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Effect of Fluidized Bed Agglomeration on Rheological and Physical Properties of Soy Protein-Enriched Milk Powder for Dietary Supplementation in Sarcopenia

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ABSTRACT: Recently, many elderly people in Korea have been consuming protein-enriched milk powders for dietary supplementation in sarcopenia. In general, protein powders are manufactured using a spray dryer, and their fine particles result in poor instant properties. Fluidized bed agglomeration (FBA) is an effective method for improving the physical properties of protein powders via particle granulation, such as flowability and wettability. Therefore, the effects of FBA on the rheological and physical properties of isolated soy protein (ISP)-enriched skim milk powder (SMP) were investigated in this study. The size of ISP-enriched SMP particles was significantly increased by FBA, leading to changes in the Carr index and Hausner ratio from fair flowability and intermediate cohesiveness to good flowability and low cohesiveness, respectively. The wettability of the granulated particles was also improved by FBA, and they exhibited a shorter wetting time below 10 s. However, a slight color change was observed after the FBA process. These findings contribute to the production of protein-enriched food powders with improved properties.

Keywords: fluidized bed agglomeration, milk powder, sarcopenia, soy protein, wettability

INTRODUCTION

Sarcopenia (sarx: muscle+penia: reduction) is defined as a decrease in muscle mass and strength, and is mainly caused by aging and malnutrition. The number of people over 65 years of age is predicted to increase globally to approximately 1.5 billion in 2050, and a drastic increase in elderly people may be accompanied by an increase in sarcopenia (Liao et al., 2019; Park et al., 2021). Sufficient protein intake is necessary to prevent muscle loss in elderly people, and high-protein supplements are a growing market, especially due to the rapid increase in the aging population (Agarwal et al., 2015; Kobayashi et al., 2016). Currently, protein-enriched food powders are consumed in Korea due to this change in the elderly population, and are typically produced by mixing milk powders, spray-dried proteins, and small amounts of vitamins and minerals for functionality (Seo, 2022b).

Soy protein is widely used in protein-enriched foods because of its nutritional value and functionality, and isolated soy protein (ISP) is the most widely used commercial product with high soy protein (Machado et al., 2014). Although ISP has a low methionine content, it contains all essential amino acids and can provide high-quality proteins required for humans (Xiao, 2008; Nishinari et al., 2014). According to protein digestibility-corrected amino acid scores, the composition of soy protein is similar to that of animal-based proteins of high quality (e.g., milk and eggs). Soy protein is also highly digestible (92∼96%) and can effectively promote muscle growth and strength. Owing to these advantages, they can be effectively used as an alternative to animal proteins in protein-enriched foods (Singh et al., 2008).

ISP is typically produced by spray drying, and spray-dried protein powders exhibit extremely poor flowability and wettability due to their fine particle size, which results in poor hydration properties (Dacanal and Menegalli, 2010; Atalar and Yazici, 2019). Fluidized bed agglomeration (FBA) is the transformation of small fine particles to large porous particles with improved physical properties such as flowability, blending capacity, and rehydration, and is commonly used in the food industry to overcome these problems (Park and Yoo, 2020; Atalar and Yazici, 2021). Although several studies have investigated the effects of FBA on ISP (Dacanal et al., 2013; Machado et al., 2014) and dairy-based powders (Turchiuli et al.,

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2013; Ji et al., 2017; Wu et al., 2020), no study has focused on the influence of the FBA in mixed systems of soy protein and skim milk powder (SMP). Thus, in this study, we investigated the effect of FBA on the rheological and physical properties of ISP-enriched SMP and the impact of the ISP ratio on this process.

MATERIALS AND METHODS

Materials

ISP (Supro 661) containing 88.5% protein, 3.2% fat, 1% sugar, and 3.9% ash, and SMP (37% protein, 0.5% fat, 49% lactose, and 7.9% ash) was obtained from Solae Company (St. Louis, MO, USA) and Seoul Dairy Cooperative (Ansan, Korea), respectively. The ISP-enriched SMP samples were manufactured by mixing at ratios of 0:10 (SMP), 1:9 (ISP1/SMP9), 2:8 (ISP2/SMP8), and 3:7 (ISP3/SMP7) using a tumbler mixer (Inversina 2 L, Bioengineering AG, Wald, Switzerland).

