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# Physicochemical Properties of Jujube Powder from Air, Vacuum, and Freeze Drying and Their Correlations

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Abstract Physical and chemical properties of jujube powder with different powder sizes prepared by different drying methods were determined, and correlations of the properties were statistically analyzed. Air-, vacuum-, and freeze-dried (AD, VD, and FD, respectively) jujube were milled and sieved to produce powders. Powder size, morphological characteristics, color, volatile profile, moisture sorption isotherm, water solubility, rehydration, total sugar content, and polyphenol content of the powder were determined. Hunter L, a, and b, degree of browning, and volatile profile varied depending on drying methods. Moisture sorption isotherms exhibited typical Type II sigmoidal shape. Polyphenol content was highest in AD powder (2.6±0.1 mg/g). Powder size, morphology, water solubility, and total sugar content were not different by different drying methods. Correlations identified among the properties indicate rehydration of the powder could be maximized by controlling particle size and water activity of the powder. Rehydration and powder size of AD, VD, and FD powder were significantly correlated (p < 0.001) ( $R^2 = 0.92$ , 0.88, and 0.94, respectively). Property correlations determined could be useful in customizing properties of jujube powder and increasing commercial uses of the powder.

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## Introduction

Jujube (Zizyphus Jujuba Miller) has a long history in its use as a food ingredient and is consumed more than 30 countries in the world (Fang et al., 2009). Dried or half-dried jujube is a most popular form of jujube for food application and mostly prepared by sun drying in Asia countries, including China, Korea, and Japan (Li et al., 2009; Sun et al., 2009). Sun drying, the most economical way of drying, is usually made in the field of harvesting or a greenhouse before shipment. Dried jujube is used in sliced or powdered form in foods and characterized by high concentrations of sugars, ascorbic acid, and polyphenols (Kim and Joo, 2005; Zhu et al., 2010). Dried jujube has been also known to have medicinal functionalities and used as Asian-traditional anticancer, refrigerant, sedative, stomachic, styptic, and tonic medicines (Crawford, 2002; Sun et al., 2009). Interest in the use of dried jujube has been increasing in Asia countries due to its high nutritional and functional properties. Irrespective of wellknown wholesome properties of the dried jujube, its preparation for efficient mass production, especially production of powder, has not been systematically investigated. Further drying of sundried jujube was done for quality control of raw material and powder obtained from the dried jujube. Thus, the effect of different drying methods on the physical and chemical properties of the powder must be investigated for developing industrial production of the powder.

Determining the properties of jujube powder, including powder size, morphological characteristics, color, flavor, water solubility, rehydration, total sugar content, and polyphenol content, is important for its applications to instant convenience food products (Zhang et al., 2005; Lee et al., 2008; Fang et al., 2009). Pearson correlation, multiple linear regression, and response surface analysis (RSA) are used to determine relationships among the properties (Schmidt et al., 1979). Pearson correlation and regression analysis measure the strength of linear dependence between variables (Pearson, 1920; Breiman and Friedman, 1985). RSA is a method for studying geometric relationships among response variables, which are generated by mathematical models (Myung et al., 2000).

Identifying correlations of the properties of jujube powder may allow preparation of the powder that possesses a desired property by manipulating other properties of the powder that significantly affect the property of interest. Thus, the objectives of the present study were to determine physical and chemical properties of jujube powder obtained from hot-air oven drying, vacuum drying, and freeze drying, including powder size, morphological characteristics, color, volatile profile, moisture sorption isotherm, water solubility, rehydration, total sugar content, and polyphenol content and to study correlations among the properties using Pearson correlation, multiple linear regression, and RSA.

## **Materials and Methods**

**Materials.** Jujube was harvested from an orchard (Boeun, Korea) in 2009 at a mature stage and sun-dried in a clean greenhouse for 20 days. Sun-dried jujube was transported to the laboratory in a packaging box and kept in cold storage at 4°C. The fruit without physical injuries or apparent decay was selected for study. The moisture content of dried jujube was 40.8 g/100 g jujube (dry basis), determined at Korea Food Research Institute (Sungnam, Korea). Chemicals, trichloroacetic acid, lithium chloride (LiCl), potassium acetate (CH<sub>3</sub>COOK), magnesium chloride (MgCl<sub>2</sub>), potassium carbonate (K<sub>2</sub>CO<sub>3</sub>), magnesium nitrate (MgNO<sub>3</sub>), sodium chloride (NaCl), potassium chloride (KCl), phenol, sulfuric acid, methanol, folin-ciocalteu, sucrose, tannic acid, and sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>) were purchased from Sigma-Aldrich (St. Louis, MO).

