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# Browning, Starch Gelatinization, Water Sorption, Glass Transition, and Caking Properties of Freeze-dried Maca (*Lepidium meyenii* Walpers) Powders

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**Abstract: The browning, gelatinization of starch, water sorption, glass transition, and caking properties of freeze-dried maca (*Lepidium meyenii* Walpers) powders were investigated and compared with a commercial maca powder. The freeze-dried maca powders had lower optical density (browning) and higher enthalpy change for starch gelatinization than the commercial maca. This resulted from a difference in thermal history. The equilibrium water contents of the freeze-dried maca powders were higher than those of commercial maca at each water activity ($a_w$) because of differences in amorphous part. The glass transition temperature ($T_g$) was evaluated by differential scanning calorimetry. There was a negligible difference in the anhydrous $T_g$ (79.5–80.2 ºC) among the samples. The $T_g$-depression of freeze-dried maca powders induced by water sorption was more gradual than that of the commercial maca due to a difference in water insoluble material content. From the results, critical water activity ($a_{wc}$) was determined as the $a_w$ at which $T_g$ becomes 25 ºC. There was negligible caking below $a_w$ = 0.328. At higher $a_w$, the degree of caking remarkably increased with a large variation depending on the samples. The degree of caking could be described uniformly as a function of $a_w/a_{wc}$. From these results, we propose an empirical approach to predict the caking of maca powders.**

**Key words: maca, water sorption isotherm, glass transition temperature, caking, gelatinization**

## INTRODUCTION

Maca (*Lepidium meyenii* Walp.), a carbohydrate-rich vegetable root of the Brassicaceae family, is native to the high plateau of the central Andes of Peru and is one of the few vegetables adapted to high altitudes (3,800–4,500 m) and severe environmental conditions.[1)] The maca root comes in several colors such as yellow, white, purple, and black. Although the phenotypic color does not considerably affect the primary nutritional composition (carbohydrate, protein, and fat),[2)3)4)] secondary metabolites such as glucosinolates, macaenes, and macamides have been reported to variate.[3)5)6)7)8)] The consumption of maca has notably increased all over the world because of interest in its bioactive compounds.[2)9)]

There has been little effort to understand the physical properties of maca in comparison with its chemical and physiological properties. Maca powder is a multicomponent powder, and the amorphous part shows a glass-to-rubber transition (glass transition) upon changes in temperature and water content.[10)] Glass transition is characterized by the glass transition temperature ($T_g$).[11)] Amorphous food powders in the glassy state ($T < T_g$) are physically stable because of an extremely low molecular mobility and/or high viscosity.[12)] The $T_g$ of hydrophilic food materials decreases with increasing water content, because water acts as a plasticizer to the amorphous part. The effect of water content on $T_g$ has been described as the $T_g$-curve. From the $T_g$-curve, the water content at $T_g$ = 25 ºC (as a typical ambient temperature) can be determined as critical water content ($w_c$). In addition, $w_c$ can be converted to critical water activity ($a_{wc}$) through the relationship between equilibrium water content and $a_w$.[13)] Glass transition occurs at 25 ºC when water content and $a_w$ become higher than $w_c$ and $a_{wc}$, respectively. The $w_c$ and $a_{wc}$ parameters are useful for the prediction of physical deteriorations induced by water sorption at ambient temperature. For example, the caking of amorphous food powders can be understood based on the $w_c$ and/or $a_{wc}$.[14)15)16)] Caking is an undesired physical phenomenon in which free-flowing powders agglomerate into hard lumps due to deformation and bridging of sticky particles as a result of plasticization and the decrease of surface viscosity.[14)16)]

In our previous study, the glass transition and caking properties of a commercial maca powder cultivated in Peru were investigated.[10)] We found that the maca powder showed an extended glass transition, reflecting a wide distribution of molecular mobility. In addition, the degree of

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Abbreviations: $a_w$, water activity; $a_{wc}$, critical water activity; $w_c$, critical water content; DSC, differential scanning calorimetry; $T_g$, glass transition temperature; DM, dry matter.


