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Research Article

Effects of Dry-, Wet- and Freeze-grinding Pretreatment Methods on the Physicochemical Properties of Maitake Mushroom (*Grifola frondosa*) Superfine Powders

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Abstract: The dendritic caps and stipes of maitake mushrooms (*Grifola frondosa*) were pretreated by dry-, wet-, or freeze-grinding followed by jet milling to produce superfine powders. The effects of the pretreatment grinding conditions on the physicochemical properties of these powders were investigated. Compared to the dry and wet processes, the freeze-grinding pretreatment effectively reduced particle sizes (cap, 6.75 μm; stipe, 5.76 μm) and produced narrow and uniform particle size distributions. For cap or stipe, For the same material (cap or stipe), powders from the freeze-grinding pretreatment exhibited higher values of specific surface area, bulk density, water holding capacity, but worse color values than the dry- and wet-ground powders. For the same grinding pretreatment method, cap powders exhibited higher values for the water solubility index and mobility than stipe powders. For the same environmental humidity, the Halsey model showed the best goodness-of-fit for the moisture sorption isotherms of the superfine powders.

Keywords: Maitake mushroom (*Grifola frondosa*), mathematical model, micronization, microstructure, physicochemical properties

INTRODUCTION

The maitake mushroom, *Grifola frondosa,* is a Basidiomycete fungus belonging to the Polyporaceae family. With its unique appearance, the maitake is commonly known as the hen-of-the-woods or coral reef mushroom (Shin and Lee, 2014). This edible mushroom is widely used as a culinary ingredient and dietary supplement in Asia because of its enticing flavor and nutritional value (Hong *et al*., 2013; Tsao *et al*., 2013). It is rich in amino acids, vitamins C, D_2 and B_2 and minerals Mg, P and Ca, as well as a good source of dietary fiber (Cao *et al*., 2003). Maitake mushrooms exhibit multiple physiological activities, including anti-tumor (Tsao *et al*., 2013), antioxidant (Shin and Lee, 2014), hypotensive (Talpur *et al*., 2002), hypoglycemic (Mau *et al*., 2004) and immune-stimulating effects (Chen *et al*., 2012).

Fresh mushrooms easily deteriorate because of their high moisture content and nutrient density. In terms of preservation, studies have increasingly focused on various processes for the drying of mushrooms (Cui *et al*., 2013). Moreover, maitake mushroom properties are not easy to work with; they soften during cooking, which limits more extensive use. In order to increase the use of maitakes and promote the development of the maitake industry, the mushrooms can also be supplied as processed powders, which are functional food materials that can be added to a variety of foods. For example, Seguchi *et al*. (2001) prepared maitake powder without heating and used it in bread-making experiments and Lee *et al*. (2007) and Lee and Lee (2007) prepared a sponge cake with *G. frondosa* powder. The extent to which the powders can be used depends on their characteristics, which are contingent on particle size and the method applied in powder production. Commonly used methods include routine grinding and micronization (Zhang *et al*., 2012). Superfine maitake powders obtained from micronization, which may have more utility, have properties that are not found in powders from conventional grinding methods (Zhao *et al*., 2009). Superfine grinding can effectively improve a powder's particle size and crystalline structure, making the technology useful as a food processing tool. Compared to conventional particulate materials, the resulting superfine powders can possess good physicochemical properties such as dispersibility, absorptivity, optical properties, solubility and chemical and catalytic properties (Hemery *et al*., 2011; Wu *et al*., 2012). Superfine powders are also more easily absorbed by the body, which would consequently improve the quality of food products. In addition, because the superfine grinding technology is a purely physical treatment, nutrient retention in the raw material is higher, which preserves the products' natural attributes.

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The effects of micronization treatments on the characteristics of powdered products may vary depending on the grinding method and raw materials (Chau *et al*., 2007). A new method was recently investigated that used a freeze-grinding process in which rice was frozen with liquid nitrogen prior to dry grinding (Ngamnikom and Songsermpong, 2011), but only a few physicochemical properties were studied. The present work aims to observe the differences in the physicochemical properties of superfine powders of maitake mushroom dendritic caps and stipes produced by jet milling after dry-, wet- and freeze-grinding pretreatments, in order to broaden their application scope.

MATERIALS AND METHODS

Material preparation: Fresh maitake mushrooms (*G. frondosa*) were procured from a local market and stored in the refrigerator at 4°C. The initial moisture content of the mushrooms was 89±1.5% (kg H_2O/kg mushrooms). The specimens were finely colored and free of pests, disease and mechanical damage. The mushrooms were washed in clean water and the dendritic caps and stipes were separated. The protein conversion factor was N×5.7. The moisture, crude protein, fat and total ash contents in the raw ingredients were determined using AOAC methods (Nos. 925.09, 920.87, 920.85 and 923.03; AOAC (Association of Official Analytical Chemists) (1990). The total sugars were determined by an enzymatic procedure according to the AOAC (Association of Official Analytical Chemists) (1997).

