

Effects of Different Drying Methods on Physico-Chemical Properties of *Dendrobium Officinale*

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Abstract—*Dendrobium officinale* has been used in China for several thousand years as a health food and a source of nutrients. The aim of this study was to investigate the effects of different drying methods on the characterization of *D. officinale*. The physico-chemical properties, i.e. color retention, shrinkage rate, functional components contents, moisture sorption isotherms and FTIR spectrum of *D. officinale* samples through sun drying (SD), oven drying (OD), vacuum drying (VD), infrared radiation drying (ID) and freeze drying (FD) were investigated. The results revealed that different drying methods showed significant difference on color retention, shrinkage rate and water soluble polysaccharides content of *D. officinale*, but not total dendrobine or protein content. As can be seen from the FTIR spectra, no new chemical bonds were produced between different dried *D. officinale* samples. In the same water activity, the moisture content in freeze dried samples at equilibrium was slightly higher compared with other four samples. FD samples presented the best quality in color and shrinkage protection, but with respect to the water-soluble polysaccharides content for *D. officinale*, OD, VD and ID samples were better than FD samples, and OD ones was the highest. The results showed that the quality of dried *D. officinale* depends on the drying method and conditions. Considering the retention of polysaccharide content, OD is considered promising for drying *D. officinale*.

Index Terms—*Dendrobium officinale*, Different drying methods, Physico-chemical properties

I. INTRODUCTION

Dendrobium officinale Kimura et Migo is a precious herbal plant in Traditional Chinese Medicine, which are mainly distributed in the subtropical and tropical regions of China [1, 2]. Since ancient times, many *Dendrobium* plants have been used as ingredients for traditional medicines, nutraceutical beverages and health-promoting food products [3]. Among all the *Dendrobium* herbs in China, *D. officinale* stems and leaves have been believed by traditional medical practitioners to have the best medical properties for the treatment of throat inflammation, fever and chronic superficial gastritis or as a tonic for promoting the production of body fluid and improving the quality of life [4, 5]. The previous research

has proved that polysaccharides and alkaloids were major active constituents of *Dendrobium* herbs [6]. As for the polysaccharides, many studies have demonstrated their pharmacological activities including antioxidant, immune stimulating, antitumor activities, and neuro-protective effect [7-12] and anti-inflammatory as well as anti-A β injury for alkaloids [4].

The very convenient and commonly used method to preserve fresh harvest plant tissues is to dry them to avoid spoilage and deterioration, extend their shelf life and reduce storage volume [13, 14]. Several drying methods are commercially available. Freeze drying (FD) a relatively new process of drying, ensures the preservation of all thermolabile compounds in the initial raw material, and gives the highest product quality, but its relatively high production cost is the major disadvantage [15]. Vacuum drying (VD) is performed under low pressure and makes the best use of the fact that the boiling point of water is reduced as the pressure is reduced [15-17]. Relatively commonly used oven drying (OD) is not as efficient and use more energy, the longer drying time and higher temperatures which needed usually result in inferior product quality. Sun drying (SD) is the evaporation of water from products by sun or solar heat, assisted by the movement of surrounding air, this method is relatively slow, because the sun does not cause rapid evaporation of moisture [14, 15]. Infrared radiation drying (ID) has been widely applied in recent years, and its shorter drying time, a better final dried product quality, and more energy savings in the process are revealed as the most important advantages [18, 19]. Many authors have studied quality of dried products produced by freeze drying, vacuum drying, oven drying, infrared radiation drying, and sun drying [13, 18, 20]. The duration and temperature of the drying process are the most important factors affecting the quality of dehydrated products, and the selection of the optimal method is determined by quality requirements, raw material characteristics and economic factors [15].

Physico-chemical properties of the powder are closely related to processing, package, storage, transportation, mouthfeel, and functional properties [21]. The micronization of various materials by superfine or nanotechnology to micro- or nano-sizes alters their

surface properties and functional properties and results in new applications [22, 23].

To the best of our knowledge, the characteristics of *D. officinale* prepared by different drying methods have not been reported thus far. In this paper, the aim of this study was to compare some physico-chemical properties of dried *D. officinale* produced by different drying methods, e.g., sun drying (SD), oven drying (OD), vacuum drying (VD), infrared radiation drying (ID) and freeze drying (FD).

