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Effect of Particle Size on the Solubility and Dispersibility of Endosperm, Bran, and Husk Powders of Rice

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Abstract Size effects of rice product powders on physical properties including suspension stability were investigated in this study. Endosperm, bran, and husk powders of rice with different size particles were prepared using the pin crusher or the ultrafine air mill. The physical properties of the powders were examined using particle size analysis, scanning electron microscopy, and spectrophotometry. The peak of the volume-weighted particle distribution of ultrafine endosperm particles was at 5.4 μm whereas those of the bran and the husk appeared at 65 and 35 μm, respectively. Ultrafine particles of the endosperm and the husks dispersed better than larger-sized particles. As time elapsed, the dispersibility decreased, but the ultrafine particles were precipitated at the slowest rate. Our results suggest that ultrafine particles, including future nanosized particles, provide improved solubility and dispersibility resulting in better stability in the food colloidal suspension.

Keywords: rice, ultrafine milling, microparticle, solubility, dispersibility

Introduction

Micronizing or nanosizing is an emerging technique used to improve physical and biological properties including solubility and stability (1). Sub-micron sized particles offer a number of distinct advantages over microsized particles. Ultrafine particles have in general relatively higher intracellular uptake compared to large and coarse particles (2,3). Due to their sub-micron size, ultrafine particles are generally taken up efficiently by cells (4). Also, by modulating particle characteristics, one can control the release of a core material from the particles to achieve the desired level in the target sites for the required duration for optimal efficacy. In smaller particles, the encapsulated materials within the particle can diffuse easily to the surface (5).

Some of the challenges in creating, maintaining, and characterizing stable suspensions have been met with certain assumptions of ideality, e.g., spherical particles, mono-dispersed size distributions, and homogeneity in particle properties. A stable suspension can be prepared from a colloidal system where the continuous medium is an aqueous solution and the dispersed matter is mainly insoluble small particles. However, it is difficult to achieve a stable suspension since real particle suspension is poly-dispersed due to the interactions among the particles. The particles in the colloidal suspension tend to adhere together and form aggregates resulting in increasing particle size which may settle due to gravity. For many food applications, none or only a very small number of the particles should precipitate after prolonged storage.

According to Stoke's law, a smaller particle precipitates slower. Small sized particles are preferred to make the suspension stable. Thus, the ability to reproducibly disperse particles in suspension and to maintain a stable suspension is important in food applications.

Rice, the most widely grown food grain crop, serves as the staple food for about half of the population of the world. Only the endosperm of rice is consumed while its milling co-products, bran and husks, are underutilized. Thus, rice bran and husks are inexpensive. Rice bran has a high nutritional value with 6-14% total dietary fiber, 12-23% lipid, 12-15% protein including a higher content of lysine than rice endosperm protein or any other cereal bran proteins (6). The major constituents of rice husks are 38% alpha cellulose, 22% lignin, 20% ash, and 19% silica (7). Since rice bran and husks are high in health-related nutrients, they have the potential to be used as food ingredients. However, micro-sized powders of these healthful ingredients should be limited because they are inferior in food qualities such as texture and taste. For example, the coarse cereal ingredient offered a grainy flavor and a gritty texture in all fiber-fortified yogurts (8). Hopefully, it is considered that ultrafine cereal ingredients may improve functional properties such as solubility and stability and thereby expand to myriad the possibilities for the use of rice products.

The objectives of this study were to prepare ultrafine endosperm, bran, and husk powders of rice with different sizes and to investigate their size effect on physical properties such as solubility and dispersibility.

Materials and Methods

Materials Endosperm, bran, and husks of rice were obtained from a local grain processing plant (Gangwon Nonghyup, Korea). The samples were cleaned by eliminating impurities, and were subsequently put into plastic bags and placed in a refrigerator until milling.