Powder preparation:

Dry-grinding pretreatment: For dry grinding, the dendritic caps and stipes were dried to a water content of 8.0±0.5% at 45°C (because high temperature causes enzymatic browning) in an electric thermal dryer (Type-101-2, Shanghai Boxun Industry and Commerce Co., Ltd., China). The dried materials were ground with a JSP-200 high-speed pulverizer (Jinsui, Yongkang, China) until all particles of the powder passed through a 60 mesh sieve.

Wet-grinding pretreatment: For wet grinding, the dendritic caps and stipes were chopped manually. The pulverization process was carried out for 30 sec in a collider mill. The seriflux was poured into a 200 mesh gauze filter to remove excess water. The wet, ground residue was dried in an electric thermal dryer at 45°C until the water content was 8.0±0.5%. Then the powder was passed through a 60 mesh sieve.

Freeze-grinding pretreatment: For freeze-grinding, the dendritic caps and stipes were cut into 1 cm^2 pieces that were steeped in liquid nitrogen for 1 min. The frozen samples were ground with the JSP-200 high-speed pulverizer; this process was repeated twice. The seriflux was then poured into a 200 mesh gauze filter to remove excess water. The wet, ground residue was dried in an electric thermal dryer at 45°C until the water content was 8.0±0.5%. Then the powder was passed through a 60 mesh sieve.

The resulting dry-, wet- and freeze-pretreated flour samples were passed through a 60 mesh sieve, micronized by jet milling (feeding frequency, 40 Hz; working pressure, 7 kg/cm^3; QLM-80K, Shangyu City Heli Powder Engineering Co., Ltd., China), packed in plastic bags and stored at room temperature. Six different powders were obtained, designated as: DGPS (Dry-Grinding Pretreated Stipe), DGPC (Dry-Grinding Pretreated Cap), WGPS (Wet-Grinding Pretreated Stipe), WGPC (Wet-Grinding Pretreated Cap), FGPS (Freeze-Grinding Pretreated Stipe) and FGPC (Freeze-Grinding Pretreated Cap).

Particle size analysis and Scanning Electron Microscopy (SEM): The particle sizes and size distributions of the six superfine powders were measured using a Mastersizer LA-920 laser particle size analyzer (HORIBA Ltd., Japan). Morphological characterization of the superfine powders was accomplished with a scanning electron microscope (KYKY-2800, Zhongke Scientific Instrument, Inc., Beijing, China) operated at a 20.0 kV acceleration voltage. The powders were coated using a gold-palladium alloy coater (Bal-Tec Co., Manchester, NH) and the samples were observed at 4000× magnification (Hemery *et al*., 2011; Sosa *et al*., 2012; Sowbhagya *et al*., 2008).

Color measurement: The color of the superfine powder samples was evaluated using an SMY-2000 color reader (Exact Science Inc., Beijing, China) with the CIELAB color scale. The total color change (ΔE), green [-] to red [+] (a), blue [-] to yellow [+] (b) and light (L) were calculated by the reader. The values for the raw maitake fruit body, represented as L_0, a_0 and b_0, obtained as reference points, were 45.16, 0.76 and 6.73 for the cap and 47.48, 0.91 and 10.93 for the stipe, respectively (Demirkesen *et al.*, 2011; Nath and Chattopadhyay, 2008).

Bulk density measurement: The bulk density (g/mL) was determined according to Bai and Li (2006) with slight modification. A 10 mL measuring cylinder (W_1) was filled with a superfine powder sample up to the mark and then weighed (W_2). The bulk densities of the mushroom powders were calculated as follows: $d = (W_2 - W_1) /10$.

Angles of repose and slide measurements: The angle of repose (α) was defined as the maximum angle subtended by the surface of a heap of powder against the plane which supported it (Taser *et al*., 2005). Filler was fixed vertically above a piece of glass at a distance (*H*) of 2 cm. The test powders were continuously poured into the filler, exiting freely until the tips of the powder cones touched the filler outlet. The diameter (2*R*) of the cone was measured for each type of powder. Values of α were calculated using the following formula: α = arctan (*H*/*R*). The sliding angle was estimated by gradually lifting the glass plane until about 90% of the superfine powder surface had moved. The angle between the vertical (*H*) and horizontal (*L*) distance is termed the angle of slide (β), according to the following formula: β = arctan (*H*/*L*) (Zhao *et al*., 2009; Zhang *et al*., 2012).