FBA process

FBA was performed using a laboratory-scale FBA granulator (Fluid Bed Lap System, Einsystem Co., Ltd., Cheonan, Korea) according to the method described by Seo (2022b). The injected air temperature was set to 95±1°C, and the product temperature in the vessel was maintained at 55±1°C. Distilled water (binder solution) was pumped using a peristaltic pump (10 mL/min) and sprayed for 40 min at a pressure of 1.5 bar. Subsequently, the granulated ISP-enriched SMP particles were dried for 10 min without spraying the binder solution.

Measurement of particle size distribution

The particle size distributions were analyzed using a Mastersizer 3000 (Malvern Instruments Ltd., Worcestershire, UK) according to the method described by Seo (2022a). The surface-weighted mean diameter (D[3,2]) and span (dispersion index) were measured to evaluate the particle size and distribution of the ISP-enriched SMP particles. These values were determined using the following equations:

$$\mathrm{D}[3,2] = \frac{\sum n_i d_i^3}{\sum n_i d_i^2} \qquad (1)$$

$$\mathrm{Span} = \frac{\mathrm{Dv90} - \mathrm{Dv10}}{\mathrm{Dv50}} \qquad (2)$$

where d_i is the diameter of the paricles in i class and n_i is the number of particles with the same d_i. Dv10, Dv50, and Dv90 are the average diameters corresponding to the cumulative volumes of 10%, 50%, and 90% particles, respectively.

Bulk and tapped density measurements

The bulk density (ρ_{bulk}) and tapped density (ρ_{tapped}) were determined based on the mass/volume ratios for the ISP-enriched SMP particles before and after 1,250 taps, respectively, using a BT-301 powder density tester (Bettersize Instrument Ltd., Dandong, China).

Measurement of flowability and cohesiveness

The Carr index (CI) and Hausner ratio (HR) were calculated to evaluate the flowability and cohesiveness of the ISP-enriched SMP particles using equations (3) and (4), respectively:

$$\mathrm{CI} = \frac{\rho_{tapped} - \rho_{bulk}}{\rho_{tapped}} \times 100 \qquad (3)$$

$$\mathrm{HR} = \frac{\rho_{tapped}}{\rho_{bulk}} \times 100 \qquad (4)$$

The levels of flowability (%) and cohesiveness based on CI and HR values are summarized in Table 1.

Measurement of wetting time

Wetting time (t_w), defined as the time required to fully wet and immerse in water, was determined using the method described by Seo (2022a). The ISP-enriched SMP samples (5 g) were added to distilled water (100 mL) at 24±1°C.

Analysis of color

L* (lightness), a* (redness), and b* (yellowness) were measured according to the method described by Seo (2022b) using a Hunter Lab colorimeter (Hunter Associates Laboratory Inc., Reston, VA, USA) to evaluate the color properties of ISP-enriched SMP particles.

Rheological measurement of rehydrated beverages

The agglomerated powder sample (50 g) was rehydrated in 200 mL of drinking water to measure the flow behavior of solutions with ISP-enriched SMP particles. After 5 min equilibration at 25°C, the flow curve was recorded

Table 1. Classification of powder flowability and cohesiveness based on CI and HR

	Classification	
CI (flowability)	<15	Very good
	15~20	Good
	20~35	Fair
	35~45	Bad
	>45	Very bad
HR (cohesiveness)	<1.2	Low
	1.2~1.4	Intermediate
	>1.4	High

CI, Carr index; HR, Hausner ratio.

at a shear rate from 0.4 to 100 s^{-1} using HAKKE Roto Visco-1 (Thermo Fisher Scientific, Waltham, MA, USA) with a cone-plate (35 mm diameter) geometry.

Statistical analysis

All experiments were conducted in triplicate, and the data are expressed as the mean±standard deviation. One-way analysis of variance and Tukey's test at $P<0.05$ were performed for statistical analysis of data using the IBM SPSS Statistics 24 software (IBM Software, Armonk, NY, USA).

RESULTS

Particle size distribution

The particle size profiles of the raw and granulated ISP-enriched SMP particles are shown in Fig. 1, and a monomodal size distribution was observed for all samples. The peaks of the raw ISP-enriched SMP particles migrated from the small-particle-size region to the large-particle-size region as the FBA proceeded, indicating that the particles of the raw powder samples were effectively granulated by FBA.