II. MATERIALS AND METHODS

A. Raw Material

Fresh *D. officinale* used in this study was purchased from Jiangsu Hongjingtang Biological Technology Co., LTD (Jiangsu Province, China). The samples were washed with tap water, and then the excess water on the surface of *D. officinale* was removed with filter paper. Afterwards, *D. officinale* was cut into small parts (about 1 cm length) for further drying. Drying methods were as follows.

B. Drying Methods

Sun drying (SD): 150 g sample, under the sun.

Oven drying (OD): batch tray dryer (ED 240, Binder GmbH, Germany), 150 g sample, drying temperature 60 ± 1 °C, air velocity 1.5 m/s.

Vacuum drying (VD): vacuum oven (VD 53, Binder GmbH, Germany), 150 g sample, drying temperature 60 ± 1 °C, at pressure of 100 mbar.

Infrared radiation drying (ID): Infrared radiation dryer (Taizhou Senttech Infrared technology Co., Ltd, Taizhou, China), 150 g sample, drying temperature 60 ± 1 °C, air velocity 1.5 m/s.

Freeze drying (FD): Alpha 1-2 LD plus freeze dryer (Christ, Saxony, Germany), cold trap temperature of -55 °C, freezing temperature -50 °C, at pressure of 0.3 mbar.

Drying was continued until the mass of the sample reached to the lowest equilibrium moisture content (EMC). EMC of samples were checked using a gravimetric method at 105 °C [24]. Dried samples at EMC were ground into a fine powder with the particle size in the range of 150-450 µm.

C. Average Shrinkage Rate

The length and diameter of a minimum of 20 *D. officinale* parts were measured before and after each drying treatment to determine the average shrinkage rate, as follows:

$$d_0 (\%) = \frac{d_1 - d_2}{d_1} \times 100\%. \quad (1)$$

where d_1 was the average length or the diameter of *D. officinale* parts before drying; d_2 was the average length or the diameter of *D. officinale* parts after drying. All measurements were performed in triplicate.

D. Colour Retention

The colour of *D. officinale* powder samples was

measured using a Hunter Lab UltraScan Pro1166 0.1 nm optical resolution at wavelength in the range of 350-1050 nm. EasyMatch QC software (Hunter Associate Laboratory Inc., Reston, USA) is attached to the equipment. The UltraScan Pro measures the reflected colour of food products, measured colour represented by Hunter's colour value. L^* , a^* and b^* values indicate lightness, redness (+)/(-) greenness and yellowness (+)/(-)blueness [25], respectively. Total colour differential (ΔE) was also calculated as follows:

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

E. Total Protein Content

The total protein content of different dried *D. officinale* was evaluated by nitrogen determination using the Kjeldahl procedure, by a SH220 Graphite Digester (Jinan Hanon Instruments Co., Ltd, Jinan, China) and K9840 Kjeldahl Distillation Unit (Jinan Hanon Instruments Co., Ltd, Jinan, China). The protein content was then calculated as % N \times 6.25. All the chemicals and solvents for the Kjeldahl method were obtained from Sinopharm Chemical Reagent Co., Ltd (Shanghai, China). All the samples were analyzed in triplicate.

F. Water Soluble Polysaccharides Content

The total water soluble polysaccharides content was determined based on the color reaction of polysaccharides and their derivatives with phenol and concentrated sulfuric acid [26]. The powders (0.5 g) of *D. officinale* were defatted twice with petroleum ether in Soxhlet extractor. Then the dregs were successively extracted thrice with the boiling distilled-water (50 mL), each time for 0.5 h. After removal of the solid powder by vacuum filtration, the supernatant were dried at 50 °C under vacuum in a rotary evaporator (RV06-ML, IKA, Germany) until all water was vaporized. The polysaccharides were then washed with 80% ethanol, which was vaporized again in the rotary evaporator at 45 °C. The residue was then dissolved with distilled water to form a solution. The solution was then transferred to a 250 mL flask, which was diluted to 250 mL with distilled water. One milliliter of the solution was pipetted into a 10 mL centrifuge tube and 1 mL of 5% phenol was added. After shaking for 2 min, 5 mL of concentrated sulfuric acid (H_2SO_4) (98% v/v) was added to the solution and shaken for another 5 min. The mixture was shook well and put into boiling water bath for 20 min and ice bath for 5 min in order. The concentration of water-soluble polysaccharides in the solution was determined quantitatively by measuring the absorbance at 490 nm using a spectrophotometer (Unico 2802 UV/VIS Spectrophotometer, Shanghai, China).