**Jujube drying.** Sun-dried jujube was pitted, sliced, and dried. Drying was conducted in an hot-air oven dryer (VS-1202D2, Vision Scientific Co., Ltd., Bucheon, Korea) at 80°C for 48 h, a vacuum dryer (OV-11, Jeio Tech Co., Ltd., Seoul, Korea) at 50°C and 5 kPa for 24 h, and a freeze dryer (FD 5505, Ilshin Lab Co., Ltd., Yangju, Korea) at -45°C and 1.3 Pa for 72 h to reduce the moisture content of the jujube (40.8 g/100 g; dry basis) to less than 20 g/100 g, which is considered most appropriate for food application of the powder (Kim and Joo, 2005). Moisture contents of hot-air-, vacuum-, and freeze-dried (AD, VD, and FD, respectively) powder were  $19\pm 2$  g/100 g,  $19\pm 2$  g/100 g, and  $18\pm 2$  g/100 g (dry basis), respectively.

**Jujube powder preparation.** Dried jujube was milled using an analytical mill (M20, IKA<sup>®</sup>, Staufen, Germany) with different mesh-sized sieves (32, 32–53, 53–100, 100–150  $\mu$ m) to obtain

powder with different particle sizes. The powder was equilibrated to 11, 15, and 23% relative humidity (RH), corresponding to water activity of 0.11, 0.15, and 0.23, respectively, in desiccators at 25°C before testing. The water activity was measured with a water activity meter (AquaLab pawkit, Decagon Devices, Pullman, WA).

**Powder size.** The average size and the distribution of the sizes of powder dispersed in 10 mL of distilled water were determined using a particle size analyzer (Analysette 22 MicroTec plus, Fritsch GmbH, Idar-Oberstein, Germany).

**Morphological characteristics.** The shape of the powder was examined by a vacuum scanning electron microscope (SEM) (JSM 5410LV, JEOL Ltd., Tokyo, Japan). The dried powder was attached to SEM-stubs using a double-sided carbon tape coated with gold.

**Color and degree of browning.** The color of the powder was measured using a colorimeter (CR-300, Minolta Co., Ltd., Osaka, Japan) and expressed as Hunter *L* (brightness), *a* (red-green component), *b* (yellow-blue component) values. The degree of browning was spectrophotometrically determined. One gram powder was added to the mixture of 40 mL distilled water and 10 mL of 10% trichloroacetic acid solution. The powder solution was kept at  $23\pm3^{\circ}$ C for 2 h and then filtered through a filter paper (Whatman No. 2, Whatman Ltd., Maidston, UK). The absorbance of the filtrate at 420 nm measured by a spectrometer (Optizen 2021UV, Mecasys Co., Ltd.) was recorded as the degree of browning (Lee et al., 2003; Kim et al., 2007).

**Volatile profiles.** Volatile profiles of jujube powder were analyzed by the method of Noh et al. (2005) using an electronic nose system (SMart Nose300, SMart Nose<sup>®</sup>, Marin-Epagnier, Switzerland) installed with an automatic headspace sampler (SMart Nose<sup>®</sup> Autosampler, SMart Nose) and a mass spectrometer (Quadrupole Mass Spectrometer, Balzers Instruments, Marin-Epagnier, Switzerland). The number of channels of the electronic nose system was 191 (bargraph mode, 10–200 amu). The flow rate of 99.999% nitrogen purge gas was 230 mL/min. Powder samples (2 g) were incubated for 15 min at 70°C with an agitation at 30 rpm. Selected 20–30 ion fragments peaks were analyzed by discriminant function analysis (DFA) using statistics software (SMart Nose).

**Moisture sorption isotherm characteristics.** Moisture equilibrium data were obtained by the static gravimetric method using different saturated salt solutions (Spiess and Wolf, 1987). Triplicate samples (2 g each) in 57-mm disposable dishes without lids were placed inside desiccators (24 cm diameter) containing saturated salt solutions with water activity ranging from 0.11 to 0.84 (LiCl, CH<sub>3</sub>COOK, MgCl<sub>2</sub>, K<sub>2</sub>CO<sub>3</sub>, MgNO<sub>3</sub>, NaCl, KCl). The desiccators were placed in an incubator (VS-1203P3N, Vision Scientific Co., Ltd) at 30°C. The moisture content of powder was determined by following the Association of Official Analytical Chemists method (1998) and expressed on a dry basis. Samples were weighed every 24 h until the difference in two consecutive weight measurements was less than 0.0005 g.

Water solubility. Water solubility was determined by the method

of Schoch (1964). Initial solid content of powder was determined by an oven drying method using a hot-air oven dryer (VS-1202D2, Vision Scientific Co., Ltd.) at  $105^{\circ}$ C for 24 h. The powder (0.1 g) was suspended in 10 mL of distilled water with stirring at 120 rpm for 5 min. The suspension was placed in a refrigerator at 5°C for 30 min and then centrifuged at  $500 \times g$  and 4°C for 30 min. The supernatant was poured into a test tube and dried in the hot-air oven dryer at  $105^{\circ}$ C for 24 h. The solid content was measured to determine the water-soluble solid content. The solubility (%) was defined as the ratio of the water-soluble solid content to initial solid content (×100).