**Fig. 1.** Maca root phenotypes.

caking drastically increased at an $a_w$ under the rubbery state. Since maca powder showed extended glass transition behavior, the molecular mobility required for caking was thought to have been provided gradually by the increase in $a_w$ under the rubbery state. The commercially available maca powder, however, had an unclear thermal history. Sliced maca is air-dried, powdered, and sterilized in different ways depending on the manufacturer.[1)4)6)7)] During processing, it is thought that the gelatinization of starch and browning progressively occur. These factors will affect the physical properties of the amorphous powders.

The purpose of this study was to understand browning, starch gelatinization, water sorption isotherm (relationship between equilibrium water content and $a_w$), $T_g$-curve, and caking behavior of freeze-dried maca powder. The freeze-dried maca powder was prepared from fresh maca roots cultivated in Japan. Since freeze-drying enables the drying of maca non-thermally, it is expected that browning and starch gelatinization do not occur during the processing.

## MATERIALS AND METHODS

***Materials.*** Fresh maca roots cultivated in Japan were purchased from the Maca Japan Corp., Tokyo, Japan. The roots were visually classified into yellow, white, purple, and black as shown in Fig. 1. For comparison, the commercial maca powder employed in our previous study[10)] was also used for some experiments.

***Sample preparation.*** The maca roots were peeled manually and cut into small portions. The slices were distributed in an aluminum container and frozen in a freezer at −20 ºC. The frozen samples were then transferred to a pre-cooled chamber and freeze-dried at a pressure of approximately 11 Pa as the temperature increased from −35 ºC to 5 ºC in a constant stepwise manner over a 48-h period. The freeze-dried solids were powdered using a mixer and stored below 5 ºC prior to use.

***Extent of browning.*** The freeze-dried maca and commercial maca powders were diluted to 0.05 g/mL with distilled water. The samples were centrifuged at approximately 1,000 × $G$ for 10 min, and the supernatant was filtered with a filter paper (pore size = 4 μm). Optical density at 420 nm ($OD_{420}$) was evaluated for the supernatant using a UV-visible spectrophotometer (Biospec-1600; Shimadzu Co., Kyoto, Japan). In this way it was possible to quantify pigments produced by either enzymatic[17)] or non-enzymatic browning.[18)] The measurements were performed in triplicate and the results were averaged.

***Enthalpy change for starch gelatinization.*** To confirm the gelatinization properties of starch in maca, gelatinization temperature ($T_{gel}$) and enthalpy change ($\Delta H_{gel}$) were investigated using differential scanning calorimetry (DSC 60, Shimadzu Instruments Inc., Kyoto, Japan). Alumina powder was used as a reference, and temperature and heat flow were calibrated with indium and distilled water, respectively. Freeze-dried maca powder samples (4.8–6.0 mg) were placed into a DSC aluminum pan, and the water content adjusted above 70 % (w/w) by adding distilled water.[19)] The DSC measurements were obtained at 5 ºC/min in a temperature range of 10 to 100 ºC. The measurements were performed in triplicate and the results were averaged.

***Water sorption isotherm.*** Freeze-dried maca powder (approximately 0.5 g) was placed in an aluminum dish (diameter = 40 mm), and the residual moisture in the samples was removed by vacuum drying at 25 ºC (stage temperature) for 6 h. The dried samples were equilibrated at 25 ºC for longer than 7 days in a desiccator with saturated salts: LiCl ($a_w$ = 0.113), $CH_3COOK$ ($a_w$ = 0.225), $MgCl_2$ ($a_w$ = 0.328), $K_2CO_3$ ($a_w$ = 0.432), $Mg(NO_3)_2$ ($a_w$ = 0.529), NaBr ($a_w$ = 0.576), KI ($a_w$ = 0.688), and NaCl ($a_w$ = 0.753). The equilibrium water content of the samples was determined gravimetrically by oven-drying at 105 ºC for 16 h. The measurements were performed in triplicate for freeze-dried yellow maca powder and singly for the freeze-dried white, purple, and black maca powders.