The standard curve for quantitative analysis of total water-soluble polysaccharides content in different dried samples was plotted using anhydrous dextrose (Drying at 105 °C to achieve constant weight) (Sinopharm Chemical Reagent Co., Ltd, Shanghai, China) as a standard solution and distilled water was used as blank solution. The standard curve is shown in Fig. 1A.

G. Total Dendrobine Content

The powders (0.5 g) of *D. officinale* were infiltrated with 2 mL ammonium hydroxide and sealed for 30 min. Then the mixture were extracted with 10 mL chloroform at 80 °C for 2 h. After removal of the solid powder by filtration, 2 mL of the filtrate was pipetted into a 60 mL separating funnel and 5 mL of 0.2 M potassium biphthalate (Adjusted the pH to 4.5 with NaOH) and 0.04% bromocresol green were added. The mixture was fully shook and then kept still for 30 min. After the chloroform layer was filtered through degreasing cotton, Six milliliters of the filtrate was pipetted into a 10 mL centrifuge tube and 1 mL of 0.1 N Sodium hydroxide anhydrous ethanol solution was added and shook well. The concentration of total dendrobine content in the solution was determined quantitatively by measuring the absorbance at 620 nm using a spectrophotometer (Unico 2802 UV/VIS Spectrophotometer, Shanghai, China).

The standard curve for quantitative analysis of total dendrobine content in the dried samples was plotted using dendrobine (98.87%, National Institutes for Food and Drug Control, Beijing, China) as a standard solution and chloroform was used as blank solution. The standard curve is shown in Fig. 1B.

H. FTIR Analysis

The different *D. officinale* powders were prepared with potassium bromide (KBr) pellet method. FTIR spectra from 400 to 4000 cm^{-1} were collected by a Nicolet IS10 Fourier transform infrared spectrophotometer (Thermo Nicolet Corporation, USA). Five replicated spectra were collected for every sample. The background spectrum was obtained against the KBr.

I. Test Procedure for Moisture Sorption Isotherm

Moisture sorption isotherm was determined according to the method of [27] with some minor modifications. The moisture contents of powder samples were determined by drying in an oven at 105 °C [24]. The equilibrium moisture content of the powders was determined using a gravimetric technique by Conway dish method. Saturated salt solutions of NaOH (a_w 0.070), MgCl_2 (a_w 0.330), $\text{Mg}(\text{NO}_3)_2$ (a_w 0.528), NaCl (a_w 0.757), KBr (a_w 0.807), KCl (a_w 0.842), BaCl_2 (a_w 0.901) and $\text{K}_2\text{Cr}_2\text{O}_7$ (a_w 0.986) were used in outer layer of the Conway dish. *D. officinale* powders of 40 mesh (0.5 g) with different drying methods were accurately weighted into a weighing bottle and put inside the inner layer of the

Conway dish which was firmly sealed and kept at 25 °C. Sample were weighed every 24 h until the equilibrium was achieved as indicated by the difference of two consecutive weights less than ± 0.0005 g. Equilibration moisture of each sample was determined by the vacuum oven method [24]. All determinations were performed in duplicate.

J. Statistical Analysis

All experiments were conducted in triplicate, and the results are expressed as the mean \pm standard deviation. Data were analyzed by analysis of variance (ANOVA) and Duncan's multiple range comparison test using SPSS ver. 17.0 software (SPSS, Inc., Chicago, IL, USA). P -values < 0.05 were considered significant.