Hydration properties: To determine the Water-Holding Capacity (WHC) of a sample, a washed and dried centrifuge tube was tared (*M*, g) and reweighed after introduction of a powder sample (2 g, M_1, g). The powder was blended with water in a 0.05:1 (m/V) ratio. The dispersion was immersed in a water bath at 60°C for 30 min and then immediately cooled in ice water for 30 min. The tube was centrifuged at 5000 rpm for 20 min. The supernatant was removed and the centrifuge tube with the sediment (M_2, g) was weighed again (Zhao *et al*., 2009; Zhang *et al*., 2012). The WHC of the mushroom powders was calculated as follows:

$$\text{WHC (g/g)} = (M_2\text{-}M) / M_1$$

To determine the Water Solubility Index (WSI), a powder sample (S_1, g) was weighed and poured into water in a 0.02:1 (m/V) ratio. The dispersion was heated in a water bath at 80°C for 30 min and then centrifuged at 5000 rpm for 15 min. The supernatant was carefully poured into a pre-weighed evaporating dish (S_2, g) and dried to constant weight at 103±2°C, whereupon the sediment (S_3, g) was weighed again. The WSI of the mushroom powders was calculated as follows: WSI (%) = $(S_3 - S_2) / S_1 * 100$.

The Swelling Capacity (SC) was assessed by slowly pouring a powder sample (1 g, *M*) into a graduated test tube with a stopper and recording its volume (V_1). Distilled water (10 mL) was added and the tube was shaken until a homogeneous dispersion was achieved. The dispersion was stored in a water bath at 25°C for 24 h to fully swell the powder. Then, the volume (V_2) of the wet powder was recorded. The SC of the mushroom powder was calculated as follows: SC (mL/g) = $(V_2 - V_1) / M$ (Lecumberri *et al*., 2007).

Moisture sorption isotherm measurements: Moisture absorption isotherms were measured according to the previously described method (Lee *et al*., 2007; Zhang *et al*., 2012). A superfine mushroom powder sample was dried in an oven at room temperature for 48 h to achieve a uniform moisture content. The equilibrium moisture content of the superfine powders was determined using a gravimetric technique via the diffusion method (the Conway dish method). The determination of moisture content was performed by the direct drying method over saturated salt solutions of NaOH (water activity, a_w, 0.070), $MgCl_2$ (a_w, 0.331), NaBr (a_w, 0.577), KCl (a_w, 0.842) and $K_2Cr_2O_7$ (a_w, 0.986). Several isotherm models, including the Halsey, Brunauer-Emmett-Teller (BET), Kuhn, Bradley, Caurie, Chung-Pfost and Oswin models, were used to fit the measured moisture sorption data (Yanniotis and Blahovec, 2009).

Statistical analysis: All the analyses were performed in triplicate and are shown as means±Standard Deviations (S.D.). Duncan's ($p<0.05$) multiple range tests were performed using SPSS (IBM SPSS Statistics, IBM Corp., Version 17.0).

RESULTS AND DISCUSSION

Proximate composition: The chemical compositions of the superfine maitake (*G. frondosa*) mushroom powders are shown in Table 1. For the same pretreatment method, the mushroom cap and stipe components displayed significant differences in content ($p<0.05$), except for fat. The total sugar content in the maitake caps (48.87 g/100 g) was much higher than in the stipes ($p<0.05$). Moreover, the protein, fat and ash contents of the maitake caps were higher than the stipes. The protein content, as high as 28.68 g/100 g, was significantly higher than in other edible fungi. Thus, in terms of nutrient content, the dendritic cap of the maitake mushroom is superior to the stipe. This finding is consistent with the results for *Lentinus edodes* reported by Zhang *et al*. (2012). For the same superfine powders (cap or stipe), the dry-grinding pretreatment retained significantly more nutrients than wet- and freeze-grinding processes. This may be ascribed to the removal of water during filtering in the latter, which resulted in the loss of water-soluble constituents.

Particle size: The particle sizes and size distributions of the superfine powders as obtained by laser particle

Table 1: Proximate compositions of maitake mushroom powders as affected by grinding pretreatment method[a]

Powder	Protein (g/100 g)	Total sugar (g/100 g)	Ash (g/100 g)	Fat (g/100 g)
DGPC	28.68±0.160[b]	48.87±0.351[b]	5.84±0.090[b]	1.81±0.153[b]
WGPC	28.29±0.066[c]	47.23±1.604[b]	3.98±0.185[c]	1.78±0.058[b]
FGPC	28.06±0.146[c]	46.50±1.442[bc]	3.39±0.161[d]	1.81±0 .078[b]
DGPS	27.29±0.052[d]	44.03±1.011[cd]	5.64±0.081[b]	1.74±0.115[b]
WGPS	27.15±0.062[d]	43.45±0.390[d]	3.10±0.125[e]	1.76±0.100[b]
FGPS	26.86±0.088[e]	42.70±1.400[e]	2.98±0.076[e]	1.79±0.058[b]