**Rehydration.** Rehydration was determined by the method of Medcarf and Gilles (1956). One gram powder was added to 20 mL distilled water and left standing at  $25^{\circ}$ C for 1 h. The mixture was centrifuged at 2,000 × g for 15 min. The weight of precipitate was measured as the weight of rehydrated powder. The degree of rehydration was determined using the following equation;

#### Degree of rehydration =

Weight of rehydrated powder (g)–Initial dried powder weight (g) Initial dried powder weight (g) ×100

**Total sugar content.** Total sugar content was determined by following the method of Sohn et al. (2002). The dried jujube powder (1 g) was dispersed in 10-mL distilled water. One milliliter dispersed solution was added to the mixture of a 1-mL phenol solute in 5 mL  $H_2SO_4$ . The absorbance of the solution was measured at 480 nm using a spectrophotometer (Optizen 2021UV, Mecasys Co., Ltd.), and the sugar content was determined from a calibration curve that was generated with sucrose solutions.

**Polyphenol content.** The amount of total polyphenols was determined using the method of Swain et al. (1959). Nine milliliters of extract from 10 g of the powder was mixed with 1 mL of 0.2 N Folin-Ciocalteu solution. One milliliter of 40% Na<sub>2</sub>CO<sub>3</sub> solution was mixed with the extract solution for 1 h at  $23\pm3^{\circ}$ C. The absorbance of the final solution was measured using a spectrophotometer (Optizen 2021UV, Mecasys Co., Ltd.) at 720 nm.

Statistical analysis. All experiments were performed in triplicate. An ANOVA test (Duncan's multiple range tests) was carried out using SAS (SAS<sup>®</sup> version 9.2, SAS Institute Inc., Raleigh, NC) to estimate the significant difference ( $\alpha$ =0.05). Pearson correlation coefficient, multiple linear regression, and RSA were conducted using SAS. A face-centered cube design (FCD) was used in RSA to measure effects of two individual variables (average powder size and water activity) on dependent variables (water content, water solubility, rehydration, total sugar content, and polyphenol content) of the powder. The complete design consisted of 11 experimental points including three replications of the center points. Regression equations are described in a polynomial expression (Koch, 2005).

# **Results and Discussion**

**Powder size.** Average sizes of dried jujube powder are summarized in Table 1. The sizes ranged from 16.0 to 148.8  $\mu$ m. A normal size distribution was exhibited from all the powder samples (data not shown). Powder size was not different depending on different drying methods (Table 1). The average powder size was closer to the sieve maximum size than the minimum size (Table 1). This may be related to the shape of the powder (Fig. 1). More powders with shorter dimensions could pass the sieve during the shaking operation of sieving.

**Morphological characteristics.** Micrographs of the powder are exhibited in Fig. 1. Dried jujube powder had amorphous structures with some porosity. Milling induced during powder preparation could form biomaterials with an amorphous structure (Zhao, 2009). Morphologies of the powder obtained from different drying methods were not distinguishable (Fig. 1).

Color and degree of browning. The values of Hunter L, a, and b and degree of browning of the jujube powder are displayed in Table 2. The lightness (L value) increased as the size of jujube powder decreased. This phenomenum was also observed in distiller-dried grains with solubles (DDGS), a mix of particulate materials processed from yellow corn, with L varying from 44.9 to 59.6 (Liu, 2008). The powder from 100-150 µm sieve had the highest values of a and degree of browning. The L values of AD and VD powder were higher than those of FD powder (Table 2). The values of a, b, and degree of browning were highest (4.23, 19.78, and 0.23, respectively) in AD powder, probably because AD powder was exposed to more heat energy compared to VD powder or FD powder. Non-enzymatic browning which took place between reducing sugars and amino acids in the powder during heat-induced air-drying could have contributed to the color change of AD powder.

**Volatile profiles.** A DFA plot of the results from the volatile analysis of jujube powder prepared by different drying methods is illustrated in Fig. 2. The result of DFA could be grouped according to the drying methods. The direction of arrow indicates decrease in the amount of volatiles. More volatiles were detected from VD and FD powders. This might be due to less loss of volatiles by the drying methods conducted at relatively low drying temperatures with vacuum processing. The quantity of volatile components was dependent upon the particle size of jujube samples (Fig. 2). The smaller the powder size was, the less volatile components were detected, possibly due to more loss of volatiles of smaller powder owing to its larger surface area to volume ratio.

**Moisture sorption isotherms.** The moisture sorption isotherms plotted with experimental data at the  $a_w$  range of 0.11–0.84 are shown in Fig. 3. The moisture sorption isotherms exhibited a typical Type II sigmoidal shape, which is common in many food materials, including *Chaga* mushroom, *Agaricus bisporus*,

