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Effect of Pre-treatment at Subzero Temperature on the Grinding Characteristics of Grains

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Summary

The effect of pre-treatment at subzero temperature of grains on the grinding process was investigated in respect of physical properties of particle. The average particle size of ground soybean and black soybean powders decreased as pre-treatment temperature decreased. The theoretical model that described grinding characteristics revealed that the freezing as pre-treatment is effective on grinding process. In all grain samples, the Bond's constant and work index showed lower values as the pre-treatment temperature decreased. The scanning electron microscopy was used for observation of surface damages on the particles by grinding process. Some cracks were seen on the surface of particles of soybean powder ground with freezing pre-treatment. On the other hand, the particles of black soybean powder showed no fractures. The freezing as pre-treatment of grains prior to grinding process is effective to controlling their grinding characteristics and microstructure damages.

Keywords: Freezing, Frozen Food, Grinding, Grains, Particle size, Energy Consumption

1. Introduction

Grinding is an operation widely used in the food industry, the most common method for cereal grain powder processing. This process uses various machines, including crushers, grinders, mills, cutters, and homogenisers[1-2]. Grinding to break cereal grains into flour can cause damage to starch granules, disruption of starch crystalline lamellae, and degradation of starch molecules[3-6]. In addition, some grinding energy turns into heat in the grinder that can make the grain rubbery and difficult to break[7].

The grinding characteristics are affected by grain properties such as size, hardness, fibre and moisture content[8]. The unit operations such as drying[9-10], soaking[11] and freezing[12] have been used to enhance the grinding efficiency and powder quality. Especially, the freeze grinding has been mainly used for separation of fish meat. Hagura et al.[13] reported that the separation of low fat meat from whole fish of Mackerel and Sardine using the freeze grinding. They used the hammer mill for separation; the temperature was controlled to subzero temperature during grinding. Hagura et al.[14] also investigated that the freeze grinding and screening for separation of flesh and bone from the backbone offal of fish. They demonstrated that the grinding time and temperature were found to be important to optimize the efficiency of the separation. In case of soybean, Noh et al.[15] revealed the freeze treatment of soaked sample affected the coagulation of soymilk and texture of tofu. When

a solution of soyproteins is frozen, the protein molecules become partially insoluble, due to the formation of intermolecular disulphide bonds. Lee et al.[16] have shown that freezing is effective in improving the taste of soybeans, as well as reducing the cooking time to one-half. From these results, it can be expected that use of frozen soybeans may change the quality of soybean products. However, there is no consideration of grinding efficiency for soybean powder.

Actually, the grinding at low temperature has been used as very efficient method for powder production with less energy consumption[17-18]. The energy consumption in grinding to a suitable particle size should necessarily be considered for the design of grinding process. The cereal grains can be changed more brittle and easily broken by pre-treatment freezing. It also has advantages such as protection of heat labile components, a short grinding time.

However, it is difficult to control the temperature during grinding because grinders should be equipped with cooling device that leads to increase of production cost. From the viewpoint of practical use, low temperature pre-treatment prior to grinding is far more convenient but no previous study presented the effect of low temperature pre-treatment on grinding grains. The objective of this study was to investigate the grinding characteristics of grains (soybean, black soybean) in terms of particle size and energy consumption. In addition, we observed the microscopic image of particles.

2. Materials and Methods

2.1 Sample

Soybean (*Glycine Max*), black soybean (*Glycine Max*, Kuromame) cultivated from Hokkaido were purchased from local market in Japan.

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2.2 Pre-treatment

The control sample without freezing pre-treatment was ground by dry grinding. The freezing before grinding was conducted in four conditions, keeping in the stocker of -20°C (852×695×858 mm, SCR-RH22VA, Panasonic corporation, Osaka, Japan), -50°C (1500×700×945 mm, SANYO, Osaka, Japan) and -80°C (550×685×945 mm, MDF-C8V1, Panasonic corporation, Osaka, Japan). The 100 g of sample stored in tightly sealed plastic bags in the stocker for 1 day. The rest is the grains of 30 g steeped in liquid nitrogen for approximately 1 min with a ration of grain to liquid nitrogen of 2:5 w/v[12] (LN).

