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Effects of Particle Size and Gelatinization of Job's Tears Powder on the Instant Properties

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Abstract

The effects of particle sizes (small, medium and large sizes) and gelatinization treatment on the changes of the instant properties of Job's tears powder were investigated. The degree of gelatinization on the different particle size samples of Job's tears powder was the highest in the small particle size, and it also showed an increasing trend regardless of pregelatinizing whether it is or not as the particle size decreased from large particle size to small particle size. The water solubility index of the pregelatinized samples was high compared to that of ungelatinized samples regardless of particle size and temperatures. The water absorption and swelling power increased as particle size and temperature were increased. The dispersibility and sinkability of ungelatinized sample was increased as particle size and temperature were increased and it also showed lower value regardless of particle size and temperature. However, the dispersibility and sinkability of pregelatinized samples were shown to have the opposite result, such that the smallest particle size of pregelatinized sample had the lowest sinkability (11.3%). The turbidity of the pregelatinized small particle size was the highest by a factor of 1.08.

Key words: particle size, gelatinization, preheating, Job's tears powder, instant properties

INTRODUCTION

Consumption of powdered beverages such as cocoa, coffee, tea, barley powder and Job's tears has been increasing. The quality of these powdered beverages is very important for the improvement of instant property parameters (1). The parameters that determine the properties of agglomerates also include properties related to primary particles, such as particle size, size distribution, shape, and surface area. The instantaneous properties of agglomerate are most desirable properties of agglomeration process and they can be measured by wettability, sinkability, dispersibility, and solubility (2). If a powder is spread on the surface of liquid, the following process takes place: (a) penetration of liquid into the porous system due to capillary action (the ability of powder to be penetrated by the liquid is called "wettability"), (b) sinking of the particles below the liquid surface ("sinkability"), (c) dispersion of powder with little stirring ("dispersibility"), (d) solution of the particles in the liquid, provided the particles are soluble ("solubility"). The properties such as wettability, sinkability, dispersibility, and solubility are subsumed under the term "instant properties" (3). Some factors that affect instant properties include the dispersion of liquid, powder composition, and powder particle size. Particularly, particle size of powder critically affects the instant properties (4,5).

Ultra fine pulverization is a method used to improve instant properties of foods like tea. This is a superfine milling process that has been effectively used for the fractionation of various grains and increased their utilization (6,7). During the milling process, mechanical energy is applied to food material, and particle size of the material is reduced, resulting in changed physical properties of the material (8,9).

In Korea, Job's tears tea is one of the most widely consumed beverages, whose popularity is still increasing and whose health-enhancing properties are being researched and reported (10,11). Consumption of Job's tears tea has been made convenient through the use of an automatic vending machine; however, Job's tears powder is not completely melted in warm water in the limited time it is prepared in an automatic vending machine. So, the sedimentation of commercial Job's

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tears powder of large particle size (over 120 μm) makes an unfavorable impression to consumers. The current studies for improving the instant properties of Job's tears powder for Job's tears powder tea are not sufficient for determining the best preparation method for automatic vending. Moreover, commercial Job's tear powders were prepared by pregelatinization before grinding. However, the influence of pregelatinization on instant properties of Job's tears powder is not yet clear.

Therefore, the effects of particle size and pregelatinization on instant properties of Job's tears powder were investigated and the basic data for sedimentation of commercial large particle size of Job's tears powder in melting has been provided.

MATERIALS AND METHODS

Sample preparations

Job's tears (*Coix lachrymajobi*) beans were purchased from the wholesale market of Dae-Jeon agricultural & marine products in Daejeon, Korea, 2008. Commercial Job's tears powder was used as a control sample (Joong-Ang Food Industry Co., Okchen, Korea). The commercial Job's tears powder was prepared by the following method. One hundred kilograms of Job's tears beans were washed under running water and soaked for 1 hr. Then the Job's tears beans were steamed for 30 min at 140°C and cooled to room temperature. After the beans were dried for 4 days in a shady place, it was roasted, cooled and ground. The purchased Job's tears beans for testing samples were pulverized by an air mill (model HTM100, Hyun-Joon powtec Co., Hwa-Sung, Korea) to produce such different particle sizes as small, medium and large sizes. The mean values of small, medium, and large particle sizes were 25.4, 28.0, and 32.0 μm, respectively. The first peaks of small, medium, and large particle size samples were 10, 50, and 120 μm, respectively (Fig. 1).