III. RESULTS AND DISCUSSION

A. The Equilibrium Moisture Content

The equilibrium moisture content (EMC) of dried samples with desired moisture content of less than 0.1 g/g dry basis are listed in Table I. EMC of the samples were in the range of 0.036–0.095 g/g dry basis. As can be seen from Table I, ID samples required shorter drying time than other samples, this phenomenon could be due to the fact that when infrared radiation was used to dry moist materials, the energy of radiation impinges on and penetrates into the materials and then converted into heat without heating the surrounding air [19].

B. Shrinkage Rate

The average shrinkage rate of dried *D. officinale* are presented in Table I. It is clear to observe from Table I that samples dried with FD method presented less tissue shrinkage or collapse as well as basically clear appearance, which suggested that parenchyma cells of FD dried samples presented in regular structure and most of them kept integral without fracture and damage. This might be attributed to the fact that freeze drying works by freezing the material and then reducing the surrounding pressure to allow the frozen water in the material to sublimate directly from the solid phase to the gas phase [28]. Huang et al. (2012) reported that FD could maintain the original cell structure of samples very well [29]. Zheng et al. (2013) found that air dried bamboo shoot slices suffered a severe cell breakage, while FD enabled to obtain dried samples with spongy texture [25].

TABLE I. A PARENT AND EMC, DRYING TIME, SHRINKAGE RATE, AND PROTEIN CONTENT OF *D. OFFICINALE* OF DIFFERENT DRYING METHODS.^{1,2}

sample	EMC (g/g dry bass)	Drying time (h)	Length shrinkage rate (%)	Diameter shrinkage rate (%)	Protein Content (%)
SD	0.095 \pm 0.028 ^a	> 48	2.29 \pm 0.31 ^c	54.61 \pm 0.28 ^b	3.02 \pm 0.05 ^a
OD	0.054 \pm 0.004 ^b	20	9.27 \pm 0.27 ^a	62.96 \pm 0.51 ^a	2.98 \pm 0.02 ^a
VD	0.043 \pm 0.005 ^c	41	8.70 \pm 0.59 ^a	69.74 \pm 0.33 ^a	3.18 \pm 0.01 ^a
ID	0.041 \pm 0.017 ^c	16	5.80 \pm 0.12 ^b	68.59 \pm 0.14 ^a	2.97 \pm 0.05 ^a
FD	0.036 \pm 0.039 ^d	35	4.57 \pm 0.34 ^c	25.49 \pm 0.21 ^c	3.10 \pm 0.07 ^a

¹EMC: equilibrium moisture content.

²Values are means \pm standard deviation of three replications.

³Means values in the same column with different letters are significantly different ($p < 0.05$).

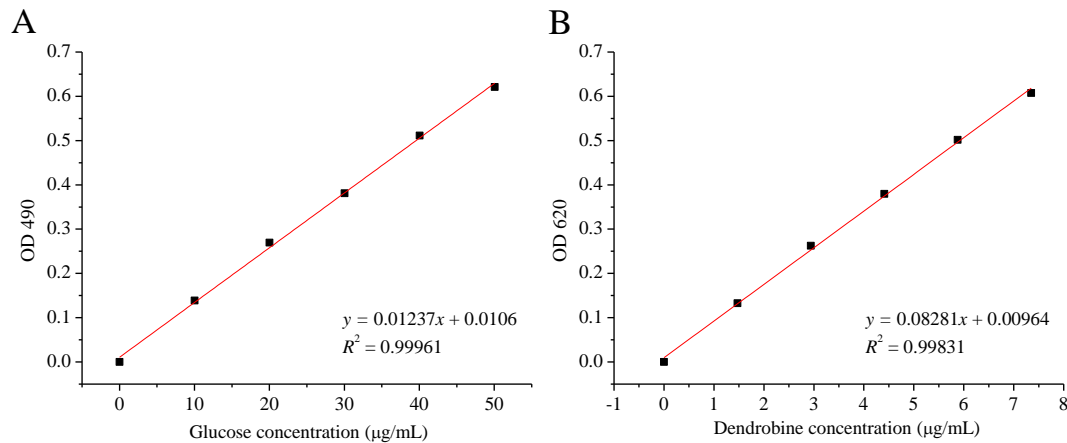


Figure 1. Profile of the standard curves of dextrose and dendrobine. (A) standard curves of dextrose. (B) standard curves of dendrobine.