2.3 Texture measurement

The compressive force of the soybean and black soybean was measured using a texture analyzer (TA-XT Plus, Stable Micro System, Godalming, UK) with a load cell (5 kg). A stainless steel cylinder probe (P/3, 3 mm in diameter) was used for the compression tests. The measurements were conducted as follows: the cross head speed was 0.5 mm/s, the strain level was 10%. The measurements were repeated 12 times, with the means used for further analysis[2, 19]. Though temperature was not controlled, measurement was made within a few seconds from taking out of stocker.

2.4 Grinding process

The frozen samples were ground with a 250W cutter type domestic grinder (SKS-A700, Tiger, Tokyo, Japan). Batch grinding was conducted on 30 g of soybean and black soybean for 10, 20 and 30 s. The temperature was not accurately controlled during grinding process but the test room temperature was almost constant as 20°C. The initial moisture contents of soybean and black soybean were about 13% and 12%, respectively.

2.5 Grinding characteristics

The powders were classified using 6 different sieve sizes [1.18; 1.00; 0.60; 0.43; 0.25; 0.15 mm]. For each batch, the ground powder was placed on the top screen, and the stack was shaken mechanically for 30 min using a motorized sieve shaker (MVS-1, AS ONE, Osaka, Japan). The particles retained by each screen were removed and weighed, and the masses of those collected by each individual screen were converted to mass fractions against the total sample weight[9, 20].

$$L_2 = \sum_{i=1}^{n} \Phi_i d_i \qquad (1)$$

where L_2 is the average particle size of the grains after grinding [mm], Φ_i is the differential weight fraction of the particles passing through an aperture of size d_i, and d_i is the average aperture size of the mesh [mm]. The initial sizes of grains were measured using vernier calipers. The diameters of 30 each grains were measured.

Based on the initial and final particle sizes, Bond's constant K_b (Eq. (2)) and the Bond work index (Eq. (4)) were calculated according to the following equations[9].

$$E = K_b\left(\frac{1}{\sqrt{L_2}} - \frac{1}{\sqrt{L_1}}\right) \qquad (2)$$

$$E = \frac{t \times P}{m} \qquad (3)$$

$$W_{ind} = \frac{K_b}{0.3162} \qquad (4)$$

where L_1 is initial size of grains [mm], K_b is the Bond's constant, E is the energy required for grinding, and W_{ind} is work index. Since E [kWh/kg] is defined as the energy required to grind 1 kg of sample, it is derived by Eq. (3) where t is grinding time [h], P is power of grinder [kW], m is weight of sample [kg]. Work index is defined as the energy required to reduce a large particle size to such a size that 80% of the product passes through 100 μm sieve. These constants indicate the energy requirements for grinding[19].

2.6 Scanning electron microscope

The samples prepared for microscopic observation had particle size in range of 0.43-0.60 mm because the largest amount of sample was distributed in this range. The powders were dried using natural drying for 24 h in a desiccator. The samples were attached using the double-side carbon tape on the stub and coated using an ion sputtering coating instrument. Then, they were observed with a FE-SEM (Field emission scanning electron microscope, S-4000, Hitachi, Tokyo, Japan) operated at an acceleration voltage of 10 kV. All of the images were obtained with magnification of 300×.

2.7 Statistical analysis

All experiments were conducted three times, and the results are given as mean values. Significant differences were evaluated by one way analysis of variance (ANOVA) using MS-Excel 2013. Correlations were tested using Pearson coefficient and one way ANOVA.