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Universal milling A pin crusher (Myungsung Machine, Seoul, Korea) with a 30-mesh sieve was used to mill the endosperm, bran, and husks of rice. It was operated at 1,000 rpm to obtain desirable particle size. Standard sieves (Φ 20 cm, Chunggye Industrial Mfg., Co., Seoul, Korea) installed on a sieve shaker (CG-213; Ro-Top, Chunggye Industrial Mfg.) were used to classify the size of the powders. The powders of the endosperm, bran, and husks of rice were classified; some particles passed through the sieve with 106 μm opening width and some particles passed through the sieve with 300 μm opening width. The rejected powders after the classification were milled again using the pin crusher. The classified powders were put into plastic bags and stored at 4°C in a desiccator until further analysis and subsequent ultrafine milling.

Ultrafine milling An ultrafine air mill (Turbo Mill, HKP-05; Korea Energy Technology, Seoul, Korea) was applied to produce ultrafine powders by grinding the endosperm, bran, and husks. The ultrafine air mill grinds the material, classifying its size at the same time in the mill. In brief, the mill consists of inlet, grinding zone, and outlet in the cooling jacket, which minimizes the increase in the material's temperature during milling. The mill includes a centrifugal air-classification system which sorts the powders depending on the size of their particles. If the size of a particle is larger than the criterion, the particle (coarse) is sent to the grinding zone of the mill. Otherwise, the particle (ultrafine) is sent to the outlet. The endosperm, bran, and husk powders produced by the universal milling were supplied into the ultrafine air mill to produce ultrafine powders. The ultrafine air mill was operated at 3 kg/hr of the feeding rate of the raw material and 100 m/sec of the circumferential velocity of the impeller in the grinding zone.

Particle size analysis After ultrafine milling, the average size and distribution of the endosperm, bran, and husk particles were determined using a commercial particle size analyzer (Mastersizer-2000; Malvern Ins., Ltd., Worcestershire, UK). For size measurement, the particles prepared were dispersed in 10 mL of distilled water at a 1:400 (w/v) ratio. The suspensions were stirred continuously at room temperature for 30 min and then measured at 25°C with a scattering angle of 90°. Average particle size was the average of 9 measurements for every sample.

Scanning electron microscopy (SEM) SEM was used to investigate the shape of the particles prepared. The samples were examined in a low vacuum scanning electron microscope (S-3500N; Hitachi Science Systems, Ltd., Ibaraki, Japan) whose resolution is 3.0 nm at high voltage but 4.0 nm at low voltage. The SEM was operated at an accelerating voltage of 10 kV. The SEM can examine the surface of the sample without fixation and/or dehydration steps which usually provide artifacts and deformation in the sample. The samples were photographed monochromically at 200× magnification. All imaging procedures were performed at the Korea Basic Science Institute (KBSI, Kangwon National University, Chuncheon, Korea).

Dispersibility measurement In order to prepare the sample for the dispersibility measurement, 0.1 g of the sample powder was dissolved in 50 mL of pH 7.4 phosphate buffered saline (D-PBS D5652; Sigma-Aldrich, Steinheim, Germany) solution. The mixture was stirred at 500 rpm for 10 min. After stirring, 1 mL of the mixture was collected into a cuvette and subsequently its absorbance was measured at 650 nm wavelength in a spectrophotometer (DU 730; Beckman Coulter, Fullerton, CA, USA). The turbidity profile of the sample was recorded for 20 min. The measurements were repeated 3 times for each sample.

Solubility measurement For the solubility measurement, 2.5 g of sample powder (dry sample weight) was added to 30 mL of distilled water by stirring at 700 rpm and placed at 25°C for 30 min. Each sample was put into a tube and then centrifuged at 7,232×g for 30 min. The supernatant was poured into an aluminum dish and dried in an oven at 105°C for 5 hr. The sediment in the tube (wet sample weight) and the dry supernatant in the aluminum dish (dry supernatant weight) were weighed. Three solubility indices were calculated using the equations as follows (9):

$$\text{Water absorption index (WAI)} = \frac{\text{wet sediment weight}}{\text{dry sample weight}}$$

$$\text{Water solubility (WS,\%)} = \frac{\text{dry supernatant weight}}{\text{dry sample weight}} \times 100$$

$$\text{Swelling power (SP)} = \frac{\text{wet sediment weight}}{\text{dry sample weight} \times \left(1 - \frac{\text{WS(\%)}}{100}\right)}$$

Results and Discussion

Ultrafine milling Endosperm, bran, and husk powders of rice with different size were prepared using the pin crusher or the ultrafine air mill. The powders of the endosperm, bran, and husks of rice were classified: ultrafine endosperm powder (E_ultra), endosperm powder which passed the sieve with 106 μm opening width (E_106), endosperm powder which passed the sieve with 300 μm opening width (E_300), ultrafine bran powder (B_ultra), bran powder which passed the sieve with 106 μm opening width (B_106), bran powder which passed the sieve with 300 μm opening width (B_300), ultrafine husk powder (H_ultra), husk powder which passed the sieve with 106 μm opening width (H_106), and husk powder which passed the sieve with 300 μm opening width (H_300).