[a]: Values are expressed as the mean±standard deviation of triplicate analyses; Data were calculated on a dry basis; [b-e]: Values bearing different superscript letters within the same row are significantly different (Duncan, $p<0.05$)

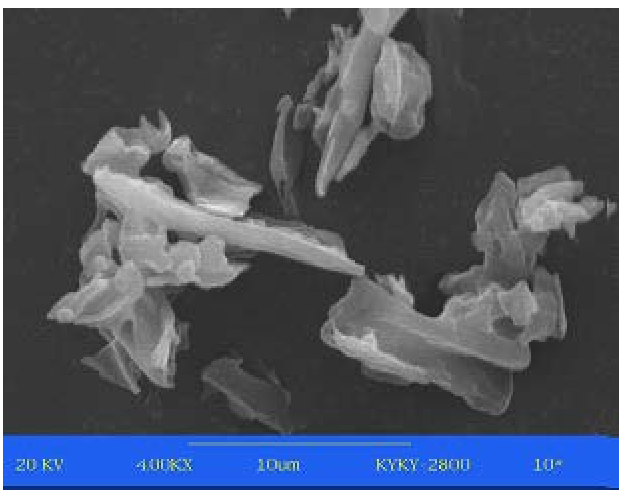
size analysis are shown in Table 2. The particle size distributions were characterized by $D_{0.1}$, $D_{0.5}$ and $D_{0.9}$ values (Giry *et al*., 2006). With the same grinding pretreatment, the wet- and freeze-processed cap powders had greater mean sizes than the stipes, but this trend was reversed for the drying pretreatment. The dry-grinding pretreatment method afforded the largest particle sizes. In contrast to the dry- and wet-grinding methods, the freeze-grinding pretreatment significantly reduced the average particle sizes of the powders (5.76 and 6.75 μm for stipe and cap, respectively). This may occur because the samples are generally brittle and easily broken at extremely low temperature (Devi *et al*., 2009). Freeze-grinding also resulted in narrow and uniform particle size distributions ("spans:" cap, 1.47; stipe, 1.56). A smaller span value indicates a narrower particle size distribution and more uniform size (Zhang *et al*., 2012). The particle size results are consistent with those for the specific surface area: a reduced particle size increases the specific surface area of the material.

Microscopic structure: Electron microscopy is a valuable tool that provides data on microstructure,

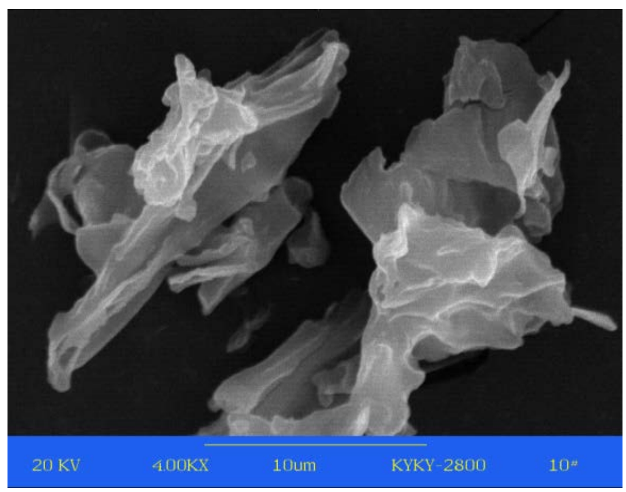
Table 2: Effects of grinding pretreatment methods on particle sizes of maitake mushroom powders[a]

Powder	$D_{0.1}$	$D_{0.5}$	$D_{0.9}$	Span[b]	Specific surface area (cm^2/cm^3)	Mean size (μm)
DGPC	3.07	7.14	15.75	1.78	10425	8.52
WGPC	2.66	6.30	13.09	1.66	12012	7.26
FGPC	2.67	6.06	11.59	1.47	12283	6.75
DGPS	3.75	9.08	21.23	1.93	8349	11.40
WGPS	2.44	5.96	12.35	1.66	12904	6.83
FGPS	2.30	5.00	10.08	1.56	14430	5.76

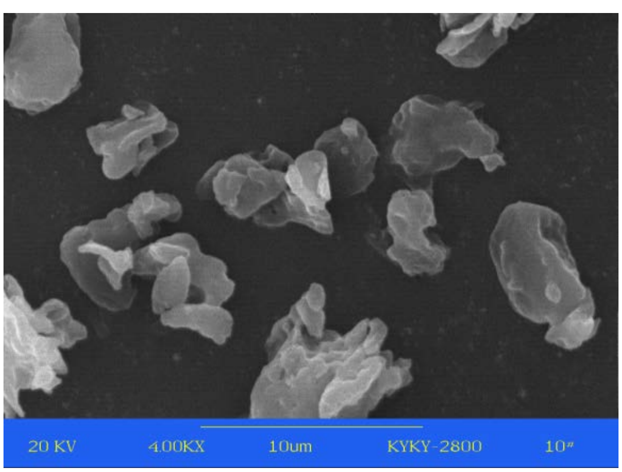
[a]: Values are expressed as means of triplicate analyses; [b]: Span = $(D_{0.9} - D_{0.1})/D_{0.5}$; [c]: $D_{0.1}$, $D_{0.5}$ and $D_{0.9}$ are the equivalent volume diameters at 10, 50 and 90% cumulative volumes, respectively



