

## Moisture Sorption Isotherm Characteristics of *Chaga* Mushroom Powder as Influenced by Particle Size

Min Ji Lee and Jun Ho Lee\*

Department of Food Science and Engineering, Daegu University, Gyeongsan, Gyeongbuk 712-714, Korea

**Abstract** Adsorption isotherms for *chaga* mushroom powder as influenced by particle size were investigated using a gravimetric technique. Samples were equilibrated in desiccators containing sulfuric acid solutions of known water activity (0.11-0.93), then placed in temperature-controlled chambers for approximately ten days. Equilibrium moisture content (EMC) of *chaga* mushroom powder increased with water activity in all samples. EMC was slightly greater in the samples comprised of smaller particle size, however there was no marked difference in appearance between the three samples. The *chaga* mushroom powder exhibited Type II behavior. When the BET model was used to determine mean monolayer values, 0.077, 0.077, and 0.070 H<sub>2</sub>O/dry solid was observed for <250, 250-425, and 425-850  $\mu\text{m}$  sized samples, respectively, however mean monolayer values were 0.121, 0.111, and 0.101 H<sub>2</sub>O/dry solid, respectively, when the GAB model was used. The experimental EMC values were related to the computed values from Henderson's model. The coefficient of determination and standard error for the linear regression were 0.997 and 0.003, respectively.

**Keywords:** *chaga*, *Inonotus obliquus*, drying, adsorption, isotherm, modeling

### Introduction

Mushrooms have received special attention due to their wide application as a nutritionally functional food as well as a source of physiologically beneficial medicines (1, 2). *Inonotus obliquus* (*chaga*) mushroom is a black parasitic fungus that grows on birch trees in colder northern climates between latitudes 45 and 50°N (3, 4). *Chaga* is one of the most popular medicinal species due to its therapeutic effects (1). It is widely used as a folk remedy in Russia with no toxicity (5) and has been used in treatment of cancers and digestive system diseases with no observed side effects (6-8). In spite of the increasing usage and application of *chaga*, most investigation have focused on evaluation of its functional properties, studies on the processing of *chaga* are limited (9-16).

Moisture sorption isotherms represent the equilibrium relationship between water activity and moisture content of foods at constant pressure and temperature (17). Moisture sorption characteristics are of great importance in understanding processing and storage principles (18). Extended shelf-life of foods can be achieved by reducing the moisture content to levels below those required by microorganisms. This information is also essential for modeling, designing, and optimizing the drying process, evaluating storage stability, and microbiological safety, determining moisture changes during storage, and selecting appropriate packaging material (19).

Numerous studies on the moisture sorption behavior of foods have been conducted during last two decades, resulting in several mathematical models being proposed to describe this behavior. However, information on moisture sorption characteristics of the *chaga* mushroom is still scarce due to most studies focusing on drying

techniques and quality aspects of cultivated mushrooms (18).

Choosing a mathematical model to describe integrated hygroscopic properties of various constituents of any substances to be stored is very useful, however the most appropriate sorption model must be selected based on the closeness of fit to the experimental data from the product as well as simplicity of the model (21). Therefore, gathering more relevant *chaga* mushroom sorption data is necessary to improve drying processes while minimizing processing costs (17).

The objectives of the present study were to provide reliable experimental data regarding water vapor sorption characteristics of *chaga* mushroom powder prepared by hot-air drying in order to suggest an existing sorption model to best predict *chaga* sorption behaviors, and to determine the constants of suggested models as influenced by particle size.

### Materials and Methods

**Materials** Fresh *chaga* mushroom was obtained from the Korean Ginseng Corp. (Imported from Baikal Herb Ltd., harvested in March 2006) and stored at room temperature before use. Hot-air drying method was used. Samples were dried using the hot air method by drying at 50°C using a hot-air drying oven (DMC-122SP; Daeil Engr. Co., Seoul, Korea) to a final moisture content of approximately 4-6%, moisture-free basis (MFB).

**Experimental procedure** The moisture content of fresh and dehydrated *chaga* mushrooms was determined by drying in a convectional oven at 105°C overnight (22). Dehydrated mushrooms were cut into approximately 3×3×3 cm pieces and milled using an analytical mill (M20; IKA, Staufen, Germany) with different particle size sieves (D-55743; FRITSCH, Idar-Oberstein, Germany) to yield the following particle sizes; 250 or less, 250-425, and 425-

\*Corresponding author: Tel: 82-53-850-6535; Fax: 82-53-850-6539

E-mail: leejun@daegu.ac.kr

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850  $\mu\text{m}$ .