|                  | Sample <sup>1)</sup> |                |                                |                            | Property            |                                   |  |
|------------------|----------------------|----------------|--------------------------------|----------------------------|---------------------|-----------------------------------|--|
| Drying<br>method | Sieve size<br>(µm)   | Water activity | Average<br>powder size<br>(µm) | Water<br>solubility<br>(%) | Rehydration<br>(%)  | Total sugar<br>content<br>(mg/mL) | Polyphenol<br>content<br>(mg/g, dry basis) |
|                  | 100-150              | 0.11           | 147.2 <sup>a</sup>             | 78.6 <sup>a</sup>          | 531.6 <sup>b</sup>  | 8.0 <sup>cd</sup>                 | 2.6 <sup>ab</sup>                          |
|                  |                      | 0.15           | 147.2 <sup>a</sup>             | $76.0^{a}$                 | 544.2 <sup>b</sup>  | 8.1 <sup>bcd</sup>                | 2.7 <sup>a</sup>                           |
|                  |                      | 0.23           | 147.2 <sup>a</sup>             | 78.8 <sup>a</sup>          | 615.5ª              | 7.6 <sup>d</sup>                  | 2.7 <sup>ab</sup>                          |
|                  | 53-100               | 0.11           | 90.3 <sup>b</sup>              | 66.8 <sup>a</sup>          | 465.1 <sup>e</sup>  | 8.6 <sup>abcd</sup>               | 2.6 <sup>ab</sup>                          |
|                  |                      | 0.15           | 90.3 <sup>b</sup>              | 79.5 <sup>a</sup>          | 482.0 <sup>d</sup>  | 8.4 <sup>abcd</sup>               | 2.6 <sup>ab</sup>                          |
|                  |                      | 0.23           | 90.3 <sup>b</sup>              | 81.4 <sup>a</sup>          | 515.1°              | 8.2 <sup>bcd</sup>                | 2.7 <sup>ab</sup>                          |
| Air-drying       | 32-53                | 0.11           | 38.9°                          | 79.5 <sup>a</sup>          | 330.5 <sup>h</sup>  | 8.8 <sup>abc</sup>                | 2.6 <sup>ab</sup>                          |
|                  |                      | 0.15           | 38.9 <sup>c</sup>              | 84.2 <sup>a</sup>          | 349.8 <sup>g</sup>  | 8.6 <sup>abcd</sup>               | 2.5 <sup>b</sup>                           |
|                  |                      | 0.23           | 38.9 <sup>c</sup>              | 80.1 <sup>a</sup>          | 381.9 <sup>f</sup>  | 9.3ª                              | 2.6 <sup>ab</sup>                          |
|                  | ≤32                  | 0.11           | 16.0 <sup>d</sup>              | 76.1ª                      | 297.7 <sup>i</sup>  | 8.9 <sup>abc</sup>                | 2.6 <sup>ab</sup>                          |
|                  |                      | 0.15           | 16.0 <sup>d</sup>              | 81.4 <sup>a</sup>          | 280.7 <sup>j</sup>  | $8.7^{ m abc}$                    | 2.6 <sup>ab</sup>                          |
|                  |                      | 0.23           | 16.0 <sup>d</sup>              | 93.4ª                      | 342.3 <sup>gh</sup> | 9.2 <sup>ab</sup>                 | 2.6 <sup>ab</sup>                          |
|                  | 100-150              | 0.11           | 145.7 <sup>a</sup>             | 73.7ª                      | 500.0 <sup>ab</sup> | 8.0 <sup>d</sup>                  | 2.2 <sup>g</sup>                           |
|                  |                      | 0.15           | 145.7 <sup>a</sup>             | 74.8 <sup>a</sup>          | 489.7 <sup>bc</sup> | 9.0 <sup>abc</sup>                | 2.3 <sup>f</sup>                           |
|                  |                      | 0.23           | 145.7 <sup>a</sup>             | 76.5 <sup>a</sup>          | 508.7 <sup>a</sup>  | 8.9 <sup>abc</sup>                | 2.5 <sup>abc</sup>                         |
|                  | 53-100               | 0.11           | 92.1 <sup>b</sup>              | 75.1ª                      | 481.8 <sup>c</sup>  | 8.1 <sup>cd</sup>                 | 2.3 <sup>ef</sup>                          |
|                  |                      | 0.15           | 92.1 <sup>b</sup>              | 76.6 <sup>a</sup>          | 459.7 <sup>d</sup>  | 8.7 <sup>bcd</sup>                | 2.3 <sup>ef</sup>                          |
|                  |                      | 0.23           | 92.1 <sup>b</sup>              | 78.6 <sup>a</sup>          | 458.0 <sup>d</sup>  | 9.3 <sup>ab</sup>                 | 2.6 <sup>a</sup>                           |
| Vacuum-drying    | 32-53                | 0.11           | 48.9 <sup>c</sup>              | 74.7 <sup>a</sup>          | 391.4 <sup>e</sup>  | 8.4 <sup>bcd</sup>                | 2.5 <sup>bcd</sup>                         |
|                  |                      | 0.15           | 48.9 <sup>c</sup>              | 78.4 <sup>a</sup>          | 334.4 <sup>f</sup>  | 8.9 <sup>abc</sup>                | 2.4 <sup>de</sup>                          |
|                  |                      | 0.23           | 48.9 <sup>c</sup>              | 78.5 <sup>a</sup>          | 319.1 <sup>f</sup>  | 9.1 <sup>ab</sup>                 | 2.6 <sup>ab</sup>                          |
|                  | ≤32                  | 0.11           | 18.1 <sup>d</sup>              | 73.8 <sup>a</sup>          | 264.3 <sup>g</sup>  | 9.8ª                              | 2.5 <sup>bcd</sup>                         |
|                  |                      | 0.15           | 18.1 <sup>d</sup>              | 74.8 <sup>a</sup>          | 249.0 <sup>gh</sup> | 8.4 <sup>bcd</sup>                | 2.5 <sup>cd</sup>                          |
|                  |                      | 0.23           | 18.1 <sup>d</sup>              | 76.8 <sup>a</sup>          | 241.5 <sup>h</sup>  | 8.9 <sup>abcd</sup>               | 2.6 <sup>abc</sup>                         |
|                  | 100-150              | 0.11           | 148.8 <sup>a</sup>             | 76.2 <sup>e</sup>          | 407.9 <sup>d</sup>  | 8.0 <sup>b</sup>                  | 2.5 <sup>ab</sup>                          |
|                  |                      | 0.15           | 148.8 <sup>a</sup>             | 76.9 <sup>e</sup>          | 527.4 <sup>a</sup>  | 9.1 <sup>ab</sup>                 | 2.0 <sup>e</sup>                           |
|                  |                      | 0.23           | 148.8 <sup>a</sup>             | 78.2 <sup>de</sup>         | 449.3°              | 8.6 <sup>b</sup>                  | 2.5 <sup>ab</sup>                          |
|                  | 53-100               | 0.11           | 92.7 <sup>b</sup>              | 80.8 <sup>abcd</sup>       | 388.2 <sup>e</sup>  | 8.6 <sup>b</sup>                  | 2.5 <sup>ab</sup>                          |
|                  |                      | 0.15           | 92.7 <sup>b</sup>              | 81.1 <sup>abcd</sup>       | 475.1 <sup>b</sup>  | 9.9 <sup>a</sup>                  | 2.3 <sup>d</sup>                           |
|                  |                      | 0.23           | 92.7 <sup>b</sup>              | 82.5 <sup>ab</sup>         | 388.8 <sup>e</sup>  | 8.5 <sup>b</sup>                  | 2.6 <sup>a</sup>                           |
| Freeze-drying    | 32-53                | 0.11           | 45.6 <sup>c</sup>              | 79.3 <sup>bcde</sup>       | 319.7 <sup>g</sup>  | 8.2 <sup>b</sup>                  | 2.4 <sup>bc</sup>                          |
|                  |                      | 0.15           | 45.6 <sup>c</sup>              | 84.7 <sup>a</sup>          | 358.3 <sup>f</sup>  | 9.9 <sup>a</sup>                  | 2.3 <sup>d</sup>                           |
|                  |                      | 0.23           | 45.6 <sup>c</sup>              | 81.1 <sup>abcd</sup>       | 364.1 <sup>f</sup>  | 8.5 <sup>b</sup>                  | 2.3 <sup>cd</sup>                          |
|                  | ≤32                  | 0.11           | 17.6 <sup>d</sup>              | 78.7 <sup>cde</sup>        | 239.1 <sup>i</sup>  | 8.8 <sup>b</sup>                  | 2.6 <sup>a</sup>                           |
|                  |                      | 0.15           | 17.6 <sup>d</sup>              | 80.7 <sup>abcd</sup>       | 270.8 <sup>h</sup>  | 8.7 <sup>b</sup>                  | 2.6 <sup>a</sup>                           |
|                  |                      | 0.23           | 17.6 <sup>d</sup>              | 81.9 <sup>abc</sup>        | 245.9 <sup>i</sup>  | 8.8 <sup>b</sup>                  | 2.4 <sup>bc</sup>                          |