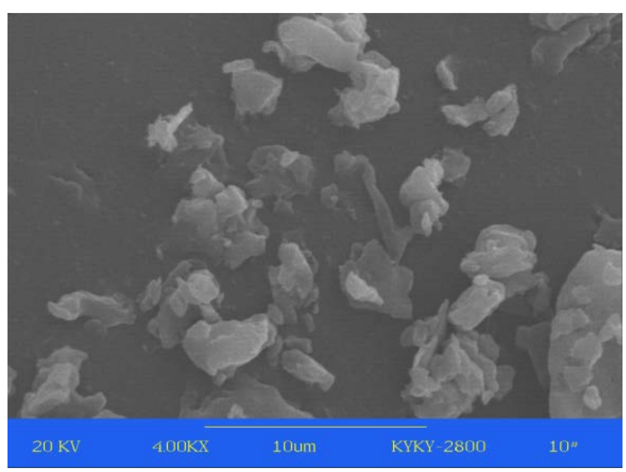
(a) DGPC



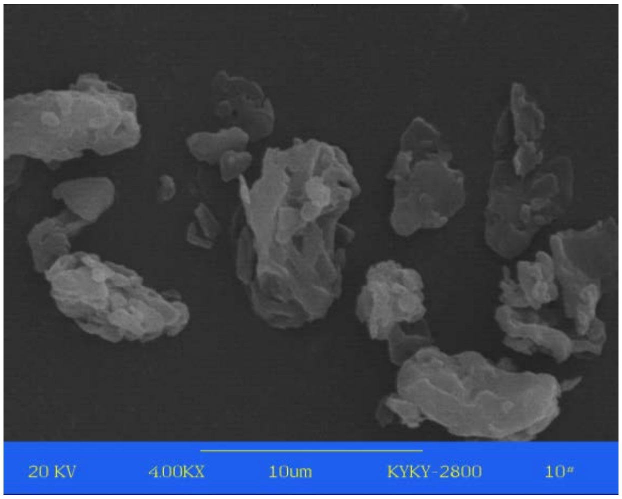
(b) DGPS



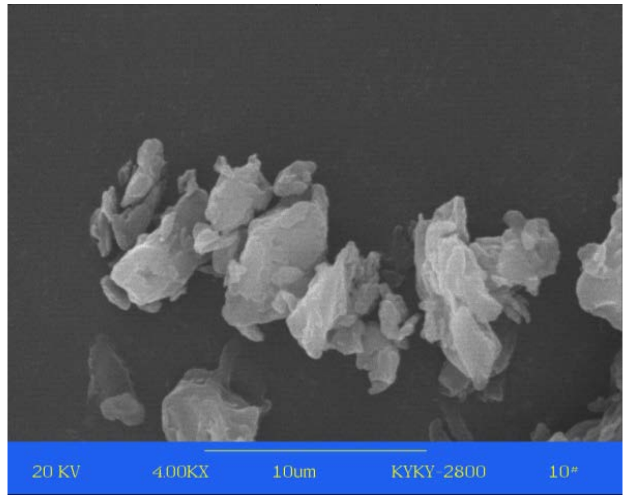
(c) WGPC



(d) WGPS



(e) FGPC



(f) FGPS

Fig. 1: SEM images of maitake mushroom powders obtained from various grinding pretreatment methods

which are necessary for a thorough understanding of the changes that occur during food processing (Nath and Chattopadhyay, 2008). SEM observations revealed the shapes and surface morphologies of the six superfine mushroom powders (Fig. 1). The microscopic structures of the caps and stipes of the superfine powders were not obviously different with the same grinding pretreatment method. However, there were significant differences between the three pretreatment processes. The powders from the dry-grinding pretreatment were more elongated with larger particle sizes and irregular surfaces. The wet-grinding pretreatment produced small round powder particles with smooth surfaces, which seemed to consist of homogeneous structures. The freeze-grinding pretreatment powders exhibited layered structures and had greater specific surface areas, in agreement with the data in Table 2. Therefore, the freeze-ground powders clumped easily, had strong adsorption abilities and should be effectively adsorbed on food surfaces because of their layered structures (Hayakawa *et al*., 1993).