The equilibrium moisture content of mushroom powder was determined using a gravimetric technique in which the weight was monitored within a standard static system of thermally stabilized desiccators. Binary saturated salt solutions made using laboratory grade and at least 99.5% pure LiCl, MgCl<sub>2</sub>, K<sub>2</sub>CO<sub>3</sub>, NaNO<sub>2</sub>, NaBr, NaCl, KCl, and KNO<sub>3</sub> were used to vary the water activity ( $a_w$ ) from 0.11 to 0.93.

To determine the sorption value, mushroom powder samples (about 2 g) were accurately weighed into a petri dish inside a desiccator. After 1 week, samples were weighed every 24 hr until two consecutive weight changes  $< \pm 0.0005$  g were recorded, at which time the sample was assumed to be at equilibrium. Experiments were done at room temperature ( $23 \pm 1^\circ\text{C}$ ) and all measurements were done in triplicate.

**Isotherm models** Experimental moisture sorption data can be described by many sorption models. The isotherm models used to fit the data are shown in Table 1. Non-linear regression was used to calculate the values of the constants and the mean relative percentage error ( $E$ ), which was used to evaluate the goodness-of-fit of the various equations, was calculated as follows:

$$E = \frac{100}{n} \sum_{i=1}^n \left( \frac{m_{pi} - m_i}{m_i} \right)^2$$

where,  $n$  is the number of experimental observations;  $m_i$  represents experimental moisture content (g/100 g dry matter) values; and  $m_{pi}$  denote values predicted from the models.

In addition to  $E$ , the standard error ( $SE$ ) was used to analyze the goodness of fit of a particular model. The  $SE$  is defined as:

$$SE = \sqrt{\frac{\sum_{i=1}^n (m_{pi} - m_i)^2}{(n-1)}}$$

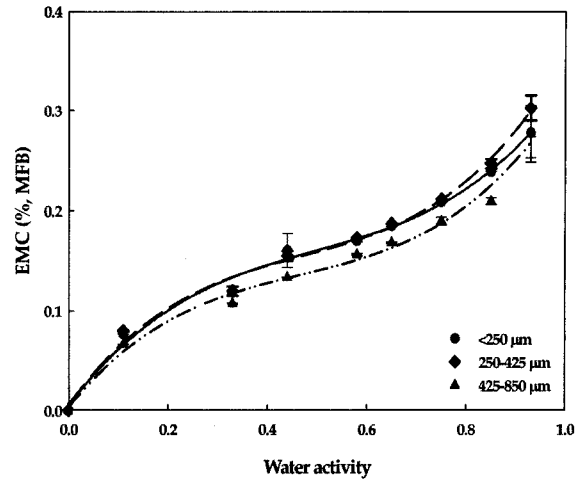


Fig. 1. The isotherm curve of chaga mushroom powders of different particle size using hot-air drying.

**Statistical analysis** The statistical analyses were conducted using the SAS statistical analysis system for Windows v8.1 (23). Means were compared using Duncan’s multiple range test at a 5% level of significance.

**Results and Discussion**

The initial moisture contents of hot-air dried chaga mushroom powders were 5.34-5.76% (MFB), depending on particle size. There was very good agreement between duplicate measurements of moisture content. Experimental equilibrium moisture content (EMC) values of mushroom powder as influenced by particle size, relative humidity, and sorption isotherm curves are shown in Fig. 1. The equilibrium moisture content at each water activity represents the mean value of three replications.

Because the initial moisture content of mushroom samples was low, absorption was the dominant factor. Similar to trends observed in many different types of

Table 1. Data on goodness-of-fit of selected sorption models for chaga mushroom at selected relative humidity (11-93%)

Model	Mathematical expression	R <sup>2</sup>	E (%)	SE (%)
BET	$a_w / [(1 - a_w)m] = 1 / (m_{0B} \cdot c_B) + (c_B - 1)a_w / (m_{0B} \cdot c_B)$	0.951-0.979	1.195-10.870	0.010-0.018
GAB	$m = m_{0G} c_0 k_G a_w / [(1 - k_G a_w)(1 - k_G a_w + c_G k_G a_w)]$	0.960-0.976	2.677-3.138	0.003-0.009
Kuhn	$m = a / \ln a_w + b$	0.723-0.809	17.811-19.622	0.031-0.040
Oswin	$m = k_0 (a_w / 1 - a_w)^{n_0}$	0.965-0.987	4.124-6.066	0.009-0.014
Bradley	$\ln \frac{1}{a_w} = K_2 K_1^M$	0.983-0.996	1.995-4.316	0.005-0.009
Caurie	$\ln m = \ln A - r a_w$	0.967-0.983	4.220-5.675	0.010-0.012
Halsey	$a_w = \exp(-a/m^n)$	0.902-0.942	8.156-10.434	0.023-0.030
Henderson	$\ln m = \ln \frac{\ln \left[ \frac{a}{(1 - a_w)} \right]}{B} - \frac{\ln A}{B}$	0.989-0.992	2.576-3.521	0.006-0.009
Chung-Pfost	$\ln a_w = -A \times \exp(-B \times m)$	0.983-0.996	1.994-4.316	0.005-0.009