3. Results and Discussion

3.1 Physical property

The compressive force of soybean and black soybean are presented in Fig. 1. Both of grains, the compressive force of control were lower than pre-treated samples. There is no significant difference between frozen samples. This result may have occurred because the grains frozen got its strength to the compression force. According to Lee et al.[10], hardness of soybean increased as moisture content of soybean decreased. Lee et al.[21] also showed a similar result on black soybean. Both of studies demonstrated that the textural property was changed by drying showed different grinding characteristics.

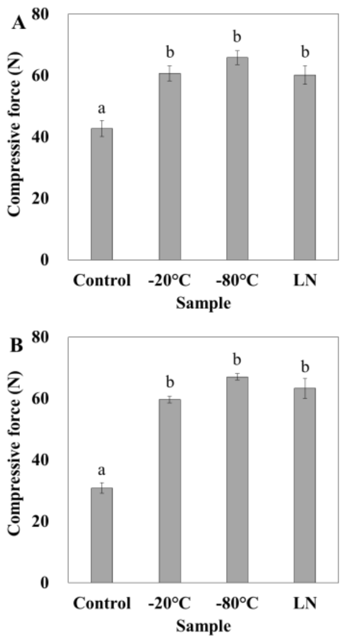

Fig. 1 Compressive force of soybean (A) and black soybean (B) (Control: dry grinding (without pre-treatment); -20°C: Stored in stocker at -20°C for 1day; -80°C: Stored in stocker at -80°C for 1day; LN: Steeped in liquid nitrogen for 1 min.)

3.2 Particle size of grain powders

The average particle sizes of soybean and black soybean were presented in Figs. 2 and 3. In all of the grains, as grinding time increased, average particle size decreased. The initial sizes of soybean and black soybean were 6.46±0.29 mm and 7.61±0.66 mm. The size reduction is more effective on black soybean, it reduced in the range of 78.55-90.95% from initial size. The soybean size changed in the range of 75.29-88.78% from initial size. These results might be due to the different moisture content, initial grain size, and hardness. In Fig. 1, the compressive force of grains increased by pre-treatment. Although the compressive force increased, the resistance to grinding was decreased, possibly due to the increase of the brittleness of grains. According to Indira and Bhattacharya[2], the size reduction of legumes was different due to their hardness and initial size. The black soybean might be the grains easy to grind by applying freezing as pre-treatment. In addition, pre-treatment temperature also affects the hardness of grains. As pre-treatment temperature decreased, the average particle size decreased. Especially, the grains steeped in liquid nitrogen (LN) showed the smallest average particle size.

According to studies on grinding of soybean and black soybean, the moisture content was important factor to size reduction. Both of grains, as the moisture contents increased, amounts of small size particle decreased than at low moisture content[10, 22]. When the moisture content was low, the grains showed brittle character. On the other hand, the high moisture content (above 100%) of soaked soybeans made the grinding process easier and could reduce the required energy and time due to textural changes[11]. Previous studies focused on the change of grind characteristics by drying and soaking.

The grinding process with pre-treatment tended to produce flour with finer particle size. The rice kernels steeped in liquid nitrogen had smaller particles than dry grinding after grinding[12]. During grinding, only one percent energy imparted to the material is utilized to create new surface areas or to loosen the bonds between the particles and remaining 99% energy is dissipated as heat which increases the temperature of the ground product[22]. This heat generation can be reduced by pre-treatment, and it may save the energy for size reduction of grains.

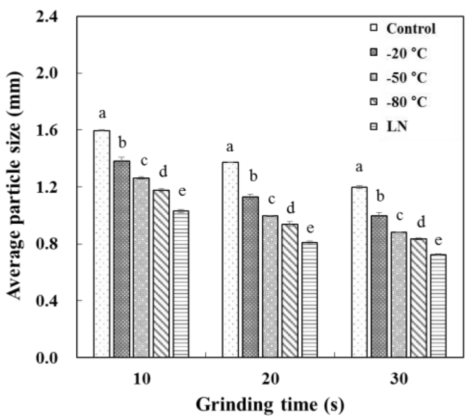

Fig. 2 Average particle size of frozen soybean by grinding time. (Control: dry grinding (without pre-treatment); -20°C: Stored in stocker at -20°C for 1day; -50°C: Stored in stocker at -50°C for 1day; -80°C: Stored in stocker at -80°C for 1day; LN: Steeped in liquid nitrogen for 1 min.)