Table 1. Physical and chemical properties of jujube powder with different powder sizes produced by different drying methods<sup>A</sup>

<sup>a-j</sup>Values followed by the same alphabet in the same column are not significantly different (p > 0.05).

*Pleurotus florida*, tapioca, and fufu (Sanni et al., 1997; Shivhare et al., 2004; Lee and Lee, 2007). Similar moisture contents were obtained among the powders at the same level of water activity regardless of powder size (Fig. 3). This phenomenon was exhibited in AD, VD, and FD powders.

**Water solubility.** The water solubility values of the jujube powder obtained by different drying methods are exhibited in Table 1. The solubility of the powder ranged from 66.8 to 93.4%, similar to the reported values of onion powder (Kim et al., 2007). The solubility of the jujube powder was not significantly affected by different

drying methods or powder sizes (p > 0.05). The water solubility is one of the most important properties characterizing instant powder foods (Picesky, 1986; Shittu and Lawal, 2007). The water solubility of jujube powder (Table 1), which is comparable to that of cocoa powder (44.2–76.6%) (Shittu and Lawal, 2007), showed that the powder could be used in tea or beverage products. Although the solubility was thought to be correlated to the content of sugar, highly soluble in water, but no significant influence was observed (Table 1), which is consistent with the results observed with cocoa powder (Shittu and Lawal, 2007).