Figure 1 shows the distribution of the endosperm, bran, and husk particles produced using the ultrafine milling. For the endosperm particles, the peak of the volume-weighted particle distribution was at 5.4 μm whereas the volume-weighted mean, i.e., D[4,3] was 9.4 μm. The peaks of the bran and the husk particles appeared at 65 and 35 μm, respectively, whereas their volume-weighted mean values were 63 and 62 μm. Even under similar milling conditions, the average particle size of the bran and the husk powders was larger than that of the endosperm powder during the light-scattered particle analysis. This phenomenon might be caused by hydrophobic interactions in the aqueous condition. Bran has a higher content of lipid and protein than endosperm. Husk also has a lot of insoluble molecules. As shown in Fig. 1a and 1c, there is a clear shoulder peak

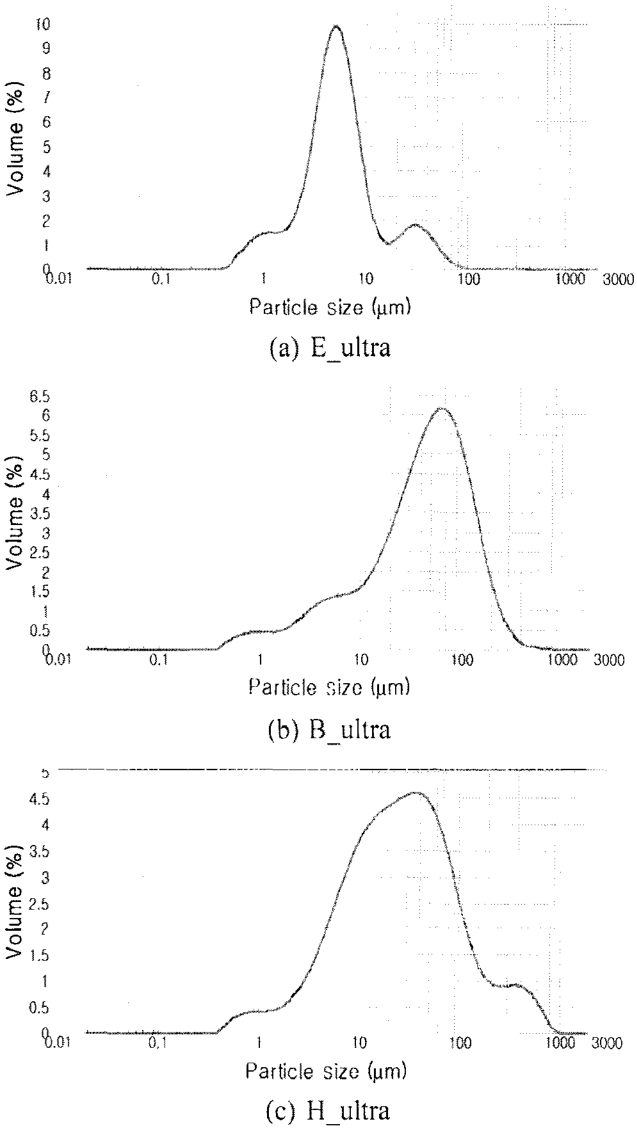

Fig. 1. Particle size analysis of ultrafine (a) endosperm, (b) bran, and (c) husk powders of rice.

after the major peak. The shoulder peak after the major peak may appear due to the aggregations among the particles. Hydrophobic interactions between hydrophobic regions on the surface of the particles lead to their aggregation, resulting in size increase (10). Thus, small hydrophobic interactions on the endosperm particles suppressed the aggregation of the molecules and subsequently resulted in smaller size during the light-scattered particle analysis.