***Glass transition temperature.*** The glass transition temperature ($T_g$) of the samples was determined using the DSC as mentioned above. Each sample (20–30 mg) was hermetically sealed into a DSC aluminum pan. To evaluate the $T_g$ of the anhydrous sample, it was vacuum-dried at 60 ºC for 6 h, placed into a DSC aluminum pan, and subsequently oven-dried at 105 ºC for 2 h. The DSC pan containing the fully dried sample was then hermetically sealed. The DSC measurements were obtained at 5 ºC/min. To reset the thermal history of the glassy samples, the DSC scan was repeated two times.[20)] In the first scan, the DSC measurement was stopped at a slightly higher temperature than the endothermic shift expected as the glass transition, and then cooled down. In the second scan, the DSC measurement was stopped at a much higher temperature than the $T_g$ suggested by first scan. The DSC thermogram was analyzed using software interfaced with the DSC, and the $T_g$ values

were determined from the onset of the endothermic shift. The measurements were performed in triplicate for freeze-dried yellow maca powder and singly for the freeze-dried white, purple, and black maca powders.

***Degree of caking.*** The degree of caking was investigated as described previously[21] with minor modifications.[10)22)] The freeze-dried maca powder samples were separated through a 1.4-mm sieve with a vibration amplitude of 4.5 mm using a mechanical shaker (MVS-1; As One Instruments Co. Tokyo, Japan). The passed powder (approximately 0.5 g) was placed in an aluminum dish (diameter = 40 mm) and vacuum-dried at 25 ºC for 6 h. The powder was equilibrated at 25 ºC for 7 days in a desiccator with saturated salts as mentioned above. The equilibrated samples were vacuum-dried at 25 ºC for 6 h. The dried samples were weighed and then sieved under the same conditions as the first treatment. After weighing the amount of sample retained on the sieve, the degree of caking was evaluated as a percentage of retained weight per pre-sieved weight. The measurements were performed in triplicate for freeze-dried yellow maca powder and singly for the freeze-dried white, purple, and black maca powders.

## RESULTS AND DISCUSSION

***Browning and starch gelatinization.***

The values of $OD_{420}$, starch gelatinization temperature, and gelatinization enthalpy are shown in Table 1. Notably, the commercial maca powder had a significantly higher $OD_{420}$ than the freeze-dried samples. This was a consequence of air-drying and thermal sterilization during processing.[23)24)] Interestingly, the freeze-dried purple and white maca powders showed slightly higher $OD_{420}$ values than the yellow and black ones. It is known that the carbohydrates in maca consist of 23 % starch, 19 % sugars, 9 % water-soluble fiber, and 23 % water-insoluble fiber.[25)] During thermal processing, some enzymes will contribute to non-enzymatic browning. For example, glucosinolates are hydrolyzed by endogenous myrosinases into glucose,[6)7)] and structural carbohydrates are hydrolyzed into low molecular weight sugars by endogenous amylase, pectinesterase, and polygalacturonase.[26)] Consequently, reducing sugars will react with the amino groups of proteins. Although there is a lack of evidence for the presence of enzymes responsible for enzymatic browning (i.e. polyphenol oxidase and peroxidase) in maca, this should still be considered. Other plants of the Brassicaceae such as rapeseed flour[27)] and cress[28)] manifest polyphenol oxidase and peroxidase. Since maca has a high polyphenol content,[9)29)] the bruised root may initiate enzymatic browning during processing.

Typical DSC thermograms for maca suspensions are shown in Fig. 2. A clear endothermic peak due to starch gelatinization was observed, and $T_{gel}$ and $\Delta H_{gel}$ were determined from the onset point and peak area, respectively.

There were negligible differences in the $T_{gel}$ values among the samples (Table 1). However, the $\Delta H_{gel}$ values depended on the samples; the values were higher in the order of yellow > black > purple > white > commercial. When the starch content was assumed to be 23 % according to a previous study,[25)] the $\Delta H_{gel}$ values for the freeze-dried maca samples ranged between 8.8 and 17.8 J/g dry starch. This range agrees roughly with that (6.2–14.6 J/g dry starch) of previous reports.[25)30)31)32)] In a comparison of the $\Delta H_{gel}$ values between the freeze-dried maca and commercial one, we found that the $\Delta H_{gel}$ of commercial maca was much lower than that of freeze-dried maca (Table 1). Partial gelatinization and enzymatic hydrolysis induced by endogenous amylases[26)] occur during air-drying and thermal sterilization. This interpretation is supported by the fact that commercial maca had a much higher $OD_{420}$ than freeze-dried maca samples.

