Jujube powder mass before rehydration, (g)
m_1 : Jujube powder mass after rehydration, (g)

Determination of powder particle size: For the micro-morphology of prepared jujube powder, observe and determine the surface morphology and structure of determined samples by using the scanning electron microscope and conduct the morphology analysis against jujube powder with different particles.

Determination of nutrients: Reductive sugar is determined by using 3, 5-dinitrosalicylic acid colorimetry (Lindsay, 1973); total sugar is determined by using phonel-sulfate method (Kim *et al*., 2012); VC is determined in accordance with GB/T6195-1986 Determination Method for Fruits and Vegetable VC Contents (2; 6-dichlorindophenol titration method) (Comprehensive laboratory of Jiangsu Agricultural Sciences Academy, GB/T6195-1986).

RESULTS AND DISCUSSION

Physical characteristics of jujube powder under different drying methods:

Effects on jujube powder rehydration and particle size: Jujube powder prepared by low-temperature superfine drying methods has better rehydration and smaller particle size than ordinary high-speed dried jujube powder. As a result, the specific surface area of dried powder is large and it is easier to absorb after eating (Fig. 1).

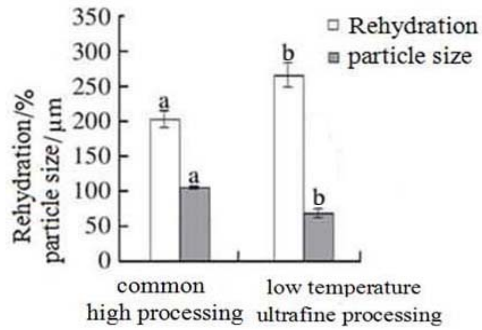


Fig. 1: Rehydration and particle size of jujube powder prepared by different processing methods

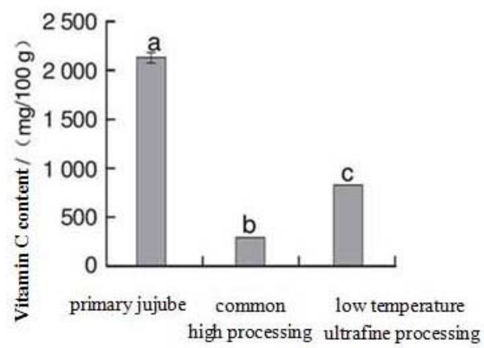


Fig. 2: Vitamin C content of jujube powder prepared by different processing methods

Determination of rehydration effects on jujube powder VC contents: Figure 2, VC contents have losses by using different drying methods, which are lower than the original jujube contents. It is very easy for VC to get heated and damaged. In the drying process, the ordinary high-speed drying method will generate lots of heat and make the dried jujube powder to lose a lot of VC; whereas for low-temperature superfine drying method, it cools the dried environment, in order to protect jujube powder from heat and oxidation during the drying process and retain VC to the maximum degree.

Effects on reducing sugar and total sugar contents of jujube powder: From Fig. 3, Jujube powder prepared by low-temperature superfine grinding drying method has higher reducing sugar and total sugar contents than the jujube powder prepared by ordinary high-speed drying method, yet the reducing sugar and total sugar contents of jujube powder produced by drying method are lower than fresh jujube, because

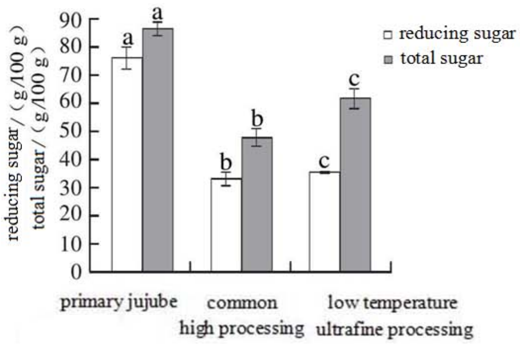
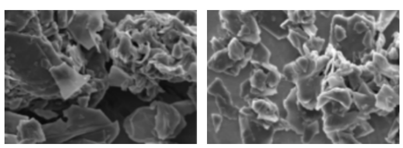


Fig. 3: Reducing sugar and total sugar contents of jujube powder prepared by different processing methods

(a) (b)

Fig. 4: Jujubes powder comparison of different processing methods; (a): Coarse powder; (b): Fine powder