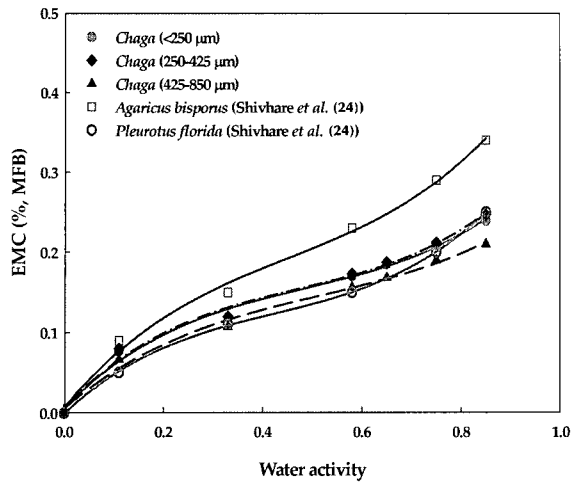


Fig. 2. Isotherms curves for different species of mushrooms.

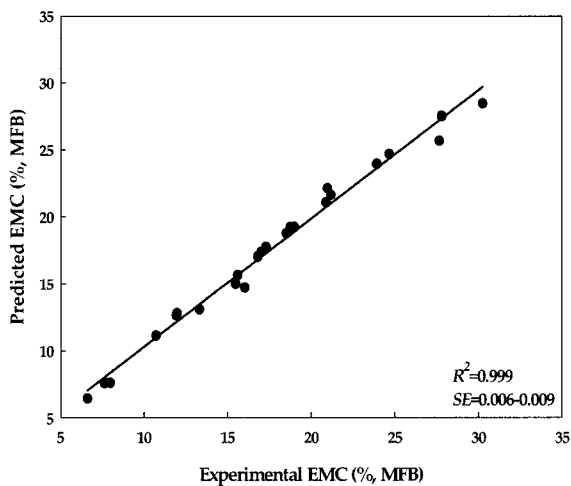


Fig. 3. Correlation between the values predicted by the Henderson model and observed values of the equilibrium moisture content (EMC) of *chaga* mushroom powders.

foods, the EMC of *chaga* mushroom powder increased with water activity in all samples. This may be due to the fact that vapor pressure of water present in foods increases with the pressure of the surroundings (24-27). Additionally, when compared to another species of mushroom, *chaga* was found to be less hygroscopic than *Agaricus* in the experimental range studied. For example, the EMC of *Agaricus* ranged from 9.50 to 34.99% (MFB) while that of *chaga* varied from 7.05 to 30.25% (MFB) (Fig. 2). This variation in sorption behavior between different species of mushrooms has been previously reported and may be attributed to the differences in the chemical composition of mushrooms (28).

Although moisture content at equilibrium was significantly lower in the largest particle size samples ( $p < 0.05$ , Fig. 1), no marked difference in visual appearance existed between the three samples; similar to results reported in a study of green tea powder (29). This difference in moisture content is probably due to the greater surface area available for moisture adsorption on small particles. Isotherm curves are typically sigmoidal in shape (Type II

Table 2. Estimated sorption model constants and monolayer moisture content for *chaga* mushroom powders based on particle size

Model	Particle size ( $\mu\text{m}$ )		
	<250	250-425	425-850
BET			
$c$	-106.386	-96.037	689.714
$m_0$	0.077	0.077	0.070
$R^2$	0.951	0.970	0.979
$E$	10.870	8.772	7.795
$SE$	0.018	0.014	0.010
GAB			
$c$	19.638	24.661	19.824
$k$	0.614	0.674	0.667
$m_0$	0.121	0.111	0.101
$R^2$	0.970	0.976	0.960
$E$	2.909	2.677	3.138
$SE$	0.006	0.006	0.009

according to the BET classification), which is typical of many sorption isotherms of food materials, however, when compared to *Pleurotus*, equilibrium moisture increased slowly with  $a_w$  (Fig. 2).