Figure 2 shows the scanning electron micrographs of endosperm, bran, and husk particles of rice at 200 times magnification. The ultrafine endosperm particles are shown as relatively similar as a sphere shape whereas the husk particles are irregular in shape. A shear force provides the dominant effect on soft materials such as fibers resulting in the angulated corners of the particle shape. Husk usually is much softer than endosperm. In addition, endosperm, bran, and husk particles are angulated as their size increases. It is a typical feature of universal milling by the pin crusher.

Table 1. Effect of mean particle size on the water absorption index (WAI) of endosperm, bran, husk powders of rice[1)]

Rice	Ultrafine	106	300	p-value
Endosperm	2.33$^{a2)}$	2.05^{ab}	1.98^{b}	0.015
Bran	1.99^{a}	1.75^{a}	2.10^{a}	0.284
Husk	2.57^{b}	3.43^{ab}	3.73^{a}	0.012

[1)]Ultrafine denotes the powder ground using an ultrafine air mill; 106 denotes the powder passed a sieve with 106 μm opening width; 300 denotes the powder passed a sieve with 300 μm opening width.
[2)]Means with the same letter in the same row are not significantly different at the 5% level.

Table 2. Effect of mean particle size on the water solubility (WS) of endosperm, bran, and husk powders of rice[1)]

Rice	Ultrafine	106	300	p-value
Endosperm	4.47$^{a2)}$	3.13^{ab}	1.65^{b}	0.005
Bran	26.40^{a}	29.75^{a}	30.00^{a}	0.344
Husk	10.78^{a}	8.25^{a}	4.15^{b}	0.016

[1)]Ultrafine denotes the powder ground using an ultrafine air mill; 106 denotes the powder passed a sieve with 106 μm opening width; 300 denotes the powder passed a sieve with 300 μm opening width.
[2)]Means with the same letter in the same row are not significantly different at the 5% level.

Table 3. Effect of mean particle size on the swelling power (SP) of endosperm, bran, and husk powders of rice[1)]

Rice	Ultrafine	106	300	p-value
Endosperm	2.44$^{a2)}$	2.25^{ab}	2.01^{b}	0.013
Bran	2.71^{a}	2.50^{a}	3.01^{a}	0.345
Husk	2.84^{b}	3.79^{a}	3.87^{a}	0.005

[1)]Ultrafine denotes the powder ground using an ultrafine air mill; 106 denotes the powder passed a sieve with 106 μm opening width; 300 denotes the powder passed a sieve with 300 μm opening width.
[2)]Means with the same letter in the same row are not significantly different at the 5% level.

Effect of particle size on solubility ***Rice endosperm powder*:** Table 1 shows the effect of particle size on the water absorption index (WAI) of rice endosperm powder. The WAI value of E_ultra was the largest. The smaller the particle size, the larger the water absorption was observed. Table 2 shows the effect of particle size on the water solubility (WS) of rice endosperm powder. The WS values of the endosperm powder were the smaller than those of bran and husk. It can be elucidated that water hardly penetrates and is absorbed into the crystalline structure of the starch in the endosperm at room temperature (11,12). The tendency of WS increased with the decreasing particle size of the rice endosperm. The WS values of E_ultra, E_106, and E_300 were 4.47, 3.13, and 1.65%, respectively. Thus, small-sized endosperm particles are more soluble as the size of the particles is in the micrometer range. Thus, the solubility of endosperm powder can be improved by making the particle size smaller. The effect of particle size on the swelling power (SP) of rice endosperm powder is shown in Table 3. In water, endosperm powder swells due to the absorption of water. The SP values increased with decreasing particle size. The SP values of E_ultra, E_106,