Pregelatinization of Job's tears powder

To prepare the pregelatinized Job's tears powders samples, pulverized Job's tears powder suspensions (40%, w/v) were prepared by combining of Job's tears powder and distilled water in glass bottle. Each Job's tears powder suspension was pregelatinized by being autoclaved under 121°C for 30 min (1.5 atm). Then, the samples were subsequently frozen in a freezer at a temperature of -70°C and freeze-dried in a freeze dryer (Ilshin Co., Gyeonggi-do, Korea) at -50°C and 1.33 Pa. After freeze drying, the sample was tested through a 100 mesh (150 μm) sieve.

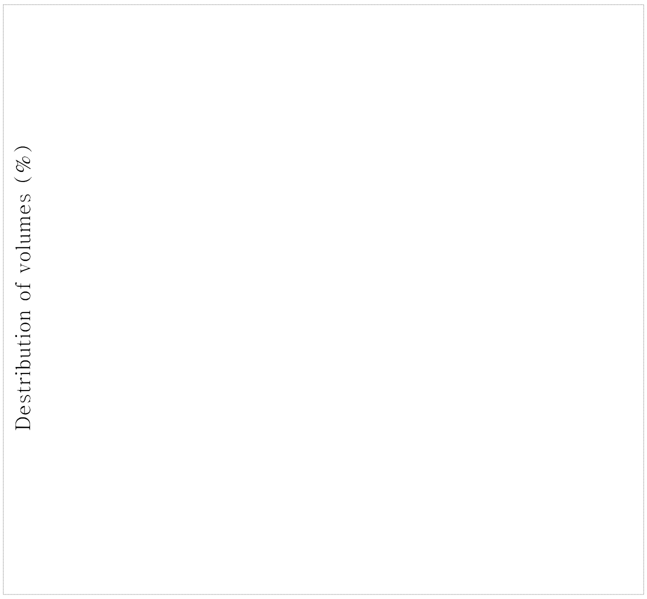

Fig. 1. Particle size distribution of Job's tears powder.

Particle size analysis

Particle size distribution of the powder was measured by laser particle size analysis using a Beckman-Counter LS 100 laser diffraction instrument (Beckman, Hialeah, FL, USA). Samples were suspended in isopropyl alcohol and measured for a 1 min time interval.

Degree of gelatinization

Degrees of gelatinization of ungelatinized or pregelatinized Job's tears powder samples were determined by measuring the amount of the reducing sugar that corresponds to the enzymatic degradation of the starch (12). The amount of the reducing sugar was determined through phenol-sulfuric acid assay. Briefly, 20 mg of the powdered sample was dispersed in 5 mL distilled water in a 50 mL centrifuge tube and 25 mL glucoamylase solution (about 20 units) was added. After incubation for 1 hr at 40°C, the reaction was stopped by adding 2 mL of 25% trichloroacetic acid (TCA). After centrifugation of the solution at 16,000 × g for 5 min, 2 mL of a diluted supernatant solution containing between 10～70 mg/mL of sugar was transferred into a test tube. One mL of 5% phenol was added, followed by rapid addition of 5 mL of concentrated sulfuric acid. The tubes were allowed to stand for 10 min, shaken, and placed in a 30°C water bath for 20 min before reading. The absorbance of the characteristic yellow-orange color was measured at 490 nm.