**Table 1.** Browning and gelatinization properties of maca powders.

| | $OD_{420}$ | $T_{gel}$ (ºC) | $\Delta H_{gel}$ (J/g-DM) |
|---|---|---|---|
| White | 0.28 ± 0.02 | 49.7 ± 0.3 | 2.0 ± 0.2 |
| Purple | 0.32 ± 0.01 | 49.2 ± 0.2 | 2.4 ± 0.1 |
| Black | 0.20 ± 0.01 | 50.0 ± 0.2 | 3.5 ± 0.1 |
| Yellow | 0.25 ± 0.03 | 47.7 ± 0.4 | 4.1 ± 0.2 |
| Commercial | 0.64 ± 0.01 | 48.4 ± 0.9 | 1.0 ± 0.1 |

***Water sorption isotherm of freeze-dried maca powder.***

The water sorption isotherm (effect of $a_w$ on the equilibrium water content) of maca powders is shown in Fig. 3. For comparison, the data for commercial maca (except for the equilibrium water content at $a_w$ = 0.688) was taken from our previous study;[10)] the equilibrium water content at $a_w$ = 0.688 was determined additionally in this study in order to match the datapoints to the others. The equilibrium water content of the maca powders increased sigmoidally with increase of $a_w$, which is typical behavior for amorphous food powders.[33)] The water sorption isotherms were analyzed using the Guggenheim, Anderson, and De Boer (GAB) equation (Eq. (1)),

$$W=\frac{W_m CKa_w}{(1-Ka_w)(1+(C-1)Ka_w)} \quad (1)$$

where $W$ is equilibrium water content (g/100 g-DM, dry matter), $W_m$ is amount of water strongly adsorbed to specific sites at the material surface (g/100 g-DM), and $C$ and $K$ are factors that correct the sorption properties of the monolayer with respect to the bulk liquid and the properties of the multilayer with respect to the bulk liquid, respectively.[34)] The $W_m$, $C$, and $K$ were determined by a quadratic regression approach.[35)] The GAB model (Eq. (1)) can be rearranged to Eq. (2),

$$\frac{a_w}{W}=\alpha a_w^2+\beta a_w+\gamma \quad (2)$$

where $\alpha = [K(1 - C)]/[W_m C]$, $\beta = (C - 2)/(W_m C)$, and $\gamma = 1/(W_m CK)$. Three parameters ($\alpha$, $\beta$, and $\gamma$) were determined from the quadratic regression curve ($a_w/W$ *versus* $a_w$ plot), and $W_m$, $C$, and $K$ were calculated (Table 2).

The $W_m$ values of freeze-dried maca powders were similar (6.90–7.29 g/100 g-DM), but slightly higher than that