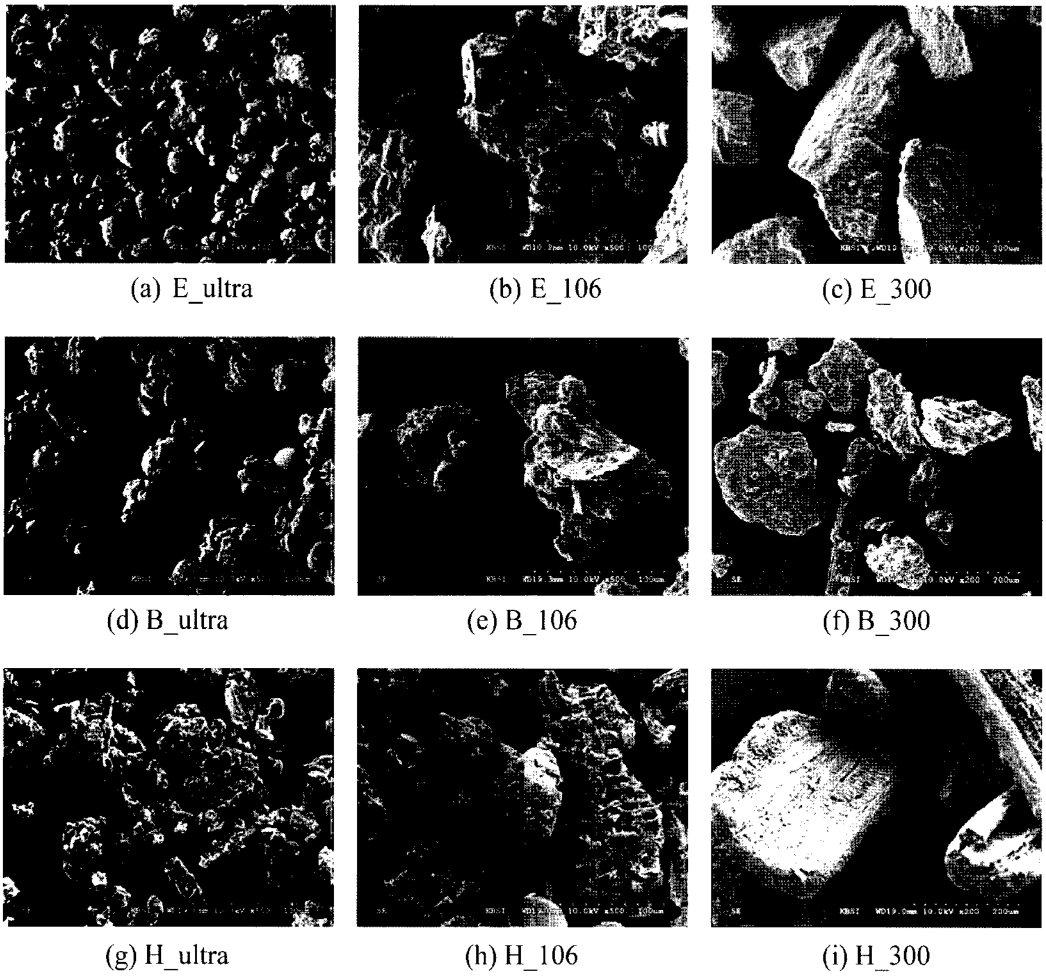

Fig. 2. Scanning electron micrographs of endosperm, bran, and husk particles of rice at 200× magnification. (a) Ultrafine endosperm powder, (b) endosperm powder passed the sieve with 106 μm opening width, (c) endosperm powder passed the sieve with 300 μm opening width, (d) ultrafine bran powder, (e) bran powder passed the sieve with 106 μm opening width, (f) bran powder passed the sieve with 300 μm opening width, (g) ultrafine husk powder, (h) husk powder passed the sieve with 106 μm opening width, (i) husk powder passed the sieve with 300 μm opening width.

and E_300 were 2.44, 2.25, and 2.01, respectively. The smaller the particle size, the larger the SP was observed. This result agrees with several previous studies which showed larger starch particles (or granules) have smaller SP (13,14).