Color: The color of food is an important factor that affects its sensory qualities. The color parameters for the maitake mushroom superfine powders after the different grinding pretreatment methods are shown in Table 3. For caps and stipes treated by the same grinding method, the samples exhibit no significant differences ($p>0.05$), excluding DGPC and DGPS. However, the L, a and ΔE values reveal obvious differences ($p<0.05$). Visually, the stipe powder color appears significantly lighter. For the three different grinding pretreatment methods, the freeze-processing method exhibited higher a and b values compared to the dry and wet ($p<0.05$), but lower L and ΔE values compared to the dry and wet. It can be concluded that the freeze-grinding pretreatment does not retain the color of the maitake mushroom in the superfine powders as the other treatment materials. It is possible that the cells are damaged and in contact with oxygen for a long time during the freeze-thaw cycles. Therefore, enzymatic browning occurred. Compared to the other two methods, the dry-grinding pretreatment produced a lighter color.

Bulk density and angles of repose and slide: The angles of repose and slide reflect changes in the fluidity of a powder (Zhao *et al*., 2009). The repose and slide angle values for the maitake mushroom superfine powders from the different preparation methods are shown in Table 4. The angles of repose for the caps and stipes exhibit no significant differences ($p>0.05$) for the same grinding pretreatment method. The freezing and drying pretreatment methods had significant differences ($p<0.05$) in terms of slip angle. The micronized powders from the dry and wet methods have significantly lower slide angle values than the freeze-ground powders derived from the same material (cap or stipe). The freeze-grinding pretreatment method results in the largest slide angles (cap, 39.35°; stipe, 40.80°), but they are still lower than those observed for *L. edodes* and ginger powder (Zhao *et al*., 2009; Zhang *et al*., 2012). This may be attributed to the smaller size and larger specific surface area observed for the freeze-ground particles, which enhance surface clumping and adhesion between particles and draw them closer together. The angles of repose for the pretreatment methods followed the sequence dry>wet>freeze. We consider this ordering to be related to the span or particle size distribution, wherein smaller particles fill the voids between larger particles and reduce fluidity. Higher adsorption results in better stability of product quality after mixing. These results are in agreement with the findings of Zhao *et al*. (2010).

Table 3: Color parameters for the six superfine maitake mushroom powders

Powder	a	b	L	ΔE
DGPC	0.56±0.0100[d]	9.58±0.0503[c]	52.53±0.1350[b]	5.40±0.1301[b]
WGPC	2.21±0.0351[c]	10.10±0.1301[b]	45.06±0.2307[d]	2.68±0.1051[c]
FGPC	2.27±0.1179[c]	11.52±0.1915[a]	44.94±0.2200[d]	2.52±0.1922[c]
DGPS	2.04±0.1351[c]	8.12±0.2233[d]	57.54±0.1801[a]	10.45±0.2300[a]
WGPS	2.65±0.1200[b]	9.95±0.2952[b]	47.33±0.1800[c]	1.24±0.1620[d]
FGPS	3.26±0.1704[a]	11.53±0.1009[a]	46.99±0.2255[c]	1.56±0.1866[d]

[a-e]: Values bearing different superscript letters within the same row are significantly different (Duncan, $p<0.05$)

Table 4: Effect of grinding pretreatment methods on bulk density and repose and slide angles of maitake mushroom powders

Powder	Repose angle (°)	Slide angle (°)	Bulk density (g/mL)
DGPC	47.37±0.7907[a]	35.88±1.1892[d]	0.4175±0.0110[cd]
WGPC	47.25±1.4039[a]	36.03±0.7558[c]	0.4152±0.0183[cd]
FGPC	44.76±0.4088[b]	39.35±1.2945[b]	0.4877±0.0098[a]
DGPS	48.02±0.7915[a]	37.22±0.3027[c]	0.3892±0.0075[d]
WGPS	47.76±0.9906[a]	36.13±1.1466[c]	0.4392±0.0172[bc]
FGPS	45.74±1.2563[b]	40.80±0.6945[a]	0.4728±0.0053[ab]

[a-d]: Values bearing different superscript letters within the same row are significantly different (Duncan, $p<0.05$)

Table 5: Effect of grinding pretreatment methods on hydration properties of maitake mushroom powders

	Hydration properties		
Powder	Water holding capacity, WHC (g/g)	Water solubility index, WSI (%)	Swelling capacity, SC (mL/g)
DGPC	3.69±0.0468[b]	45.14±0.4394[ab]	1.51±0.0493[e]
WGPC	3.53±0.0525[c]	46.12±0.5831[a]	2.15±0.0316[bcd]
FGPC	3.70±0.0451[b]	46.20±0.4278[a]	2.46±0.4968[ab]
DGPS	3.86±0.0499[a]	44.51±0.5257[b]	1.85±0.0433[cde]
WGPS	3.74±0.0545[ab]	45.77±0.1139[a]	2.38±0.2053[abc]
FGPS	3.78±0.0575[ab]	45.87±0.3269[a]	2.80±0.0892[a]