The particle size values also proved the granulation of the ISP-enriched SMP particles by the FBA process (Table 2). As the FBA proceeded, the D[3,2] of the ISP-enriched SMP particles increased, along with the other particle size values (Dv10, Dv50, and Dv90). In the case of span values, the granulated SMP and ISP1/SMP9 particles had higher spans than the raw powder particles. However, the spans of the ISP2/SMP8 and ISP3/SMP7 particles decreased after the FBA process. These results indicate that the FBA process can lead to a homogenous particle size distribution of ISP/SMP particles with a high ISP ratio.

Bulk and tapped density

The physical properties of ISP-enriched SMP particles are listed in Table 3. As FBA proceeded, ρ_{bulk} of powder particles decreased from 0.44~0.47 g/cm^3 to 0.28~0.37 g/cm^3, and ρ_{tapped} also decreased from 0.58~0.63 g/cm^3 to 0.33~0.42 g/cm^3, respectively, and increased with an increase in ISP ratio.

Flowability and cohesiveness

The flow and cohesive properties of the powder particles were predicted using the CI and HR values. In the raw powder particles, the level of CI changed from good (SMP=18.1) to fair (ISP3/SMP7=29.0) with an increase in the ISP ratio. The HR of the raw SMP particles was intermediate (1.22) and increased with increasing ISP ratio. In particular, ISP3/SMP7 exhibited a high cohesiveness (1.41; Table 3). After FBA, both the CI and HR of the granulated ISP-enriched SMP particles decreased, indicating that the flowability and cohesiveness of the pow-

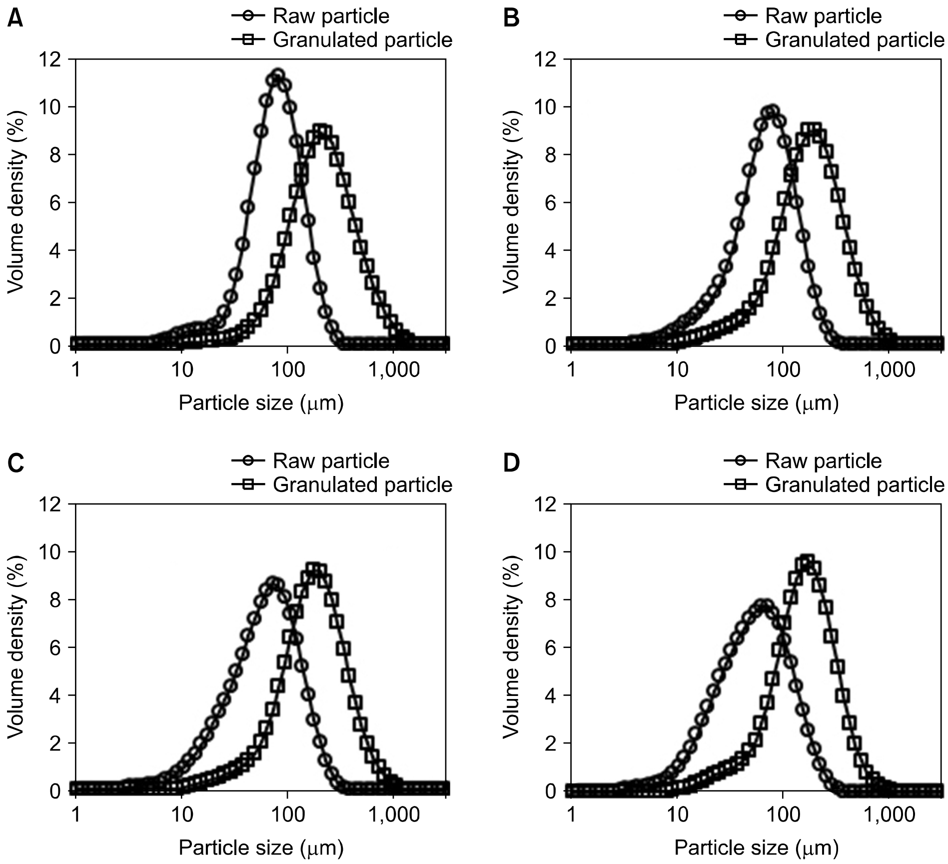

Fig. 1. Particle size distribution of raw and granulated isolated soy protein (ISP)-enriched skim milk powder (SMP) samples with different ISP/SMP ratios: (A) SMP (0:10), (B) ISP1/SMP9 (1:9), (C) ISP2/SMP8 (2:8), and (D) ISP3/SMP7 (3:7).