Measurement of instant properties

The water solubility index (WSI), water absorption index (WAI), and swelling power (SP) were measured according to the procedure of Lai and Cheng (13) with some modifications. WSI, WAI, and SP were performed different temperatures at 4, 25, 40, 55, and 70°C. One gram of raw or pregelatinized samples was suspended in 25 mL distilled water in a 50 mL volume centrifuge tube with gentle stirring for 30 min and centrifuged at 27,000×*g* for 30 min. The supernatant was dried at a 105°C dry oven. The WSI, WAI and SP were calculated as the following formulas:

$$\text{WSI }(\%)=\frac{\text{dry weight of supernatant}}{\text{dry weight of sample}}\times 100$$

$$\text{WAI}=\frac{\text{weight of sediment}}{\text{dry weight of sample}}$$

$$\text{SP}=\frac{\text{weight of sediment}}{\text{dry weight of sample}\times(1-\text{WS\%}/\ 100)}$$

The dispersibility was measured according to the procedure of Jinapong et al. (14) with some modifications. Twenty milliliters of distilled water was poured into a 50 mL volume test tube at 4, 25, and 70°C. Then, 1 g of Job's tears powder sample was added into the test tube and stirred for 10 sec. The suspension was poured through an 18 mesh (1000 μm) sieve. The passed suspension was dried at a 105°C dry oven for 10 hrs. The dispersility was calculated as the following formula:

$$\text{Dispersibility }(\%)=\frac{a\times\%\ \text{TS}}{a\times\dfrac{100-b}{100}}$$

where a = dry weight (g) of Job's tears powder
b = moisture content in the Job's tears powder
% TS = dry matter in percentage after passed through the sieve.

The measurement of sinkability experiment was performed at 25°C. One gram of Job's tears powder sample was weighed into the 5 glass dishes (ϕ 2 cm). Five hundred milliliters of distilled water was placed in a beaker and the sample was added at once into the beaker. After 10 min, the suspended samples which were not sunk were removed carefully. Then, the suspension of sample sunk was poured into separate funnel. After another 10 min, twenty-five milliliters of sample sediment suspension was gathered in a tared crucible. The sinkability was calculated as the below formula:

$$\text{Sinkability }(\%)=\frac{\text{dry weight of sediment sample}}{\text{dry weight of sample}}\times 100$$

Turbidity analysis

One gram of Job's tears powder sample was weighed into the 50 mL volume glass test tube. Forty milliliters of distilled water was added into the test tube and was mixed with 2,600 rpm for 10 sec at 90°C in a shaking water bath. The suspension was cooled for 1 hr at room temperature. The turbidity of samples was analyzed using a spectrophotometer (Spectronic 20+, Spectronic Instruments, USA) by measuring the optical density (OD) of sample suspension at A_{660nm}.

Statistical analysis

An analysis of variance (ANOVA) was performed and their differences among the samples were determined by Duncan's multiple range tests using the Statistical Analysis System software (SAS, Cary, NC, USA). The p values<0.05 were considered significant.

RESULTS AND DISCUSSION

Chemical composition of Job's tears powder

The chemical composition of Job's tears samples was shown in Table 1. The crude lipid and ash contents were not significantly different between control samples and tested samples. However, the moisture and crude protein contents were significantly different among different particle sizes of Job's tears samples. The moisture content of control sample was highest (9.7%), compared to test samples. The moisture content of Job's tear sample was increased as the particle size was decreased. The reason is thought that small particle size samples had larger surface area than large particle size samples. In the case of protein contents, the large particle size of Job's tears samples showed the highest protein content among samples.

Distribution of particle size

The particle size distribution of pulverized Job's tears powder is shown in Fig. 1. All pulverized Job's tears powder samples were displayed bi-modal size distribution. The first peak volumes of small, medium and large particle sizes of Job's tears powder were 11.83, 14.20 and 10.78 μm, respectively. The first peak volume of all samples was shown less than 15 μm. Lee et al. (15) reported that since the average diameter of Job's tears starch granule was 10∼20 μm, which was less than 16 μm.