[a-e]: Values bearing different superscript letters within the same row are significantly different (Duncan, $p<0.05$)

There were no significant differences between the caps and stipes for the same grinding pretreatment method ($p>0.05$) for bulk density (Table 4). Among the three processes, freeze-grinding produced the highest bulk densities (cap, 0.4877; stipe, 0.4728 g/mL). In this case, the lower particle size may result in a larger contact surface with the surroundings and a higher homogeneity, which would lead to a decrease in the pore spaces between particles and an increase in the bulk density value (Zhao *et al*., 2009). The bulk densities of the maitake powders were significantly higher than that of *L. edodes* (0.228 g/mL). The maitake mushroom powders with high bulk density would therefore be potential ingredients for use in instant beverages (Zhang *et al*., 2012).

Hydration properties: The differently sized powder particles exhibited distinct hydration properties (Table 5). The Water Holding Capacities (WHC) were obviously different between the superfine powders of the caps and stipes from the same pretreatment method ($p<0.05$) and the stipe powder WHC was larger than that from the cap. This might be attributed to the proximate composition of the tissues, especially with respect to protein and water-soluble cellulose contents. Additionally, there was no significant difference in WHC among the three grinding pretreatment methods ($p>0.05$).

The WSI values were not obviously different between the superfine powders of caps and stipes for the same pretreatments ($p>0.05$). However, significantly higher WSI values were observed both for wet- and freeze-ground powders than for dry-processed powders ($p<0.05$). The WSI of the maitake powder was as high as 46.2%, significantly higher than that of *L. edodes* (Zhang *et al*., 2012). The experiments revealed an obvious stratification in the tube after standing 24 h: a layer of orange liquid appeared at the bottom of the tube. This may have been due to a decrease in the powder particle size which increased the contact area between the powder and water and subsequently, the contents of soluble constituents such as chlorophyll and polysaccharides.

The Swelling Capacities (SC) were not obviously different between the superfine cap and stipe powders with the same pretreatment ($p>0.05$). For the three methods, the SC order was freeze>wet>dry. As the particle size decreased, increasing numbers of hydrophilic groups were exposed, which increased the expansion volume of the powder.

Moisture Sorption Isotherms (MSI): Plots of Equilibrium Moisture Content (EMC) versus the water activity (a_w) of the superfine maitake mushroom powders at 25°C are shown in Fig. 2. Similarly to trends observed for numerous foods, the EMC values of the powders increased with water activity (0-1) and were represented as reverse S-shaped curves. At a_w values of about 0.25 and 0.8, the adsorption rates of the powders obviously change. In the first case (a_w 0.25), the moisture can be characterized as monolayer water content in the food. When a_w molecular layers of water are around 0.8, multi are formed. The changes in the manner of water combination with adjacent molecules

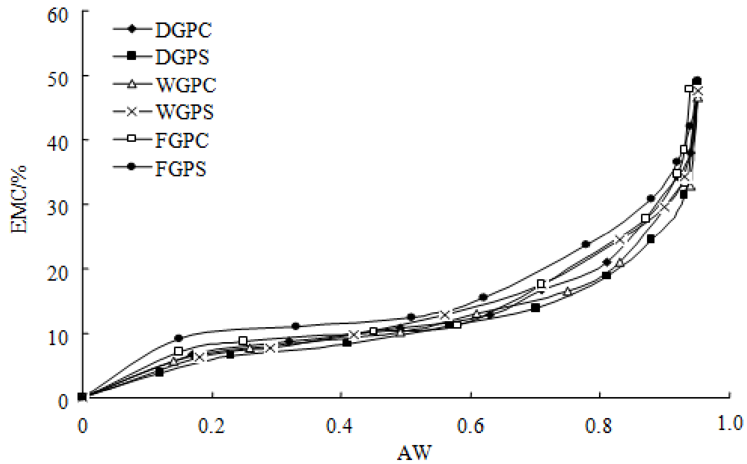


Fig. 2: Effects of different grinding pretreatment methods on moisture sorption isotherm characteristics of maitake mushroom powders

Table 6: Mathematical expressions, coefficients of determination (R^2), mean relative percentage Errors (E%), and Root Mean Square Errors (RMSE) of selected sorption models for maitake mushroom powders in the relative humidity range 10-98%[a]