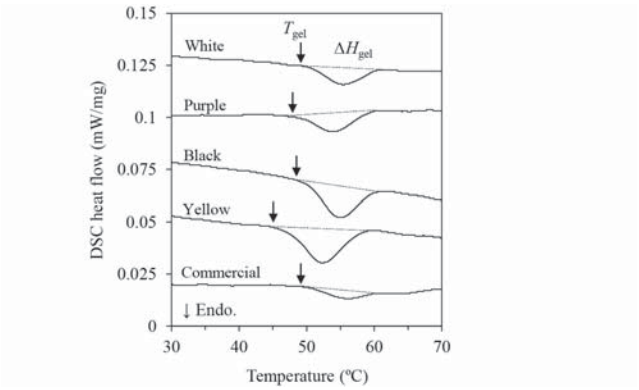


**Fig. 2.** Typical DSC thermograms of maca suspensions. Endothermic peak indicates the starch gelatinization.

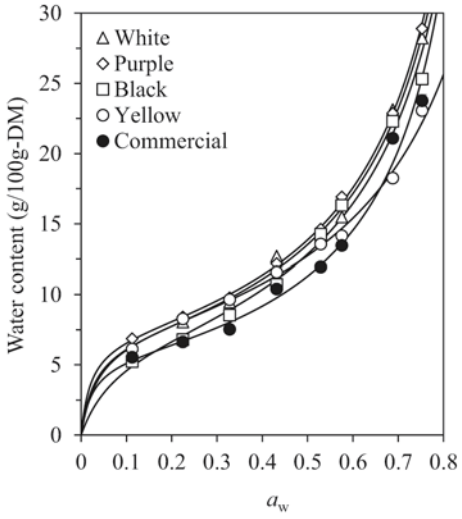


**Fig. 3.** Water sorption isotherm for maca powder samples at 25 ºC. Solid lines were obtained by the GAB fitting.

**Table 2.** GAB parameters of maca powders.

| | $W_m$ (g/100g-DM) | $C$ | $K$ | $R^2$ |
|---|---|---|---|---|
| White | 6.90 | 34.21 | 1.009 | 0.996 |
| Purple | 6.97 | 47.14 | 1.014 | 0.999 |
| Black | 6.91 | 13.28 | 1.000 | 0.988 |
| Yellow | 7.29 | 31.15 | 0.899 | 0.996 |
| Commercial | 5.54 | 40.31 | 1.041 | 0.985 |

of commercial maca powder (5.54 g/100 g-DM). The $W_m$ (monolayer water content) corresponds to the number of surface hydration sites of the powders. Commercial maca had a lower $\Delta H_{gel}$, and thus higher $W_m$ was expected in comparison to the freeze-dried maca; the larger the gelatinization, the more hydration sites are expected. The reason why commercial maca has lower $W_m$ than freeze-dried maca samples is the difference in water insoluble material content (e.g., fiber). Maca root including husk (outer skin) is traditionally dried and then powdered,[4)7)] so the water insoluble materials of the husk will reduce the hydration of commercial maca powder. There were minor differences in the $K$ values (0.899–1.041) among the samples, including the commercial maca. The fact that $K$ is near 1 indicates that the water molecules in multiple layers have similar characteristics to bulk water.[34)] The $C$ values, on the other hand, deviated largely between 13.28 and 47.14. This can be interpreted as the larger the $C$ value, the stronger the bonds between water molecules in the monolayer and the binding sites on the surface of the powder.[34)] Realistically, a small deviation in the experimental data has been amplified to result in a large difference in the $C$ values.[36)37)] The GAB parameters were used to transform water content to $a_w$ in the following section.

***Glass transition behavior of maca powder.***

Typical DSC thermograms for maca powders ($a_w$ = 0.328) are shown in Fig. 4. A clear endothermic shift due to glass transition was observed, and $T_g$ was determined from the onset point as indicated by arrows.

The effect of water content on the $T_g$ for maca powders is shown in Fig. 5. The data for commercial maca (except for the $T_g$ at $a_w$ = 0.529) was taken from our previous study;[10)] the $T_g$ value at $a_w$ = 0.529 was determined additionally in this study in order to match the datapoints to the others. The anhydrous $T_g$ was almost equivalent among the samples (79.5–80.2 ºC), though physical parameters (gelatinization enthalpy, browning, and water sorption isotherm) were slightly different among the samples. From the composition data of maca,[25)] the amorphous part will be attributed mainly to low molecular weight carbohydrates. For instance, the anhydrous $T_g$ is 68 ºC for sucrose and 90 ºC for maltose.[20)] The $T_g$ decreased with the increase in water content because of the water plasticizing effect. In addition, the $T_g$-variation at each water content became slightly broad with the increase in water content. At each water content (except for the anhydrous state), commercial maca tended to have lower $T_g$ than freeze-dried maca samples. As mentioned above, commercial maca had a lower $W_m$ than freeze-dried maca samples because of a larger amount of water-insoluble material. The high content of water insoluble material in commercial maca makes it less hydrophilic than the freeze-dried maca samples. Since glass transition occurs in the hydrophilic part of the powder, the $T_g$ will be significantly depressed by even a small increase in water content.[38)]

The effect of water content on the $T_g$ was analyzed by the Gordon-Taylor (GT) equation (Eq. (3))

$$T_g = \frac{(1-X_w)T_{g(as)} + kX_wT_{g(w)}}{(1-X_w) + kX_w} \quad (3)$$

where $X_w$ is weight fraction of water, $T_{g(as)}$ and $T_{g(w)}$ are $T_g$ for anhydrous samples and water, respectively, and $k$ is a constant. The $T_{g(w)}$ (136 K) was obtained from previous reports.[39)40)] The $T_{g(as)}$ was determined experimentally, and thus $k$ was treated as a fitting parameter. The GT parameters ($T_{g(as)}$ and $k$) are listed in Table 3.