Table 1. The chemical composition of Job's tears powder samples (%)

Constituents	Samples			
	Control	Small	Medium	Large
Moisture	9.7	6.8	6.1	5.9
Crude protein[1)]	15.7	15.2	15.8	17.3
Crude lipid	5.5	5.7	5.9	6.1
Crude ash	1.7	2.1	1.9	1.9
Carbohydrate[2)]	67.4	70.2	70.3	68.8

[1)]Calculation of protein content=N (%)×5.95 (nitrogen factor).
[2)]Calculated by difference.

Although all pulverized Job's tears powder samples had a bi-modal size distribution, volume fraction of each sample was clearly different. The sample of small particle size had a high volume fraction at a size of near 10 μm particle size and samples of medium and large particle sizes were distributed at near 50 μm and 100 μm in X axis of Fig. 1, respectively.

Effect of particle size on the degree of gelatinization

Effects of particle size on the degree of gelatinization of ungelatinized and pregelatinized samples are shown in Fig. 2. Degree of gelatinization of each group of samples was not significantly different ($p>0.05$) between ungelatinized and gelatinized samples. Control samples had higher degree of gelatinization (60.3%) than ungelatinized samples (27.0～33.2%), but there was a lower degree of gelatinization than pregelatinized samples (65.5～71.8%). However, all samples had a trend that particle size decreased as degree of gelatinization increased from small particle size to large particle size. Among samples, small particle size of pregelatinized sample of Job's tears powder showed highest degree of gelatinization by 71.8%. These results suggest the possibility of the effect of particle size on starch gelatinization. Also, in case of pregelatinized samples, the particle size reduction had an increase of surface area and amount of damaged starch; it is easier to hydrate and reduce gelatinization enthalpy (16,17).

Fig. 2. Effect of particle size on degree of gelatinization of Job's tears powder. *Indicates significant difference between control sample and ungelatinization samples ($p<0.05$). †Indicates significant difference between control and pregelatinized samples ($p<0.05$).

Effect of particle size and gelatinization treatment on water solubility index (WSI)

The water solubility index (WSI) of ungelatinized and pregelatinized samples of Job's tears powder is shown in Fig. 3. The WSI of ungelatinization and pregelatinization samples was increased as particle size decreased without the relationship with sample kind at all tested temperatures. The WSI of control sample was the lowest regardless of temperature. Also, WSI of all samples increased as reaction temperature increased. In ungelatinized samples, a steady increase of WSI was observed up to 55°C but the WSI was found to be increased above 55°C. The samples of the ungelatinized small and me-

Fig. 3. Effect of particle size and gelatinization treatment on water solubility index (WSI) of Job's tears powders at different temperatures.

dium particle sizes of Job's tears powder had higher WSI than that of the commercial sample as a control at all temperatures. Moreover, the pregelatinized samples that had high WSI showed a striking contrast to the control samples of different particle sizes at all temperatures. Therefore, pregelatinized samples showed clearer WSI than ungelatinized samples. The WSIs of pregelatinized samples had 2～3 times higher values than those of ungelatinized samples at all temperatures. Among samples, small particle size of pregelatinized particles is the most susceptible to water solubility index (41.6%). The WSI is related to conditions of favoring degradation and fragmentation of starch (18,19). Also, Tanhehco and Ng reported effects of extrusion cooking and milling on WSI of wheat powder (4). According to the report, water solubility is related to the conditions of favoring degradation and fragmentation of starch. They found that the greater starch breakdown resulted in an increase in WSI. Also, the increase of the pregelatinization for Job's tears sample is attributed to the increased numbers of small fragments of amylopectin and amylose by leaching out through opened starch granules, whose structures are changed during hydrothermal treatment. These results indicate that starch granules of pregelatinized samples are more susceptible to hydration. Therefore, WSI increases significantly after treatment of pregelatinization (20). In accordance, both particle size and pregelatinization of Job's tear powder seem to affect WSI. Therefore, the smallest pregelatinized particle size (10 μm) had the highest WSI.