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D Air-dried Jujube powedr (×1000)



E Vacuum-dried Jujube powedr (×1000)



F Freeze-dried Jujube powder (×1000)

Fig. 1 Scanning electron micrographs of jujube powder obtained from the sieve of 100-150 µm mesh after air-, vacuum-, and freeze-drying.

| Table 2. | Color | properties | of dried | jujube | powder with | n different | sizes | produced b | v different | drying | methodsA, B |
|----------|-------|------------|----------|--------|-------------|-------------|-------|------------|-------------|--------|-------------|
|          |       |            |          |        |             |             |       |            |             |        |             |

|                  |                    | -                  |                    |                    |                    |
|------------------|--------------------|--------------------|--------------------|--------------------|--------------------|
| Sieve size       | L                  | Air-drying         | Vacuum-drying      | Freeze-drying      | DGM                |
| 100–150 μm       |                    | 44.92±0.83         | 46.38±1.20         | 45.76±0.26         | 45.69 <sup>d</sup> |
| 53–100 μm        |                    | 50.90±1.19         | 52.71±1.49         | 49.72±1.05         | 51.11°             |
| 32–53 μm         |                    | 55.63±0.41         | 54.75±1.04         | 52.85±0.61         | 54.41 <sup>b</sup> |
| ≤32 μm           |                    | 59.39±0.58         | 57.49±2.03         | 53.75±0.69         | 56.88 <sup>a</sup> |
| DGM <sup>C</sup> |                    | 52.71 <sup>a</sup> | 52.83 <sup>a</sup> | 50.52 <sup>b</sup> |                    |
|                  | а                  | Air-drying         | Vacuum-drying      | Freeze-drying      | DGM                |
| 100–150 μm       |                    | 4.70±0.25          | 3.82±0.18          | 4.37±0.21          | 4.30 <sup>a</sup>  |
| 53–100 μm        |                    | 4.24±0.27          | 3.36±0.26          | 3.59±0.17          | 3.73 <sup>b</sup>  |
| 32–53 μm         |                    | 4.15±0.02          | 3.33±0.25          | 3.19±0.06          | 3.56 <sup>b</sup>  |
| ≤32 μm           |                    | 3.82±0.09          | 3.07±0.15          | 3.94±0.35          | 3.61 <sup>b</sup>  |
| DGM              |                    | 4.23 <sup>a</sup>  | 3.40 <sup>c</sup>  | 3.77 <sup>b</sup>  |                    |
|                  | b                  | Air-drying         | Vacuum-drying      | Freeze-drying      | DGM                |
| 100–150 μm       |                    | 17.90±0.44         | 17.99±0.43         | 18.71±0.14         | 18.20 <sup>b</sup> |
| 53–100 μm        |                    | 19.88±0.65         | 19.82±0.68         | 19.23±0.35         | 19.64 <sup>a</sup> |
| 32–53 μm         |                    | 20.96±0.47         | 19.51±0.49         | 19.49±0.25         | 19.99 <sup>a</sup> |
| ≤32 μm           |                    | 20.38±0.19         | 19.33±0.41         | 19.15±0.57         | 19.62 <sup>a</sup> |
| DGM              |                    | 19.78 <sup>a</sup> | 19.16 <sup>b</sup> | 19.14 <sup>b</sup> |                    |
|                  | Degree of browning | Air-drying         | Vacuum-drying      | Freeze-drying      | DGM                |
| 100–150 μm       |                    | 0.23±0             | 0.22±0             | 0.20±0             | 0.22 <sup>ef</sup> |
| 53–100 μm        |                    | 0.24±0             | 0.22±0             | 0.20±0             | 0.22 <sup>e</sup>  |
| 32–53 μm         |                    | 0.23±0             | 0.21±0             | 0.20±0             | 0.22 <sup>ef</sup> |
| ≤32 μm           |                    | 0.22±0             | 0.21±0             | 0.20±0             | $0.20^{f}$         |
| DGM              |                    | 0.23 <sup>f</sup>  | $0.22^{\rm f}$     | 0.19 <sup>e</sup>  |                    |
|                  |                    |                    |                    |                    |                    |

<sup>a-d</sup>Values followed by the same alphabet in the same column or row are not significantly different (p > 0.01) by Duncan's two-way ANOVA test. <sup>e-f</sup>Values followed by the same alphabet in the same column or row are not significantly different (p > 0.05) by Duncan's two-way ANOVA test. DGM, Duncan grouping mean. DF2

Fig. 2 A discriminant function analysis (DFA) plot of the results from the jujube powder analysis using the mass spectrometer-based electronic nose. •: air-dried jujube powder passed through the sieve of 32 µm