The $k$ value indicates the sensitivity of $T_g$-depression caused by the water plasticizing effect; the higher the $k$ value, the greater the water content dependence of $T_g$.[41)] The values of $k$ varied between 4.17 and 4.87. The values were near those of glucose ($k$ = 4.52), sucrose ($k$ = 5.42), and starch ($k$ = 5.2) as reported by Roos (1995).[42)] From

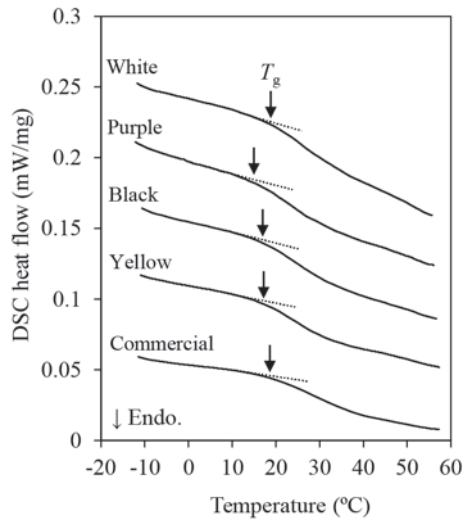


**Fig. 4.** Typical DSC thermograms of the second scan of maca powders at $a_w = 0.328$.
Arrows indicate the $T_g$.

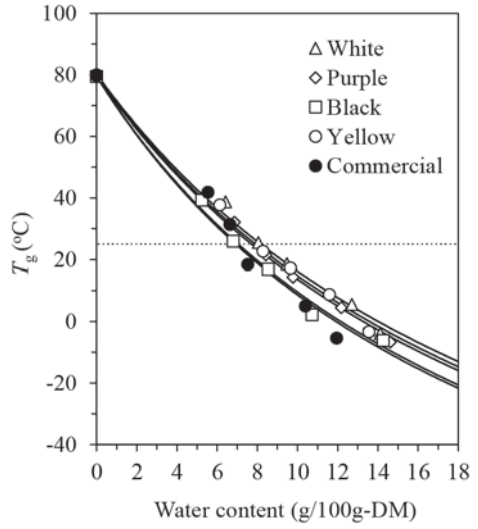


**Fig. 5.** Effect of water content on $T_g$ of maca powders.
The $T_g$-curves were obtained by the GT fitting.

the $T_g$-curves, $w_c$ was determined as the water content at which $T_g$ becomes 25 ºC (a typical ambient temperature). The $w_c$ was converted to critical water activity ($a_{wc}$) through GAB analysis (Fig. 3 and Table 2). Glass transition occurs at 25 ºC when water content and $a_w$ become higher than $w_c$ and $a_{wc}$, respectively. The $w_c$ and $a_{wc}$ values are practically important parameters for the physical stability of dry food powders at 25 ºC. The higher the $w_c$ and $a_{wc}$ values, the greater the resistance to caking of amorphous powder induced by water sorption.[13)16)] These results are also listed in Table 3.

The values of $w_c$ varied from 6.92–8.16. The commercial maca had the lowest $w_c$ among the samples. This was due to the higher $k$; the $T_g$ was remarkably depressed by the increased water content because of an enhanced susceptibility provided by the greater water-insoluble material content.[38)] The $a_{wc}$ varied in the range of 0.174–0.243. The commercial maca showed the lowest $w_c$ but the highest $a_{wc}$. As shown in Fig. 3, the commercial maca showed lower equilibrium water content than freeze-dried maca powders at each water content because of differences in water insoluble material content.