Water absorption index (WAI) and swelling power (SP)

Water absorption index (WAI) and swelling power (SP) of ungelatinized or pregelatinized samples of Job's tears powder are shown in Table 2. The WAI of control sample was higher than that of ungelatinized samples, but it was lower than that of pregelatinized samples. The WAI of all samples increased as particle size increased at all temperatures. The WAI of ungelatinized samples was shown to be lower than that of pregelatinized samples at all test temperatures. The WAI values of ungelatinized samples were the range of 1.81～4.27 and the WAI values of pregelatinized samples were 7.59～12.22. This result is similar to that reported by Tanhehco and Ng (4). They reported that WAI was dependent on the binding of water by hydrophilic groups and on the ability of macromolecules to form gel. When starch is gelatinized, water absorption increases. However, as greater dextrinization and starch fragmentation occurs, WAI then begins to decrease (4,18). In the case of particle size, the difference of WAI may be related to the degradation of starch granule that causes a reduction in the water-holding capacity of molecules as a result of destroying of the starch granule (17). On the other hand, the SP of control sample was higher than that of ungelatinized samples, but lower than that of pregelatinized samples. The SP value of ungelatinized sample was lower than that of pregelatinized sample at all the reaction temperatures. This trend was similar to the WAI result. Low value of WAI sample may be induced to be low SP and large size samples are attributed to enhancement of reassociation of amylose, which results in the change of hydration properties. Moreover, pregelatinized samples were changed in hydration properties by heat treatment. They are shown to have higher SP than ungelatinized Job's tears samples.

Dispersibility

The dispersibility of Job's tears powder resulting from

Table 2. Water absorption index (WAI) and swelling powder (SP) of pulverized Job's tears powders with particle size and different temperatures

Treatment	Particle size	WAI[2)]					SP[3)]				
		Temperature					Temperature				
		4°C	25°C	40°C	55°C	70°C	4°C	25°C	40°C	55°C	70°C
Control[1)]		3.77[d]	3.98[c]	4.32[c]	4.78[c]	5.01[c]	4.10[d]	4.42[c]	4.87[d]	5.50[d]	5.84[d]
Ungelatinization	Small	2.08[e]	1.98[d]	1.82[d]	1.81[d]	3.95[d]	2.28[e]	2.23[d]	2.11[e]	2.16[e]	5.24[d]
	Medium	2.11[e]	2.08[d]	1.97[de]	1.87[d]	4.91[c]	2.30[e]	2.32[d]	2.26[e]	2.21[e]	6.14[d]
	Large	2.23[e]	2.37[d]	2.17[e]	2.00[d]	4.27[d]	2.38[e]	2.59[d]	2.43[e]	2.29[e]	5.29[d]
Pregelatinization	Small	9.49[c]	8.07[b]	8.32[b]	7.59[b]	6.17[b]	11.48[c]	11.74[b]	12.67[c]	11.83[c]	10.56[c]
	Medium	10.53[b]	10.59[a]	10.38[a]	10.24[a]	7.71[a]	11.96[b]	13.63[a]	14.53[a]	14.47[a]	12.61[a]
	Large	12.22[a]	10.65[a]	10.43[a]	10.29[a]	7.88[a]	13.54[a]	13.22[a]	14.02[b]	13.83[b]	11.54[b]

[1)]Commercial Job's tears powder.
[2)]Water absorption index.
[3)]Swelling power.
[a-e]Different letters in a column indicate significant difference at $p<0.05$ by Duncan's multiple range test.

Table 3. Dispersibility and sinkability of pulverized Job's tears powders with particle size and different temperatures

Treatment	Sample	Dispersibility (%)			Sinkability (%)
		4°C	25°C	70°C	
Control[1)]		95.44[a]	96.13[a]	93.31[a]	28.10[c]
Ungelatinization	Small	78.94[d]	80.38[d]	82.28[d]	54.85[b]
	Medium	79.62[d]	81.42[d]	83.27[cd]	57.29[ab]
	Large	80.07[d]	81.26[d]	84.37[c]	60.95[a]
Pregelatinization	Small	83.60[c]	85.06[c]	89.74[b]	11.33[e]
	Medium	87.29[b]	87.40[b]	93.48[a]	14.99[de]
	Large	86.53[b]	87.84[b]	94.00[a]	16.58[d]