***Rice bran powder*:** Table 1 shows the effect of particle size on the WAI of bran powder of rice. The WAI values were independent of the particle size. Table 2 shows the effect of particle size on the WS of bran powder of rice. The WS values of the bran powder were exceptionally larger than those of the endosperm and husk powders. The WS of bran powder is due to the existence of soluble fiber in it. Rice bran contains soluble fiber 1.8-2.7% (6) whereas both endosperm and husk powder have less soluble components. However, the WS values were independent of the particle size. The innate properties of rice bran result in the disappearance of the size effect. Rice bran is composed of 12-15% protein and 12-23% lipid content (6). The protein-protein and protein-lipid interactions, including aggregation and/or disulfide bond, diminish the effect of particle size on solubility (15). Generally, protein-protein and protein-lipid interactions induce self-agglomeration in the aqueous environment. The effect of particle size on the SP of bran powder of rice is summarized in Table 3. The SP values were independent of the particle size. No effect of the particle size on the WS values also elucidates the reason that the SP values of rice bran powder are similar even at different particle sizes.

***Rice husk powder*:** Table 1 shows the effect of particle size on the WAI of husk powder of rice. The WAI values decreased with the decreasing particle size of the rice husk. The WAI values of the husk powder were small compared to those of bran. The small absorption of water in the husk powder is due to the high content of insoluble fibers such as cellulose and lignin, which seldom retain water. The major constituents of rice husk are 38% alpha cellulose, 22% lignin, 20% ash, and 19% silica (7). Table 2 and 3 show the effects of particle size on the WS and SP of the husk powder of rice, respectively. The smaller the particle size, the larger the WS value was observed. The SP values decreased with decreasing particle size. Both the WS and the SP of the rice powder showed patterns similar to those obtained for the endosperm powder. Briefly, smaller husk particles are more soluble and swell larger.

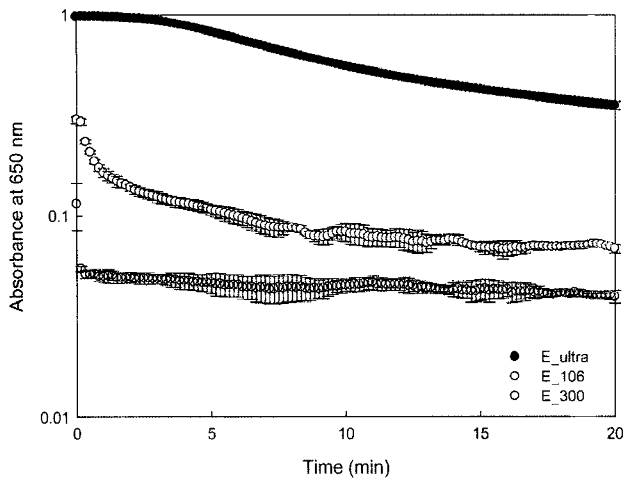

Fig. 3. The effect of particle size on dispersibility of rice endosperm powders. Ultra, 106, and 300 denote the powder ground using an ultrafine air mill, the powder passed a sieve with 106 μm opening width, and the powder passed a sieve with 300 μm opening width, respectively.

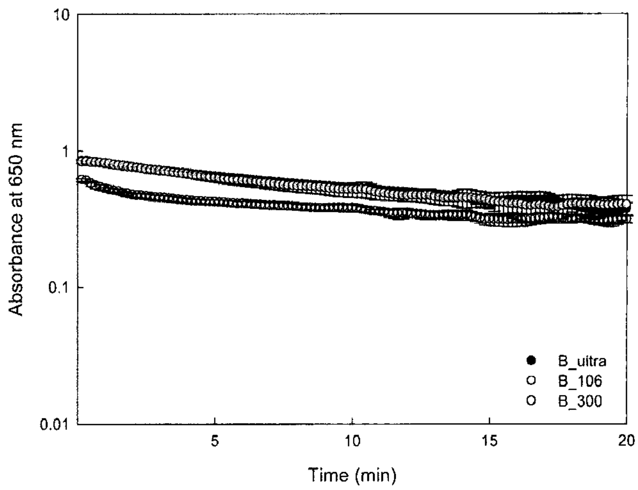

Fig. 4. The effect of particle size on dispersibility of rice bran powders. Ultra, 106, and 300 denote the powder ground using an ultrafine air mill, the powder passed a sieve with 106 μm opening width, and the powder passed a sieve with 300 μm opening width, respectively.