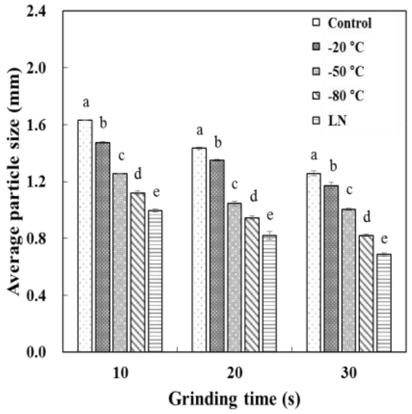

Fig. 3 Average particle size of frozen black soybean by grinding time. (Abbreviation is the same as in Fig. 2)

3.3 Grinding efficiency

Among several theoretical models to evaluate the characteristics of grinding process, Bond's law is applied in this study and Eqs. (2) and (4) have already been introduced. According to the Bond's law, the grinding characteristics such as Bond's constant, Bond's work index of grains are shown in Table 1 and 2. The constants showed lower values than previous studies. The Bond's constant of soybean with different moisture content was in range of 0.133-0.181 [kWh/kg] and work index was in range of 0.419-0.574 [kWh/kg] at grinding time 60s[10]. In black soybean with different moisture content, the Bond's content was in range of 0.054-0.333 [kWh/kg] and work index was in range of 0.171-1.053 [kWh/kg] by grinding time (10-60 s)[21]. In both of previous studies, the constants showed higher value than present data possibly because the constants were influenced by amount of sample, grinding time and the power of grinder.

Generally, the constant derived from grinding laws can describe grinding characteristics. According to Lee et al.[10], the smaller constant values indicated the less grinding energy requirement. In our study, as pre-treatment temperature decreased, the constant values decreased significantly ($p < 0.05$). This means that the requirement of energy for grinding of frozen grain is lower than control (without pre-treatment). As brittleness of grain was increased by applying an effective freezing process as pre-treatment to the natural products, more powder with a small particle size was produced for a given grinding time. Studies on grinding with other grain products reported similar results. In grinding process included freezing using the hammer mill shortened the grinding time, which was in stark contrast to the dry grinding process using the hammer mill that resulted in higher flour temperatures[12].

The work index, defined as the amount of energy required to grind a material into particles that 80% of the product passes through a mesh size of 100 µm, decreased as pre-treatment temperature decreased. Among the laws, only Bond's law could describe the energy requirement for grinding to specific particle size. The work index has been used for evaluation of energy requirement for grinding. According to Walde et al.[9], as the moisture content of wheat decreased, the Bond's work index decreased. That means the grinding energy requirement was less as moisture content was less. Velu et al.[22] also reported that the Bond's work index described that the microwave drying helped for easy grinding of the maize grains and resulted in consumption of less grinding energy. In our study, the work index was also an effective indicator for evaluating grind condition. As the pre-treatment temperature decreased, the work index decreased that resulted in less grinding energy requirement.

Additionally, the grinding efficiency also changed during grinding. The degree of size reduction decreased by grinding time. For example, the particle size of soybean powder changed in range of 4.865 to 5.429 mm from the initial size during grinding of 0-10s. However, after 10s, the size reduction was in range of 0.222 to 0.268 mm for 10s. Black soybean also showed the similar results (data are not shown). The degree of size reduction of frozen grain during 10-20s was lower than that of control. The frozen grains would have already been reduced in their size during first 10 seconds. From above consideration, the freezing as pre-treatment of grinding would realize to reduce the particle size in shorter grinding time, that led to decrease the grinding time required to the powder processing.