C. Colour Retention

The colour parameters change of *D. officinale* powders with different drying methods are presented in Table II. Significant changes ($p < 0.05$) were observed in L^* , a^* and b^* values of dried samples compared with each other, suggested that the colour of dried *D. officinale* samples was significantly affected by drying methods. Among the five dried samples, FD *D. officinale* powder showed the highest L^* and lowest a^* and b^* values compared to powders dried by the other four methods. This could be due to the fact that SD and OD samples directly contact with oxygen under high temperature and products are liable to be oxidized. While VD products dried under low pressure and low oxygen and had lower a^* and b^* than

SD, OD products but ID product, which is comparable. ΔE^* was widely applied as a colorimetric parameter to represent the color change in food during processing. Many researches regarded $\Delta E^* = 2$ as the threshold of visual discrimination [30]. The color changes when ΔE^* ranges from 0 to 2 are invisible, but those when $\Delta E^* > 2$ are evident and visible. According to Table II, SD had the largest deviation (ΔE^* value of 33.92) while FD samples deviated the least (ΔE^* value of 22.02). On the whole, the five methods showed significantly difference in color retention and FD often produces the highest possible quality in colour protection among the five industrial drying methods.

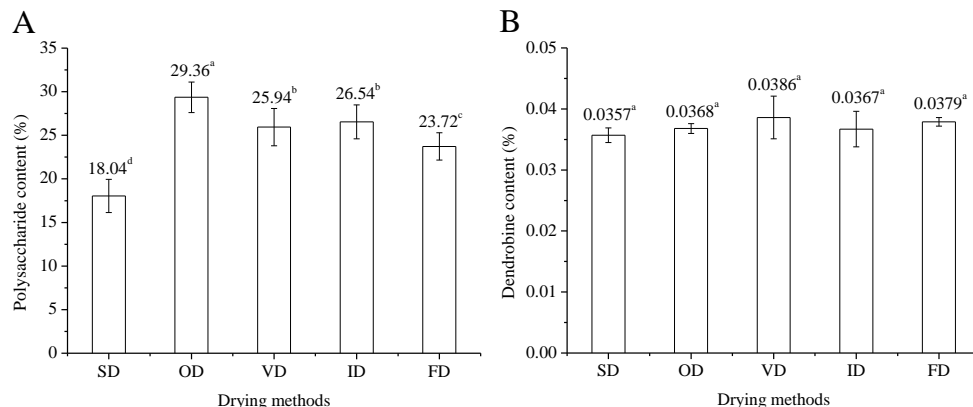


Figure 2. Effect of different drying methods on water soluble polysaccharides and total dendrobine contents. (a) water soluble polysaccharides content. (b) total dendrobine content.

D. Total Protein Content

The levels of protein content of *D. officinale* processed by different drying methods are presented in Table I. There was no significant difference ($p > 0.05$) of protein contents between different dried products, which indicated that these five drying methods had no effect on protein content of *D. officinale*.

E. Water Soluble Polysaccharides Content

Water soluble polysaccharides content of dried samples with different drying methods are showed in Fig.

2A. *D. officinale* sample dehydrated by oven drying retained the highest water soluble polysaccharides content, which is 29.36% dry base. Compared to vacuum dried and infrared radiation dried products, the water soluble polysaccharides of oven dried product was about 10% more (11.64% and 9.6%, respectively). This could be due to fact that when subjecting the foodstuff to infrared radiation, only less than 10% of the radiation is reflected back, the rest of which impinges on and penetrates into the materials and converted into heat directly [19]. This kind of high energy may cause the decomposition of polysaccharides slightly. Moreover, in

the vacuum environment, the lack or of oxygen may inhibit the respiration of plant tissues, thus may reducing the accumulation of polysaccharides. Freeze dried *D. officinale* only contained 23.72% polysaccharides, which was much lower than that of oven dried, vacuum dried, and infrared radiation dried products, indicated that drying treatment with high temperature (60 °C) could break the balance of carbohydrate metabolism, leading to the generation of macromolecular substances such as cellulose, so as to make the polysaccharides content increased. But its real mechanism still needs further researches. Sun dried samples contained the lowest polysaccharides, which is only 18.04% of dry matter

(DM). This may attributed to the fact that the plant tissues still maintain a strong respiration during primary stage of sun drying, which would result in a much consumption of sugar or other nutrients of tissues themselves to keep their physiological activities. What's more, the polysaccharides decomposition enzymes in moderate conditions of high water content and mild temperature during sun drying in the initial stage still had higher activities, which stimulated the degradation of polysaccharides ingredients in the tissues. The results indicated that oven drying method could provide a better final dried product quality of *D. officinale* in polysaccharides content.