[1)]Commercial Job's tears.
[a-e]Different letters in a column indicate significant difference at $p<0.05$ by Duncan's multiple range test.

different particle size and gelatinization treatments are shown in Table 3. For dispersibility of commercial Job's tears powder, the control had higher than ungelatinized and pregelatinized samples at 4°C and 25°C. The sample of the smallest particle size is shown to have the lowest dispersibility, regardless of gelatinization treatment. In general, it is known that fine powders have low water wetability because of their high surface tension (21). In ungelatinized samples, the dispersibilities of the three different particle size samples of Job's tears were not significantly different ($p>0.05$) at 4°C and 25°C. But they were significantly different ($p<0.05$) at 70°C. However, pregelatinized samples showed different dispersibilities with different particle sizes at all the temperatures in the significant level ($p<0.05$). And the dispersibility of the pregelatinized samples increased as temperature increased. For pregelatinized samples of medium and large particle sizes, dispersibilities were 93.5 and 94.0% at 70°C, respectively and this dispersibility was significantly similar to control (93.3%). In accordance, these results suggest that decreasing particle size leads to decreased dispersion of Job's tears powder, but gelatinization treatment could improve the dispersibility of Job's tears powder with a small particle size.

Sinkability

The sinkability factor is affected by particle size and gelatinized treatment of Job's tears powders (Table 3). Sinkability decreased as the particle size decreased. The sample with the large particle size is expected to have high sinkability. For example, the velocity of sedimentation (w) is described by the Stokes' law equation $w = \triangle\rho g x^2/18\eta$, where $\triangle\rho$ is the difference in density between solid and liquid, g is acceleration due to gravity, x is the particle diameter, and η is the viscosity of the liquid. Thus, the sediment of the sample with large particle size is more rapid (21). The gelatinization treatment greatly affected sinkability, such that the sinkability of pregelatinized sample (11.33～16.58%) was lower than that of ungelatinized sample (54.85～60.95%) and control sample (28.10%). On the other hand, the control sample had higher sinkability than that of pregelatinized sample but sinkability of control sample had lower value than that of ungelatinized sample. These data suggest that pregelatinization and particle size difference would improve sinkability of Job's tears powder.

Turbidity

The particle size of Job's tears powder affected the turbidity of Job's tears powder suspensions (Fig. 4) significantly. The turbidity of Job's tears powder suspension increased as the particle size decreased. The gelatinization treatment also significantly affected the turbidity of Job's tears powder suspensions ($p<0.05$). Turbidity of control sample suspension was higher 7.16～17.5%

Pregelatinization

Fig. 4. Effect of particle size and gelatinization treatment on turbidity of Job' tears powder suspensions (2%, w/v).

than that of ungelatinized Job's tears powder samples. But it was lower than that of pregelatinized Job's tears powder. Turbidity of the sample with the pregelatinized small particle size for Job's tear powder was highest (1.08) among samples.

These results showed that small particle size sample had a high turbidity. Generally, the turbidity increased as the solubility increased. In accordance, increasing solubility induced high turbidity and this relationship between turbidity and WSI is shown Fig. 3.

Our results showed the effects of particle size and gelatinization of Job's tears on instant properties. Particle size (small, medium, and large) had an influence on the instant properties of Job's tears powder, and pregelatinization improved the instant properties. Water solubility index was shown to be the highest in the smallest particle size of pregelatinized Job's tears powder sample. Also, water solubility index increased as temperature increased from 4°C to 70°C. Sinkability of small particle size sample was lower than that of other large particle samples. Gelatinization treatment improved instant properties like dispersibility of Job's tears powder. Therefore, by using pregelatinization for the smallest particle of Job's tears powder, it should be possible to improve such physical properties as water solubility, disperbility and sinkability for food quality of Job's tears powder beverage.

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