(a) (b)

Fig. 5: Electronic microscope of jujube powder; (a): Coarse powder; (b): Fine powder

jujube has Maillard reaction during the drying process, consuming lots of sugar substance. Jujube powder prepared by ordinary high-speed drying method has lower reducing sugar and total sugar contents, because there are lots of heat during the drying process, causing serious Maillard reaction of heat and more consumption of sugar. During the low-temperature superfine drying process, Maillard reaction is mild and sugar is well preserved, so the total sugar content is relatively high. In addition, during heat, parts of starches are converted into reducing sugar under the effect of amylase, so the re-dried jujube powder does not have higher contents as original jujube.

Definition of jujube powder and morphology analysis: The particle size of jujube powder is closely related to process method, drying method and selected raw materials. Compare the coarse jujube powder ground by grinder with fine jujube powder processed at vacuum low temperature, as shown in Fig. 4. From the figure, the particle size of coarse powder is larger than fine powder. Magnify the jujube powder in Fig. 4, as shown in Fig. 5. By comparing the particle size of jujube powder, the jujube peel in coarse powder is obviously more than fine powder, the particle size of coarse powder is obviously bigger than one of the fine powder, which is determined by drying methods of two powders. The drying method of coarse powder retains the jujube fruit in the form of small particle, yet the drying method of fine powder will not have this phenomenon. Compared with color of fine powder, the color of coarse powder is relatively dark, because jujube contains more than 75% V_C (Zhang *et al*., 2009; Xiao *et al*., 2011), whereas the drying method of coarse powder will definitely release lots of heat during the drying process, making V_C have oxidation and brown stain at high temperature, V_C is severely destroyed and the jujube powder quality is affected. When tasting both of them, obviously feel that the coarse powder has

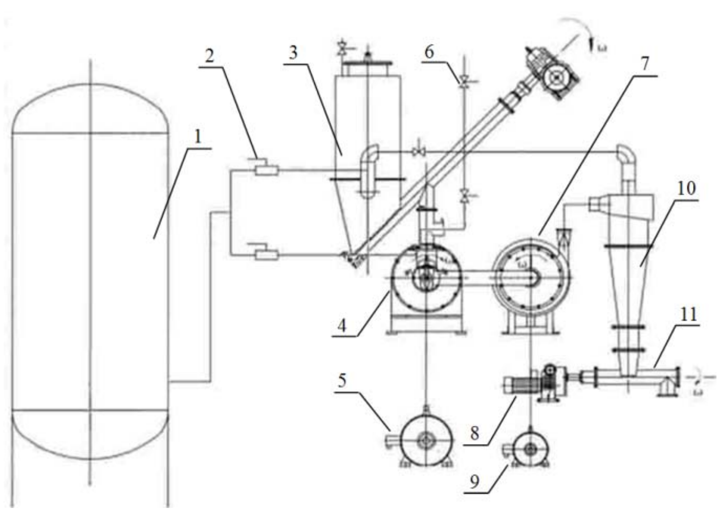


Fig. 6: Overall structure schematic diagram of red jujube cryogenic crushing grinder; 1. Liquid nitrogen canister; 2. Ball valve; 3. Screw feeder; 4. Crushing host; 5. Crushing host motor; 6. Butterfly valve; 7. Induced draft fan; 8. Frequency converter; 9. Induced draft fan motor; 10. Cyclone separator; 11. Spiral discharger

scorched bitter taste, yet fine powder does not have such a taste.

The processing equipment structure and principle of the cryogenic ultra fine crushing red jujube: The technology of cryogenic ultra fine crushing is mainly suitable for heat sensitive materials, food, biological and pharmaceutical products and the raw materials which are difficult to be crushed at room temperature. Cryogenic grinding is that frozen the containing water raw materials under low temperature and made the plastic, impact toughness and elongation of the raw materials which have been embrittlement processed. And carried on the vacuum or low temperature heating, the ice crystals were directly into gas. This kind of ultra fine crushing method is used for impact type crushing. Cryogenic ultra fine crushing application in food processing is still relatively small in China, such as broken ganoderma lucidum spore powder, pearl powder, anthocyanin extraction and eleocharis peel powder drying (Ferrari *et al*., 2012; Xu *et al*., 2011; Lu *et al*., 2010; Xiao *et al*., 2011). As the particle morphology, particle size, crystalline structure of the crushed ultra fine red jujube powder can lead to changes in physical properties and functional properties, so the crushed ultra fine red jujube powder can greatly expand the application field and can provide theoretical basis for the depth development of the red jujube.