TABLE II. COLOUR OF *D. OFFICINALE* POWDERS OF DIFFERENT DRYING METHODS.^{1,2,3,4,5,6}

Drying methods	L^*	a^*	b^*	ΔE^*
SD	70.03 ± 0.81 ^a	6.02 ± 0.34 ^a	27.17 ± 0.91 ^a	33.92 ± 1.18 ^a
OD	63.28 ± 1.37 ^b	2.16 ± 0.12 ^b	27.16 ± 0.36 ^a	32.87 ± 1.18 ^a
VD	63.19 ± 0.49 ^b	1.63 ± 0.44 ^c	23.10 ± 0.93 ^b	29.20 ± 0.72 ^b
ID	62.93 ± 1.32 ^b	2.36 ± 0.33 ^b	24.89 ± 0.64 ^b	29.04 ± 1.46 ^b
FD	71.08 ± 2.71 ^a	-1.75 ± 0.12 ^d	20.90 ± 0.70 ^c	22.02 ± 2.72 ^c

¹ L^* : the lightness coordinate.

² a^* : chromaticity coordinate in the red-green axis.

³ b^* : chromaticity coordinate the yellow-blue axis.

⁴ ΔE^* : total color differences.

⁵Values are means ± standard deviation of three replications.

⁶Means values in the same column with different letters are significantly different ($p < 0.05$).

F. Total Dendrobine Content

The total dendrobine content of *D. officinale* with different drying methods are presented in Fig. 2B. Oven dried, vacuum dried, infrared radiation dried and freeze dried products maintained the similar dendrobine content, which are around 0.037%. Even though sun dried samples contained the lowest dendrobine, which was 0.0357% of dry basis, but there is no difference between the five products, The results revealed that different drying process had no obvious effect on dendrobine content, indicated that dendrobine serves as a kind of plant hormones, its total amount remained constant during aging and drying days [4].

G. FTIR Spectra Analysis

The FTIR spectra from 400 to 4000 cm^{-1} of dried samples with different drying methods are shown in Fig. 3. In the “fingerprint” region, the spectra were very complex containing many bands assigned to main *D. officinale* components. In order to confirm the effect of drying methods on *D. officinale* chemical components, detailed peak positions and assignments of the five types of *D. officinale* samples are listed in Table III.

As can be seen from the results, the spectra with a strong and wide stretching peak around 3415 cm^{-1} for O-H and N-H stretching vibrations and a weak absorption peak of about 2925 cm^{-1} for C-H stretching vibrations [31], [32]. The band at around 1736 cm^{-1} was assigned to characteristic bending or stretching vibrations of C=O stretching. There was a broad band located at about 1621 cm^{-1} which was attributed to

COO- stretching [32]. The bands at around 1513 and 809 cm^{-1} were assigned to characteristic stretching vibrations of aromatic skeletal groups. The bands centered at round 1426 and 1377 cm^{-1} were assigned to CH₂ and CH symmetric bending [32]. The bands centered at round 1247 and 1057 cm^{-1} were assigned to characteristic bending or stretching vibrations of C-O stretching, and a weak absorption peaks of about 1157 cm^{-1} for C-O-C stretching vibrations [33]. Three bands at 1321, 897 and 606 cm^{-1} ascribed to C-N stretching, CH stretching out of plane of aromatic ring and O-H bending, respectively [32]. The results depicted that the general spectral profile of the *D. officinale* powders with different drying methods was similar, no new chemical group bands were produced in the *D. officinale* powders of different drying methods, suggested that main structure of the five *D. officinale* samples were still the same.