the jujube powder analysis using the mass spectrometer-based electronic nose.  $\bigcirc$ : air-dried jujube powder passed through the sieve of 32 µm mesh,  $\blacktriangle$ : air-dried jujube powder passed through the sieve of 32–53 µm mesh,  $\blacksquare$ : air-dried jujube powder passed through the sieve of 53– 100 µm mesh,  $\diamondsuit$ : air-dried jujube powder passed through the sieve of 53– 100 µm mesh,  $\diamondsuit$ : air-dried jujube powder passed through the sieve of 100–150 µm mesh,  $\bigcirc$ : vacuum-dried jujube powder passed through the sieve of 32 µm mesh,  $\bigcirc$ : vacuum-dried jujube powder passed through the sieve of 32–53 µm mesh,  $\bigcirc$ : vacuum-dried jujube powder passed through the sieve of 53–100 µm mesh,  $\diamondsuit$ : freeze-dried jujube powder through the sieve of 32 µm mesh,  $\blacksquare$ : freeze-dried jujube powder through the sieve of 32–53 µm mesh,  $\blacksquare$ : freeze-dried jujube powder through the sieve of 53–100 µm mesh,  $\clubsuit$ : freeze-dried jujube powder through the sieve of 52–53 µm mesh,  $\blacksquare$ : freeze-dried jujube powder through the sieve of 53–100 µm mesh,  $\clubsuit$ : freeze-dried jujube

Rehydration. The results from the rehydration of powder obtained by different drying methods are shown in Table 1. Larger-sized powder had higher values of rehydration at each level of water activity, irrespective of the drying method used (p < 0.05). The rehydration values of AD powder were higher (280.7-615.5%) than those of VD (241.5-508.7%) and FD (239.1–527.4) powders. This is consistent with the result observed with onion powder in that AD onion powder had a higher value for rehydration than VD and FD onion powders (Kim et al., 2007). However, the order of the drying methods resulting in a higher rehydration was not the same and appeared to differ depending on food sample (Ratti, 2001). This discrepancy could be because rehydration is influenced by many factors, including porosity, capillaries, cavities (air), amorphous-crystalline state, dryness, and pH of soaking water (Lewicki, 1998; Rahman and Perera, 1999).

**Total sugar content.** The range of total sugar content of all kinds of jujube powder was 8.0–9.9 mg/mL (Table 1). The total sugar content did not differ by different drying methods, water activities, and powder sizes.

**Polyphenol content.** The content of polyphenol of the powder is shown in Table 1. The polyphenol content was not significantly affected by average powder size and water activity (p > 0.05), but was affected by drying method (p < 0.05). The polyphenol



Fig. 3 Moisture sorption isotherms of air- (Fig. 3A), vacuum- (Fig. 3B), and freeze- (Fig. 3C) dried jujube powder obtained through sieves of different mesh sizes.

contents of AD, VD, and FD powders were 2.5–2.7, 2.2–2.6, and 2.0–2.6 mg/g, respectively. The AD powder had the highest polyphenol content (p < 0.05), possibly because heating process could break down covalent bonds of polyphenols, thereby liberating smaller molecules of polyphenols and forming more



| Table 3 | Pearson correlation coefficients between the physicochemical properties of jujube powder |
|---------|--|

| Drying<br>method | Property                    | Average<br>powder size<br>(µm) | Water<br>solubility<br>(%) | Rehydration<br>(%) | Total sugar<br>content<br>(mg/mL) | Polyphenol<br>content<br>(mg/g) |
|------------------|-----------------------------|--------------------------------|----------------------------|--------------------|-----------------------------------|---------------------------------|
|                  | Average powder size (µm)    | 1.000                          |                            |                    |                                   |                                 |
|                  | Water solubility (%)        | -0.403                         | 1.000                      |                    |                                   |                                 |
| Air-drying       | Rehydration (%)             | $0.954^{B}$                    | -0.317                     | 1.000              |                                   |                                 |
|                  | Total sugar content (mg/mL) | $-0.874^{B}$                   | 0.282                      | $-0.828^{\circ}$   | 1.000                             |                                 |
|                  | Polyphenol content (mg/g)   | 0.698 <sup>A</sup>             | -0.286                     | 0.680 <sup>A</sup> | -0.518                            | 1.000                           |
|                  | Average powder size (µm)    | 1.000                          |                            |                    |                                   |                                 |
|                  | Water solubility (%)        | -0.125                         | 1.000                      |                    |                                   |                                 |
| Vacuum-drying    | Rehydration (%)             | 0.933 <sup>B</sup>             | -0.082                     | 1.000              |                                   |                                 |
|                  | Total sugar content (mg/mL) | -0.276                         | 0.362                      | -0.334             | 1.000                             |                                 |
|                  | Polyphenol content (mg/g)   | -0.541                         | 0.568                      | -0.505             | 0.613 <sup>A</sup>                | 1.000                           |
| Freeze-drying    | Average powder size (im)    | 1.000                          |                            |                    |                                   |                                 |
|                  | Water solubility (%)        | $-0.592^{A}$                   | 1.000                      |                    |                                   |                                 |
|                  | Rehydration (%)             | 0.871 <sup>B</sup>             | -0.325                     | 1.000              |                                   |                                 |
|                  | Total sugar content (mg/mL) | -0.126                         | 0.498                      | 0.236              | 1.000                             |                                 |
|                  | Polyphenol content (mg/g)   | -0.285                         | 0.067                      | $-0.608^{A}$       | -0.533                            | 1.000                           |

<sup>A</sup>Correlation significant at p < 0.05.

<sup>B</sup>Correlation significant at p < 0.001.