Effect of particle size on dispersibility ***Rice endosperm powder*:** Figure 3 shows the effect of particle size on the dispersibility of rice endosperm powder in a function of time. The initial absorbance of E_ultra was larger than those of E_106 and E_300. Larger absorbance value means that the particles in a solution are dispersed better. Thus, E_ultra was dispersed better than E_106 and E_300 at the initial stage. In the case of E_300, the particles were precipitated rapidly, indicating that the initial absorbance from the spectrophotometer was less than 0.1. Thus, small-sized endosperm particles have better dispersibility at the initial stage after mixing.

As time elapsed, all the absorbance values of the endosperm particles with different size decreased. The particles of E_ultra were precipitated at the slowest rate since the size of the particles was the smallest. The particles of E_106 were precipitated faster than those of E_ultra. The E_300 particles were precipitated at the fastest rate after loading the sample in the spectrophotometer. It is concluded that small-sized endosperm particles have better dispersibility, as their size is in the micrometer range. These results agree with the previous reports that the particles are governed by Stoke's law (16). Thus, ultrafine endosperm powder has the potential to provide a stable suspension in an aqueous system, minimizing the precipitation.

***Rice bran powder*:** Figure 4 shows the effect of particle size on the dispersibility of bran powder of rice in a function of time. The initial absorbance values of B_ultra were similar to those of B_106 and B_300. It means that different-sized bran particles in aqueous solution are dispersed similarly at the initial stage. The initial absorbance values of the bran powder were larger than those of the endosperm and husk. The composition of bran, such as soluble fiber and protein, provides better dispersibility compared to other elements in rice crops. All absorbance values of the different-sized bran powders decreased in a function of time, however, the bran particles, regardless of their size, were precipitated slowly. The aqueous solution of the bran powder can be considered as a stable system since the final absorbance values at 20 min elapsed are still large as shown in Fig. 4. The protein-water and soluble fiber-water interactions make the bran aqueous solution stable (15).

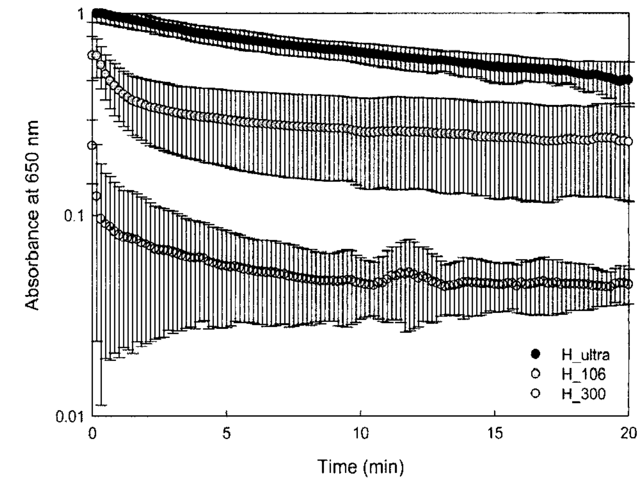

Fig. 5. The effect of particle size on dispersibility of rice husk powder. Ultra, 106, and 300 denote the powder ground using an ultrafine air mill, the powder passed a sieve with 106 μm opening width, and the powder passed a sieve with 300 μm opening width, respectively.

***Rice husk powder*:** The effect of the husk powders with different size on the stability of a solution is clear as shown in Fig. 5. The absorbance of H_ultra was larger than those of H_106 and H_300, indicating that the H_ultra particles are dispersed better in an aqueous solution. On the contrary, the H_300 particles were precipitated quickly, as soon as the sample was injected, during the absorbance measurement. The smaller the particle size, the slower the precipitation. Small-sized husk particles have better dispersibility, similar to the endosperm particles. Thus, ultrafine husk powder forms a stable aqueous suspension which is proper for the application of food beverage, including insoluble fibers.

Our results suggest that ultrafine particles, including nanosized particles, provide improved solubility and dispersibility, resulting in better stability in the suspension. The effective use of cereal- or fiber-based powder is possible by incorporating them into several prepared foods such as high energy drinks, extruded snacks, dairy products, bread, cookies, baked foods, yogurt, and cheese. Ultrafine ingredients expand their food applications, avoiding off-limits from coarse cereal ingredient such as a grainy flavor and a gritty texture. The coming of food nanotechnology is opening the way for the development of novel healthy foods.

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