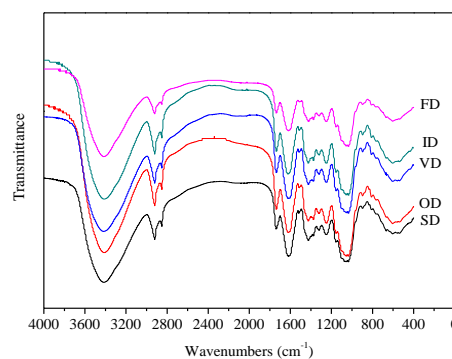


Figure 3. FTIR spectra of *D. Officinale* samples with different drying methods.

H. Moisture Sorption Isotherms

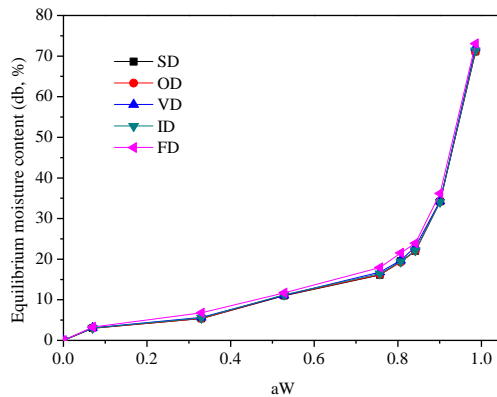


Figure 4. Moisture sorption isotherms of *D. Officinale* powders with different drying methods.

Moisture sorption isotherms, curves of equilibrium moisture content (EMC) against a_w of the powders at 25 °C are shown in Fig. 4. Similar to trends that observed in numerous foods, EMC values of the powders increased with a_w . Sorption isotherm curves of the isotherms had slightly sigmoidal shapes typical for foods. From the study we found that the moisture content at equilibrium was slightly more in the freeze dried sample, and need shorter time to reach the equilibrium at low a_w . What's more, FD samples developed mold more quickly than

other four samples at high water activity. The above phenomena may due to the fact that freeze dried samples kept porous structure and little or no shrinkage, which tended to adsorb the moisture than the other samples. Our results were in accordance with Gong et al. regarding sorption isotherm curves of cabbage powder [13].

IV. CONCLUSIONS

Over all, the present study showed that the five drying methods had a significant effect on color retention, shrinkage and water soluble polysaccharides content of *D. officinale*, but not protein and total dendrobine contents. According to the FTIR spectra, no new chemical bonds were produced between different *D. officinale* powders with different drying methods. As to the water activity, i.e. moisture sorption isotherms, the moisture content in freeze dried sample at equilibrium was slightly more compared with other four samples. Usually, FD products presents the best quality, but with aspect to the water soluble polysaccharides content for *D. officinale*, OD, VD and ID were better than FD, which only had the obvious advantages in the color and shrinkage protection. In summary, the quality of dried *D. officinale* depends on the drying method and conditions. With regard to the retention of the major ingredients in *D. officinale*, namely, polysaccharides and dendrobine, OD is very promising for dry treatment.

TABLE III. MAJOR ABSORPTIONS IN IR SPECTRA OF *D. OFFICINALE* POWDER WITH DIFFERENT DRYING METHODS.

Drying methods					
SD	OD	VD	ID	FD	
Frequency (cm ⁻¹)					Assignments
3423	3416	3418	3415	3416	O-H and N-H group stretching
2919	2919	2919	2919	2919	CH and CH ₂ stretching
2851	2851	2851	2851	2851	CH and CH ₂ stretching
1736	1736	1737	1736	1736	C=O stretching
1618	1618	1621	1622	1618	COO- stretching
1513	1513	1514	1513	1512	Aromatic skeletal vibration
1425	1426	1424	1426	1421	CH ₂ and CH symmetric bending
1378	1377	1378	1376	1377	CH ₂ and CH symmetric bending
1321	1320	1320	1321	1321	C-N stretching
1247	1247	1248	1248	1248	C-O stretching
1154	1155	1155	1156	1155	C-O-C stretching
1057	1057	1057	1057	1057	C O stretching
1035	1035	1035	1034	1035	C-C stretching
897	896	897	897	897	C-H stretching out of plane of aromatic ring
810	810	810	810	809	Aromatic skeletal stretching
605	606	606	605	605	O-H bending

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