System structure of jujube cryogenic grinder: The feed of the cryogenic ultra fine red jujube powder is the processed jujube sheets and the discharging is red jujube powder. Fill in the raw material at room temperature. The internal temperature of the grinder is cryogenic state to crush the raw material. This method can prevent agglomeration or deterioration of red jujube powder in the grinding process as local heating. Overall structure schematic diagram of red jujube cryogenic crushing grinder is shown in Fig. 6.

Liquid nitrogen is the cold source of red jujube cryogenic grinder system. Red jujube sheets which were processed early and thickness is about 3mm will be cooled in advance. The pre freezing temperature of red jujube will be lower 3~ 5°C than the eutectic point -32°C (Luo *et al*., 2011; Xu *et al*., 2011). Red jujube pieces at low temperature brittleness and easy crushing state is sent to the host by crushing screw feeder. High speed rotary multistage tool shears and impacts brittle red jujube pieces in crushing host. Between the red jujube and the cutter, cutter dish and between the red jujube particles be repeated impact, collision, shear and friction in order to reach the purpose of crushing. In the crushing process, adopts the refrigerant secondary injection methods to compensate for jujube heat in the process of crushing (Xiao *et al*., 2011; Lu *et al*., 2010).The injection amount of refrigerant can be adjusted through the cryogenic valve. The air flow generated by the high pressure air flow and the cold medium pressure expansion of the red dates can be discharged through the rotating window of the crushing mill's static classifier. When crushed materials reach the required fineness, will follow the airflow into the discharge system to achieve preliminary classification result and when the relatively thick particles size on the impact to the static classifier blade, will be rebound back into crusher cavity for secondary processing.

Blower will feed the crushed particles and the expanded airflow into the cyclone separator. Through adjusting the rotational speed of blower and the classification particle size of cyclone separator, processing personnel can accord the features of the processed material to adjust the size of the required particles fineness. The use of the air blower and the cyclone separator can reduce the time of the back temperature, shorten the production cycle, reduce the use of the ground and the material can be used for packing and transportation.

After being crushed, red jujube particles are selected, separated and classified by the air stream in the cyclone separator and the fine powder's collection of red jujube was carried out by induced draft fan. The red jujubes particles which do not meet the requirements of fineness will return to the feed bin continue to crush through the spiral feeder. Most of the cooling air will return to the feed bin for cycle use.

Pulverizing system principle analysis of red jujube: The raw material is pre frozen in the process and the inert gas is filled. There is no combustion and chemical decomposition in the process. It completely is a physical change process, so the low temperature crushing process of red jujube powder method is a purely physical preparation method. The results of pre freezing treatment can make the free water of the red jujubes and a part of water adsorbed on the solid crystal lattice gap or combined with hydrogen bonding in some polar groups to be solid water. There is no surface tension between the cell and cell and the relative position has not changed. The pre freeze drying of jujube slices can reduce the agglomeration of jujube powder.

The raw material of pre process is red jujube slices. It has very little free water and the existence of bound water make the processing jujube powder occur to reunite. When the pre freezing temperature reached the eutectic point of red jujube, the combination water of red jujube will be frozen. The cooling rate is faster, the super cooling temperature is lower and the number of the formed nuclei will be more. Crystals have not yet come to grow and have been frozen. The numbers of formed crystal particles are more and the crystal particles are finer. On the contrary, the cooling rate is slower, the grain size is smaller and the grain size is also more thick and big. So, forget the uniform particles and the smaller size particles, not only pre freezing in the process, but also the two injection of refrigerant in the process, to ensure the particle size is micron scale.