Table 2. Particle size and distribution of raw and granulated ISP-enriched SMP samples with different ISP/SMP ratios

Sample	D[3,2] (μm)	Dv10 (μm)	Dv50 (μm)	Dv90 (μm)	Span (–)
Raw powder					
ISP	22.0±0.0^{a}	13.0±0.0^{a}	28.3±0.1^{a}	53.2±0.2^{a}	1.42±0.01^{a}
SMP	58.2±0.4^{e}	35.4±0.2^{e}	76.4±0.7^{e}	148.7±2.1^{b}	1.48±0.02^{a}
ISP1/SMP9	47.5±0.1^{d}	24.8±0.1^{d}	69.8±0.2^{d}	147.3±0.6^{b}	1.76±0.01^{b}
ISP2/SMP8	40.4±0.2^{c}	20.1±0.1^{c}	62.1±0.3^{c}	140.3±0.6^{b}	1.94±0.01^{de}
ISP3/SMP7	36.1±0.1^{b}	18.0±0.1^{b}	54.6±0.1^{b}	134.3±0.6^{b}	2.13±0.01^{f}
Granulated powder					
SMP	147.3±3.2^{h}	79.9±2.0^{h}	198.0±5.3^{h}	471.7±22.8^{e}	1.98±0.05^{e}
ISP1/SMP9	123.7±1.5^{g}	67.1±0.6^{g}	177.7±2.5^{g}	419.7±13.6^{d}	1.98±0.05^{e}
ISP2/SMP8	121.7±1.2^{g}	66.6±0.4^{g}	172.7±2.5^{g}	392.7±13.5^{d}	1.89±0.05^{cd}
ISP3/SMP7	110.7±1.2^{f}	60.1±0.0^{f}	160.7±1.2^{f}	353.7±5.5^{c}	1.83±0.03^{bc}

Values are presented as mean of three measurements±SD.
Mean values in the same column with different letters (a-h) are significantly different (P<0.05).
ISP, isolated soy protein; SMP, skim milk powder.

der particles were significantly improved by FBA. In particular, ISP2/SMP8 and ISP3/SMP7 particles showed very good flowability (CI<15) and low cohesiveness (HR<1.2). These results suggest that FBA can improve the flowability and cohesiveness of ISP/SMP samples with a high ISP ratio to a greater extent than those with a low ISP ratio.

Wetting property

The wetting properties of the ISP-enriched SMP particles are presented in Fig. 2 and Table 3. The raw powder particles formed a lump on the water surface and were not immersed in water, resulting in a long t_w of over 120 s. In contrast, the t_w of granulated SMP and ISP-enriched SMP particles significantly decreased to 4.33~8.67 s, and the granulated powder particles showed a short t_w of less than 20 s, which is the instant criterion proposed by Chever et al. (2017).

Color properties

The color properties (L*, a*, and b*) of ISP-enriched SMP particles are summarized in Table 4. The L* value decreased with increasing ISP ratio, and the granulated ISP-enriched SMP particles showed lower L* values than their raw counterparts. This decrease in L* indicates darkening of color. Moreover, granulated ISP-enriched SMP particles have higher b* values compared to those of their raw counterparts. These observations suggest that FBA can make ISP-enriched SMP particles dark and yellow.