500

400

300

0.15

Rehydration (%)

| Drying method | Properties       | Independent variables | Coefficient        | $\mathbb{R}^2$      |
|---------------|------------------|-----------------------|--------------------|---------------------|
|               | Water solubility | Constant              | 98.82              | 0.24                |
|               |                  | Average powder size   | -0.12              |                     |
|               |                  | Rehydration           | 0.04               |                     |
|               |                  | Total sugar content   | -4.14              |                     |
| A in Amrina   |                  | Polyphenol content    | 3.28               |                     |
| Air-drying    | Rehydration      | Constant              | -27.93             | 0.92 <sup>A</sup>   |
|               |                  | Average powder size   | 1.55 <sup>A</sup>  |                     |
|               |                  | Water solubility      | 1.53               |                     |
|               |                  | Total sugar content   | 9.34               |                     |
|               |                  | Polyphenol content    | 46.69              |                     |
|               | Water solubility | Constant              | 51.35 <sup>A</sup> | 0.38                |
|               |                  | Average powder size   | -0.00              |                     |
|               |                  | Rehydration           | 0.01               |                     |
|               |                  | Total sugar content   | 0.13               |                     |
| ¥7            |                  | Polyphenol content    | 8.85               |                     |
| vacuum-arying | Rehydration      | Constant              | 151.90             | $0.88^{\mathrm{A}}$ |
|               |                  | Average powder size   | 1.48 <sup>A</sup>  |                     |
|               |                  | Water solubility      | 3.22               |                     |
|               |                  | Total sugar content   | -25.06             |                     |
|               |                  | Polyphenol content    | 31.16              |                     |
|               | Water solubility | Constant              | 42.36              | 0.65                |
|               |                  | Average powder size   | -0.04              |                     |
|               |                  | Rehydration           | 0.03               |                     |
|               |                  | Total sugar content   | 1.45               |                     |
| Enter dation  |                  | Polyphenol content    | 7.98               |                     |
| Freeze-drying | Rehydration      | Constant              | 8.43               | 0.94 <sup>A</sup>   |
|               |                  | Average powder size   | 1.19 <sup>A</sup>  |                     |
|               |                  | Water solubility      | 7.18               |                     |
|               |                  | Total sugar content   | 13.62              |                     |
|               |                  | Polyphenol content    | -184.74            |                     |

Table 4 Multiple linear regression models predicting the physicochemical properties of jujube powder

<sup>A</sup>Regression significant at p < 0.001.

moles of polyphenols (Jeong et al., 2004). Phenolic compounds formed by non-enzymatic browning in AD also could increase the polyphenol content, because the polyphenol-determining method using the Folin-Ciocalteu reagent can response to phenolic compounds (Hong et al., 2011). Similar results of polyphenol content were observed from drying studies with tangerine epicarp and onion (Jeong et al., 2004; Kim et al., 2007).

**Correlations among the properties.** Pearson correlation, multiple linear regression, and response surface analysis were applied to the property data to obtain correlations among the properties. The results from Pearson correlation coefficient analysis indicated that the level of rehydration and the average powder sizes had positive correlations (AD: r=0.954, p < 0.001; VD: r=0.933, p < 0.001; FD: r=0.871, p < 0.001) (Table 3).

Multiple linear regression analysis was conducted to determine which variables (water solubility, total sugar content, and polyphenol content) are significantly affected by water solubility or rehydration. Results from the multiple linear regression analysis are summarized in Table 4. In AD powder, rehydration was more strongly related to the other properties ( $R^2$ =0.92) than water solubility ( $R^2$ =0.24) (Table 4). A fairly strong correlation between rehydration and average powder size was shown with VD powder ( $R^2$ =0.88). Water solubility was not significantly related to any of the properties of VD powder (p >0.05) (Table 4). In FD powder, both water solubility and rehydration showed correlations with the other properties ( $R^2$ =0.65 and 0.94, respectively) (Table 4). Furthermore, rehydration showed significant correlation to average powder size in AD, VD, and FD powders (p <0.001).

The models obtained from RSA for AD, VD, and FD powders with respect to rehydration (y) were  $y=311.000329+2.425031x_1-9.46587x_2-0.005886x_1x_1+0.016618x_2x_1+0.373288x_2x_2$ ,  $y=329.140722+3.458194x_1-15.221496x_2-0.010959x_1x_1+0.015569x_2x_1+0.368428x_2x_2$ ,  $y=-438.154407+2.586081x_1+82.547066x_2-0.007215x_1x_1+0.007834x_2x_1-2.412708x_2x_2$ , respectively. Rehydration was highly correlated with the independent variables of average powder size (x<sub>1</sub>) and water activity (x<sub>2</sub>). The rehydration, average particles size, and water activity were well explained by the models ( $R^2$ =0.98-0.99, p < 0.001). Response surfaces generated

with rehydration, powder size, and water activity of air-, vacuum-, and freeze-dried jujube powders are illustrated in Fig. 4. The correlations among the properties indicate rehydration of the powder could be maximized by controlling particle size and water activity of the powder. The correlations determined herein could be used to improve the property of interest by controlling other properties that significantly affect the property of interest.

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