**Table 3.** $T_{g(as)}$, $k$, $w_c$, and $a_{wc}$ of maca powders.

| | $T_{g(as)}$ (ºC) | $k$ | $R^2$ | $w_c$ (g/100g-DM) | $a_{wc}$ |
|---|---|---|---|---|---|
| White | 80.2 | 4.17 | 0.993 | 8.16 | 0.228 |
| Purple | 79.8 | 4.39 | 0.998 | 7.71 | 0.174 |
| Black | 79.5 | 4.77 | 0.994 | 7.04 | 0.223 |
| Yellow | 80.0 | 4.29 | 0.995 | 7.90 | 0.210 |
| Commercial | 79.7 | 4.87 | 0.972 | 6.92 | 0.243 |

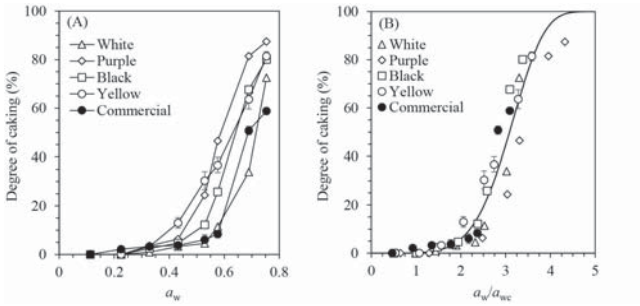


**Fig. 6.** Effect of $a_w$ (A) and $a_w/a_{wc}$ (B) on the degree of caking of maca powders.
The solid line (B) was obtained by the stretched exponential function fitting.

### *Caking behavior of freeze-dried maca samples.*

The effect of $a_w$ on the degree of caking for the freeze-dried maca samples is shown in Fig. 6A. The data for commercial maca was taken from our previous study.[10)] The degree of caking for the maca powders was almost negligible below $a_w = 0.328$. At higher $a_w$, the degree of caking remarkably increased with a large variation depending on the samples.

As mentioned above, the caking of amorphous powders is strongly affected by glass transition. In order to understand the degree of caking of the maca samples as a function of glass transition, $a_w$ was normalized by the $a_{wc}$ (Fig. 6B). It is known that the degree of caking for maltodextrin is negligible below $a_{wc}$, but it increases drastically up to 100 % higher than the $a_{wc}$ value.[10)] In contrast, the degree of caking for commercial maca increased progressively with increase in $a_w$. As discussed previously, maltodextrin shows a homogeneous glass transition, and thus a drastic increase in molecular mobility will occur just above $a_{wc}$. Maca powder, on the other hand, shows a broad glass transition, and thus molecular mobility increases gradually above $a_{wc}$. From Fig. 6B, we see that the degree of caking of maca samples increased drastically up to 88 % at conditions higher than $a_w/a_{wc} = 2.0$. This behavior was independent from the type of maca. This indicates that the large variation of the degree of caking observed above $a_w = 0.432$ is due to differences in $a_{wc}$.

The effect of $a_w/a_{wc}$ on the degree of caking was analyzed by the stretching exponential function (Eq. (4)).

$$\text{Degree of caking}(\%)=100\left\{1-\exp\left[-\alpha\left(\frac{a_w}{a_{wc}}\right)^n\right]\right\} \quad (4)$$

where $\alpha$ and $n$ are constants depending on the system. From the fitting analysis, $\alpha = 1.26\times10^{-3}$ and $n = 5.60$ were obtained ($R^2 = 0.920$). This equation is mathematically equivalent to the Avrami equation. The Avrami model describes effect of annealing time on the degree of crystallization at a constant temperature.[43] Given that crystallization is an orderly aggregation of particles, the Avrami equation is analogically applicable for the caking (agglomeration) of powders. A novel modification of the proposed equation (Eq. (4)) is that the Avrami equation was changed from "time-dependency" of crystallization to "$a_w$-dependency" of caking. The present equation will be useful for predicting the caking of maca powder from the viewpoint of $a_{wc}$. It is important to understand the applicability of the model in other types of food powders.

## CONFLICTS OF INTEREST

The authors declare no conflict of interests.

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