Flow behavior of rehydrated solutions

The flow curves of solutions with granulated ISP-enriched SMP particles are shown in Fig. 3. The viscosity of the rehydrated solution with SMP decreased as the shear rate increased. This tendency is shear-thinning behavior, and it decreases with an increase in the ISP ratio. The solution with the ISP3/SMP7 particles exhibited Newtonian behavior, indicating a constant viscosity in the range of 1~100 s^{-1}. Because of the different flow behaviors of the SMP and ISP-enriched SMP particles, the rehydrated solutions with SMP had a higher viscosity than those with ISP-enriched SMP at low shear rates. In contrast, at a high shear rate (100 s^{-1}), the rehydrated solutions with ISP-enriched SMP had a higher viscosity than those

Table 3. Physical and rehydration properties of raw and granulated ISP-enriched SMP samples with different ISP/SMP ratios

Sample	ρ_{bulk} (g/cm^3)	ρ_{tapped} (g/cm^3)	Carr index (%)	Hausner ratio	t_w (s)
Raw powder					
SMP	0.47±0.01^{f}	0.58±0.01^{e}	18.1±0.68^{b}	1.22±0.01^{b}	>120
ISP1/SMP9	0.47±0.01^{f}	0.60±0.01^{f}	22.6±0.62^{c}	1.29±0.01^{c}	>120
ISP2/SMP8	0.46±0.01^{f}	0.62±0.01^{fg}	24.9±0.96^{c}	1.33±0.02^{c}	>120
ISP3/SMP7	0.44±0.01^{e}	0.63±0.01^{g}	29.0±0.95^{d}	1.41±0.02^{d}	>120
Granulated powder					
SMP	0.29±0.01^{b}	0.35±0.01^{b}	17.0±1.42^{b}	1.21±0.02^{b}	4.33±0.58^{a}
ISP1/SMP9	0.28±0.01^{a}	0.33±0.01^{a}	16.6±1.08^{b}	1.20±0.02^{b}	4.67±1.15^{a}
ISP2/SMP8	0.34±0.01^{c}	0.39±0.01^{c}	13.3±0.82^{a}	1.15±0.01^{a}	5.33±1.53^{a}
ISP3/SMP7	0.37±0.01^{d}	0.42±0.01^{d}	12.0±0.22^{a}	1.14±0.01^{a}	8.67±2.52^{b}

Values are presented as mean of three measurements±SD.
Mean values in the same column with different letters (a-g) are significantly different (P<0.05).
ISP, isolated soy protein; SMP, skim milk powder; t_w, wetting time.

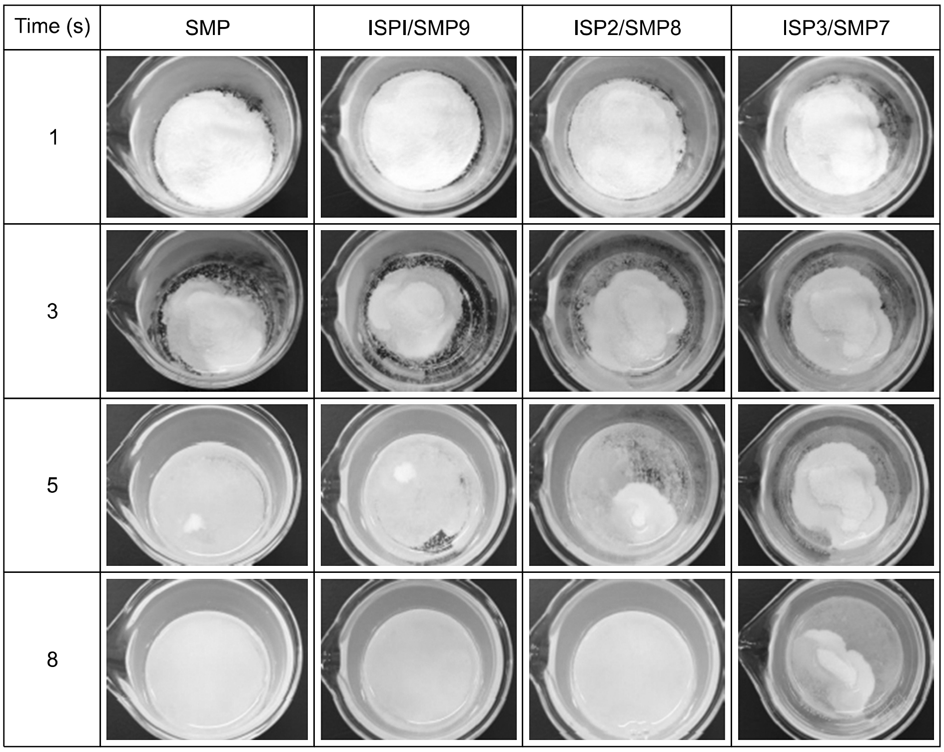

Fig. 2. Wettability of granulated skim milk powder (SMP) and isolated soy protein (ISP)-enriched SMP samples with different ISP/SMP ratios.

with SMP.