Sorption models presented in Table 1 were tested for their effectiveness in describing the isotherms of mushroom powders. The results of nonlinear regression analysis fitting the sorption equations to the experimental data are shown in Table 2 and 3. These models were selected based on their effectiveness at describing the sorption isotherms of several foods as well as simplicity of computation. The regression coefficients were computed using the least squares technique and their respective coefficient of determination ( $R^2$ ),  $E$ , and  $SE$  values for each model are also reported. Mean relative percentage error ( $E$ ), which is generally considered good when  $< 10\%$  (30, 31), ranged from 2.576-3.521% in this study, the lowest being obtained when the Henderson model was used. The Henderson model also showed the best fit with respect to  $R^2$  (0.989-0.992) and  $SE$  (0.006-0.009%). The experimental EMC values were related to the computed values from Henderson's model as shown in Fig. 3. The coefficient of determination and standard error for the linear regression were 0.999 and 0.006-0.009, respectively. Other models, including the Chung-Pfost, Bradley, and Oswin models, also satisfactorily predicted the equilibrium moisture content of the mushroom. Shivhare *et al.* (24) reported that the Chung-Pfost model showed the best fit for the moisture adsorption isotherms of *Agaricus bisporus* and *Pleurotus florida*; while moisture desorption isotherms of *Morchella esculenta* were best described using the GAB and Ratti equations (20).

The Brunauer-Emmett-Teller (BET) and Guggenheim-Anderson-de Boer (GAB) equations were used to calculate monolayer moisture content ( $m_0$ ) (Table 2). The monolayer content is the amount of moisture adsorbed at specific sites according to the BET theory (32). When the BET model

**Table 3. Estimated sorption model constants for chaga mushroom powders based on particle size**

Model	Particle size ( $\mu\text{m}$ )		
	<250	250-425	425-850
<b>Kuhn</b>			
$b$	6.782	6.594	6.311
$a$	-58.709	-56.147	-61.670
$R^2$	0.723	0.795	0.809
$E$	19.622	17.811	18.267
$SE$	0.040	0.036	0.031
<b>Oswin</b>			
$\ln a$	-1.897	-1.880	-2.103
$n$	0.275	0.285	0.298
$R^2$	0.965	0.987	0.983
$E$	6.066	4.124	5.061
$SE$	0.014	0.009	0.009
<b>Bradley</b>			
$\ln K_2$	2.219	2.046	1.936
$\ln K_1$	-16.828	-15.445	-16.773
$R^2$	0.983	0.996	0.990
$E$	4.316	1.995	2.758
$SE$	0.009	0.005	0.006
<b>Caurie</b>			
$\ln A$	-2.629	-2.635	-2.801
$r$	1.460	1.506	1.573
$R^2$	0.967	0.983	0.975
$E$	4.854	4.220	5.675
$SE$	0.011	0.100	0.012
<b>Halsey</b>			
$\ln a$	-5.313	-5.250	-5.327
$n$	2.529	2.524	2.394
$R^2$	0.902	0.942	0.932
$E$	10.434	8.156	10.163
$SE$	0.030	0.023	0.023
<b>Henderson</b>			
$\ln A$	-0.696	-0.702	-0.791
$B$	2.432	2.377	2.264
$R^2$	0.990	0.989	0.992
$E$	2.576	3.521	2.987
$SE$	0.006	0.008	0.009
<b>Chung-Pfost</b>			
$B$	-16.828	-15.445	-16.773
$\ln A$	2.129	2.045	1.936
$R^2$	0.983	0.996	0.990
$E$	4.316	1.994	2.758
$SE$	0.009	0.005	0.006

was used, the mean monolayer values were 0.077, 0.077, and 0.070 H<sub>2</sub>O/dry solid for <250, 250-425, and 425-850  $\mu\text{m}$  sized particles, respectively, however when the GAB model was used, the mean values were 0.121, 0.111, and 0.101 H<sub>2</sub>O/dry solid, respectively. Although there was no clear relationship between  $m_o$  and particle size, it appeared to decrease with increased particle size, probably due to the larger surface area of the smaller particle-sized samples. It should be noted that  $m_o$  values obtained using the GAB equation were higher than those obtained using the BET model, which is in agreement with results obtained by others (33-36).

This study demonstrates that *chaga* mushroom powder belongs to a class of food powders that are highly hygroscopic by nature. The information obtained from this study, particularly regarding the monolayer moisture content, will help determine which packaging systems should be used for *chaga* powder to provide maximum product protection.

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