Preparation principle of low temperature ultra fine jujube powder: Low temperature processing superfine jujube powder belongs to g the ultra-fine powder equipment, the following problems must be considered: the formed nucleation problem of gas-solid two phases. When design in:

- Whether the gas phase formed the nucleation. Only the over saturation and large enough system can uniform form the nucleation in the gas phase and get micron scale jujube powder.
- Critical grain size. Be according to the nucleation theory to form stable critical radius of nucleus, the Eq. (2) is as follows:

$$r = \frac{2\sigma}{\Delta g_{\upsilon}} \tag{2}$$

σ : The free energy of surface
Δg_{υ} : The difference value in free energy of the gas solid two phase volume

Due to σ along with the temperature change is not obvious, so r depends mainly on Δg_{υ}. And Δg_{υ} mainly depends on the degree of super saturation. The saturation's degree is greater, the Δg_{υ} will be greater. The degree of super saturation is mainly accomplished by local cooling. The temperature decrease has little effect on σ, so the degree of super saturation increases and the critical core size will decreases. Super saturation the Eq. (3) is as follows:

$$S = K(P_A^a \cdot P_B^b \cdot P_C^c) \tag{3}$$

K : The equilibrium constant
A, B, C : Both considered as ideal gases

The degree of super saturation S has a great influence on the rate of crystal nucleus formation. If S can be improved greatly, the rate of nucleation will be increased obviously, the nucleation growth rate was slightly inhibited. On the other hand, it can decrease the degree of super saturation, which is beneficial to the growth of the crystal nucleus, the nucleation process is inhibited. In order to control the particle size, it is necessary to control steam concentration and the number of particles. In the processing device, some of the particles that have been generated are growing up, while others new particles are forming. It is very difficult to separate the two processes of nucleation and growth into different parts of the device and this problem is the key to the design of the technology and equipment of the uniform micro nano powder. Pre cooled raw materials before cold mill processing to make jujube pieces be quickly frozen for lower the eutectic point. In the processing, it is carried out for two times cold filling which is to promote the formation of the crystal nucleus and to reduce the temperature in the host. Choose multiple overlapping thin blades to control the size of crystal nucleus growth, thus can effectively control the uniform size of jujube powder particle size.

The crushing strength of low temperature superfine jujube powder: Cryogenic superfine crushing technology can make the center particle diameter of jujube powder decrease from 80-120 mesh (74~178μm) which is from the traditional crushing process to 18 to 38μm. This range is far less than the upper limit size 200 mesh (75μm) of superfine powder in Chinese medicine, which is prescribed by the Chinese pharmacopoeia in 2000. Jujube fiber is the main factor that affects the red jujubes particle size and the taste and it has a protective effect on the cells of the red jujubes. Low temperature super fine grinding can increase the cell wall broken rate of the jujubes and make the surface area increase. The main chemical composition of jujube are released efficiently such as proteins, amino acids and alkaloids, jujube polysaccharide has health care function include anti-aging, blood and enhance the phagocytosis of macrophage.

The crushing force in the crushing host machine shown in Fig. 3 is f, the Eq. (4) is as follows:

$$f = \frac{[1.79(6)\frac{5}{3m}\rho^{-1}\pi^{\frac{2m-5}{3m}}(\frac{1-V^2}{Y})^{\frac{2}{3}}(S_0V_0^{\frac{1}{m}})^{\frac{5}{3}}]^{\frac{1}{2}}X^{\frac{-5}{2m}}}{t} \tag{4}$$

ρ : Red jujube density (kg/dm^3)
m : Powder Weibull uniformity coefficient
V : Poisson's ratio
V_0 : Unit volume
Y : Young's modulus of elasticity
X : Particle sizeo

The relationship can be seen from the Eq. (4) between the jujube powder particle size, the processing cutting tool and the impact force of powder. The smaller the particle is, the greater the required force for processing is. The size of the crushing force provides theoretical basis for the choice of cutting tool size, the choice of the box body and the choice of the material in the crushing machine. The maximum diameter of the cutting tool group is 260mm. The maximum rotation speed of the tool spindle is 3000r/min and rotational speed is about 64 m/s.

CONCLUSION

Compared with jujube powder prepared by ordinary high-speed drying method, jujube powder prepared by low-temperature superfine grinding method has better color, smaller particle size and good preservation of nutrients. Therefore, the low-temperature superfine grinding drying system has well solved the difficult issue that jujube piece cannot carry out superfine grinding at constant temperature. During the drying process, the temperature can be controlled, the raw materials can also be changed. Select the best grinding temperature according to different raw materials, so that the grinding fineness of raw materials can reach up to 800 mesh (38μm).

ACKNOWLEDGMENT

2013 Youth (Ph.D.) research funded projects of Harbin University (HUDF2014-011).

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