DISCUSSION

Particle size distribution in food powders is regarded as an important factor that can affect powder characteristics, including density (ρ_{bulk} and ρ_{tapped}), rheology (CI and HR), and dispersion properties (t_w) (Lee and Yoo, 2021). FBA induced significant granulation of the SMP and ISP-enriched SMP particles, as demonstrated by the increased particle size. FBA consists of three stages: moisture absorption, adhesion, and fixation. First, the binder solution is sprayed onto fine powder particles to make their surfaces sticky. The sticky particles then bind together, resulting in the agglomeration of the powder particles. Finally, the agglomerated particles are dried and fixed using hot air (Barkouti et al., 2013; Lim et al., 2021). The ISP-enriched SMP particles were effectively granulated by the FBA process, resulting in a large particle size. However, the granulation level of ISP-enriched SMP particles decreased slightly as the ISP ratio increased. According to Barkouti et al. (2013), particle granulation during FBA is strongly affected by the composition of the powder and binder materials. The SMP used in this study contained approximately 49% lactose, which is highly hydrophilic. This hydrophilic sugar can easily absorb the sprayed binder solution and promote particle agglomeration (Atalar

Table 4. Color properties of raw and granulated ISP-enriched SMP samples with different ISP/SMP ratios

Sample	Color		
	L*	a*	b*
Raw powder			
SMP	96.0±0.09^{g}	2.80±0.07^{f}	13.8±0.18^{a}
ISP1/SMP9	93.3±0.18^{e}	1.37±0.02^{e}	13.3±0.10^{a}
ISP2/SMP8	91.7±0.18^{d}	0.76±0.02^{d}	13.5±0.16^{a}
ISP3/SMP7	91.1±0.08^{c}	0.43±0.01^{b}	13.7±0.03^{a}
Granulated powder			
SMP	94.0±0.31^{f}	3.09±0.08^{g}	18.1±0.05^{d}
ISP1/SMP9	91.5±0.16^{cd}	1.38±0.05^{e}	16.3±0.64^{b}
ISP2/SMP8	89.8±0.12^{b}	0.59±0.03^{c}	16.4±0.13^{bc}
ISP3/SMP7	88.4±0.10^{a}	0.06±0.02^{a}	17.0±0.05^{c}

Values are presented as mean of three measurements±SD. Mean values in the same column with different letters (a-g) are significantly different (P<0.05).
ISP, isolated soy protein; SMP, skim milk powder.

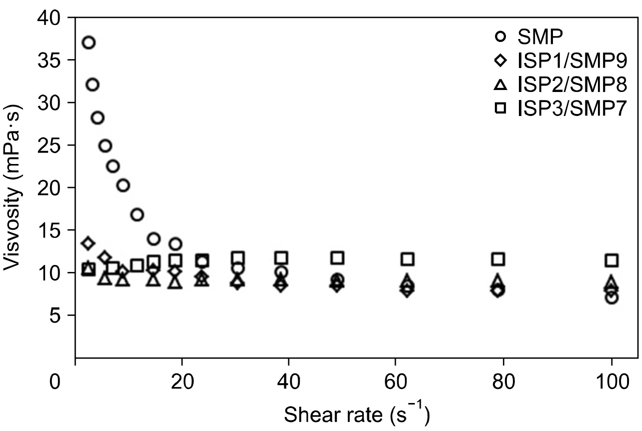

Fig. 3. Flow curves of rehydrated beverages with granulated skim milk powder (SMP) and isolated soy protein (ISP)-enriched SMP samples.

and Yazici, 2019). Consequently, ISP with a small amount of hydrophilic sugar can be less granulated during FBA than SMP with a high content of hydrophilic sugar.