Model	Mathematical expression[b]	R^2	E%	RMSE
Halsey	$a_w = \exp(-a/m^b)$	0.9921-0.9990	-4.734-3.184	0.0144-0.0396
BET	$m = aba_w / [1 + (b - 2)\ a_w - (b - 1)\ c^2]$	0.7378-0.8758	6.605-20.198	5.4396-7.3552
Kuhn	$m = a/\ln a_w + b$	0.9675-0.9862	2.512-7.494	1.7659-2.5616
Bradley	$m = \ln(1/a_w)/ab$	0.8204-0.9068	22.221-34.069	0.1187-0.1585
Caurie	$\ln m = \ln a - ba_w$	0.9462-0.9781	2.849-14.032	0.0230-0.0775
Chung-Pfost	$\ln a_w = -a*\exp(-bm)$	0.8972-0.9578	-3.000-(-7.613)	3.0555-4.5712
Oswin	$m = a\ [a_w/(1 - a_w)]^b$	0.9707-0.9912	-1.553-(-7.868)	1.3728-2.4048

[a]: E was determined by the following equation: $E = \frac{100}{n}\sum_{i=1}^{n}(\frac{s_{pi} - s_i}{s_i})$, where n is the number of experimental observations; s_i: The experimental moisture content values; s_{pi}: The value predicted from the model; [b]: m, equilibrium moisture content (%, dry basis); a, b, and c: Constants; a_w: Water activity

Table 7: Nonlinear regression parameters of the Halsey model ($a_w = \exp(-a/m^b)$) for the different maitake mushroom powders

	Parameters		
Powder	a	b	R^2
DGPC	66.105	1.898	0.9990
DGPS	32.478	1.698	0.9933
WGPC	60.798	1.898	0.9974
WGPS	39.464	1.680	0.9975
FGPC	84.108	1.975	0.9964
FGPS	72.560	1.943	0.9921

alter the moisture absorption rates of the sample. After superfine grinding, the a_w values of the maitake cap powders were lower than the stipe for the dry-grinding pretreatment under the same EMC, but the wet- and freeze-processed materials gave the opposite results. With the same material (cap or stipe), the a_w values of superfine maitake cap powders were lower under the same EMC. These findings agree with the results observed for shiitake mushrooms by Zhang *et al.* (2012). There were significant differences between the three methods of pretreatment, affording the order dry>wet>frozen. The results indicate that powders obtained after the freeze-grinding pretreatment are more stable than the wet- and dry-treated powders when they are stored under the same conditions, especially relative humidity. These findings are in agreement with the results related to the powder particle size reported by Lee *et al*. (2007) (Table 2).

The seven sorption models presented in Table 6 were assessed computationally (SPSS 17.0) for goodness-of-fit of the maitake superfine powder data. The isotherms were described in terms of the coefficient of determination (R^2), mean relative percentage Error (E%) and Root Mean Square Error (RMSE). These models were selected based on their computational simplicity and effectiveness at describing the sorption isotherms of several foods (Zhang *et al*., 2012). The results revealed that the fits of the BET and Bradley models had larger errors than the others because of lower degrees of conformity in the initial segment of the model regression-fitting curves. The degree of conformity of the Caurie model regression-fitting curve obviously decreased when fitting the MSI data, but the change in the R^2 value was very small. The Oswin and Halsey models were well fitted to the data. Considering R^2, RMSE and E%, the Halsey model was chosen because of its good ability to satisfactorily predict the EMCs of the six superfine powders, with high R^2 (0.9921-0.9990) and low absolute RMSE values (0.0144-0.0396) (Table 6 and 7). The Halsey regression model parameters are shown in

Table 7. The regression equation can also be used to interpolate the values of a_w and the water content in maitake superfine powders.

CONCLUSION

Powders of the dendritic caps and stipes of maitake mushrooms were prepared by jet milling after three different pretreatment methods. The effects of dry-, wet- and freeze-grinding pretreatment methods on the physicochemical properties of the maitake mushroom superfine powders were investigated. The protein, total sugar, fat and ash contents in the maitake caps were higher than in the stipes, indicating their higher nutritional value. Under the same conditions, the average particle size of the powder pretreated by freeze-grinding was smaller but the color was worse than from the other methods. Freeze-ground powders exhibited layered structures after superfine grinding and had higher specific surface areas, as observed by SEM analysis. Use of this technique in food processing could improve the availability of nutrients as well as sensory qualities. Particle size plays an important role in powder properties. For example, as the particle size decreases, the specific surface area increases and the hydration properties are significantly improved which are desirable properties for food materials used as additives. With the same material (cap or stipe), the a_w of superfine maitake cap powders was lower under the same EMC. There were significant differences (a_w) among the three methods of pretreatment, in the order dry>wet>freeze. The results indicate that powders from the freeze-grinding pretreatment would have better stability and greater resistance to deterioration under storage than the wet- and dry-pretreated powders when held under the same conditions, especially relative humidity. These findings strongly suggest the use of these prepared maitake mushroom powders as functional food additives.

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