Homogenous particle distribution is very important in powder products, and the particle distribution can be evaluated using a span value (Chever et al., 2017). The high span value of the granulated SMP and ISP1/SMP9 particles may be explained by partially fractured particles during FBA. The frictional force is generated by the collision between agglomerated powder particles in the fluidized bed, which can break weakly agglomerated particles (Nascimento et al., 2020; Lee et al., 2021). However, the span of the ISP3/SMP7 particles decreased significantly after FBA. This lower span value suggests that the distribution of the ISP3/SMP7 particles changed to a narrow homogenous distribution during FBA. Consequently, the FBA process can significantly improve the particle-size distribution of mixed raw powders with a wide distribution.

FBA resulted in lower ρ_{bulk} and ρ_{tapped} compared to those of the raw SMP and ISP-enriched SMP particles. A similar tendency was reported by Lim et al. (2021), who investigated FBA of probiotic-encapsulated SMPs. This result can be attributed to the increased particle size and empty space in the porous structures of the agglomerated powder particles (Ji et al., 2015; Wu et al., 2020). Consequently, the density of the powder products can be reduced by FBA due to the formation of porous structures.

The flowability and cohesiveness are important physical properties of food powders because these values are related to the weighing, transfer, and mixing of the powders (Lee and Yoo, 2021). The CI and HR levels of the ISP-enriched SMP changed to good flowability and low cohesiveness after FBA. In general, larger particles have a smaller surface area than smaller particles, which can reduce the frictional force generated between powder particles (Park and Yoo, 2020; Seo, 2022a). Therefore, FBA can improve the flowability and cohesiveness of ISP-enriched SMP particles by increasing the particle size.

FBA also improved the dispersion properties of ISP-enriched SMP particles, as proven by the shorter t_w (below 10 s). Similar results have been reported in previous studies on other isolated protein powders, including pea protein isolate (Nascimento et al., 2021) and milk protein isolate (Wu et al., 2020). Following FBA, the porous particles have empty spaces in the granulated powder structure, which can help the powder particles quickly absorb water and promote the dispersion of the powders (Custodio et al., 2020; Wu et al., 2020). From these results, it was concluded that the rheology and dispersion properties of the ISP-enriched SMP particles were significantly influenced by the change in the particle size and structure of the powders by FBA.

The color properties of the raw SMP and ISP-enriched samples were changed via FBA. After FBA, L* decreased and b* increased. A similar tendency was reported by Lee et al. (2021), who observed that FBA decreased the L* of carboxymethyl cellulose powders. The reduction in L* can be explained by particle growth, which can decrease the whiteness of food powders, as described by Sakhare et al. (2014). The high b* value of the granulated ISP-enriched SMP particles can be attributed to the Maillard reaction, which produces brown pigments at high temperatures (Seo et al., 2018). Therefore, the color of the ISP-enriched SMP particles can be influenced by particle growth and Maillard reaction that occurs during FBA.

The flow behavior of the rehydrated beverages was influenced by the ISP/SMP ratio. The SMP solution (50 g/200 mL) showed a shear-thinning behavior, which was similar to the results of Sauer et al. (2012), who reported that micellar casein solutions displayed shear-thinning behavior at concentrations above 7.5%. According to Seo and Yoo (2021), casein is a main protein in bovine milk, and most casein proteins exist as macromolecular casein micelle structures. In general, shear-thinning behavior can be explained by a decrease in the entanglement of three-dimensional macromolecules (Varesano et al., 2008). Therefore, the rehydrated solution with SMP exhibits shear-thinning behavior owing to the breakdown of casein micelle aggregates at high shear rates. In the case of the apparent viscosity at a shear rate of 50 s^{-1}, the rehydrated solution with the ISP3/SMP7 sample had a higher apparent viscosity compared to that of the SMP solution. The same result was reported by Beliciu and Moraru (2011) for micellar casein-soy protein mixtures due to the higher viscosity of soy protein.

In summary, FBA significantly improved the rheological and physical properties of ISP-enriched SMP particles. The increase in particle size due to FBA improved their physical properties (CI, HR, and t_w). Among the granulated powder samples, the ISP3/SMP7 sample exhibited the lowest CI (12.0) and HR (1.14) values. Additionally, the granulated ISP-enriched SMP particles exhibited a short t_w of less than 10 s. However, the FBA process led to a slight change in color (L* and b* values). The results of this study can be used to produce protein-enriched milk powders with good rheological and physical characteristics.

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AUTHOR DISCLOSURE STATEMENT

The author declares no conflict of interest.

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