Table 1 Bond's constant of grain powders

		Bond's constant, K_b (kWh/kg)		
Sample	Grinding time (s)	10	20	30
Soybean	Control	0.058	0.101	0.134
	-20	0.051	0.085	0.114
	-50	0.047	0.076	0.103
	-80	0.044	0.072	0.099
	LN	0.039	0.065	0.089
Black soybean	Control	0.055	0.098	0.133
	-20	0.050	0.092	0.125
	-50	0.044	0.076	0.110
	-80	0.039	0.069	0.094
	LN	0.036	0.061	0.083

Table 2 Bond's work index of grain powders

		Bond's work index, W_{ind} (kWh/kg)		
Sample	Grinding time (s)	10	20	30
Soybean	Control	0.184	0.318	0.423
	-20	0.160	0.268	0.362
	-50	0.148	0.241	0.327
	-80	0.139	0.229	0.313
	LN	0.124	0.204	0.281
Black soybean	Control	0.174	0.310	0.415
	-20	0.159	0.294	0.391
	-50	0.138	0.238	0.346
	-80	0.126	0.220	0.296
	LN	0.114	0.198	0.261

The correlation between temperature conditions, particle size and Bond's constants in soybean and black soybean were shown in Table 3 and 4, respectively. These data suggest a positive correlation exists between the pre-treatment temperature and particle size in both grain. The temperature condition significantly correlated to the average particle size and theoretical constants of grains. Thus the pre-treatment temperature of grain samples was the variable dominated the size reduction and energy consumption in this study. Similar findings were reported for black pepper[23]. Mean diameter of black pepper powder decreased as grinding temperature decreased. The specific energy consumption was significantly dependent on the grinding temperature in the range of from -120 to -40 °C. In our study, although the temperature was not controlled during grinding, the pre-treatment has influenced the size reduction effectively.

Table 3 Correlation between temperature conditions of soybean, particle size, Bond's constant (K_b) and Bond's work index (W_{ind})

	TEMP	PS	K_b	W_{ind}
TEMP	1			
PS	0.706**	1		
K_b	0.379*	-0.251	1	
W_{ind}	0.378*	-0.252	1	1

TEMP: pre-treatment temperature of grains
PS: average particle size
*, ** indicate that the results are significant at $p<0.05$ and $p<0.01$, respectively

Table 4 Correlation between temperature conditions of black soybean, particle size, Bond's constant (K_b) and Bond's work index (W_{ind})

	TEMP	PS	K_b	W_{ind}
TEMP	1			
PS	0.806**	1		
K_b	0.408*	-0.046	1	
W_{ind}	0.408*	-0.046	1	1

Abbreviation is the same as in Table 3

3.4 Microstructure of particles

The scanning electron microscopy (SEM) images showed surface of particles from grain powders at grinding time 30s (Fig. 4). Figures 4A-E (soybean) showed protein body and cell wall on the surface of particles. Almost the same structure was observed by Rovaris[24]. Some fractures were observed on the surface of powder made from frozen soybean. These cracks might be created by freezing process as pre-treatment. The size reduction mechanism consists of deforming and breaking. Breaking of grains started along cracks in their structure when diverse forces were applied[7]. In case of soybean, existence of many cracks on surface of particles can make easy to break.

On the other hand, the particles of black soybean powder had no cracks according to SEM images (Figs. 4F-J). The grain was reduced to small particle by erosion mechanism along with grinding process. In the erosion phenomenon, the outer surface of the irregular particle may possibly undergo a friction resulting from grinder cutter causing the small irregularities on the surface to disappear generating smaller particles with more regular shapes[25]. Therefore, the cracks might be broken during grinding process so that the small particles were made. In Fig. 1, the compressive force of the black soybean increased more than that of soybean by pre-treatment. From these results, the freezing as pre-treatment on black soybean was more effective (Fig. 3). As seen in Table 1 and 2, the Bond's model constants of black soybean powder is lower than soybean powder frozen at -80°C and steeped liquid nitrogen. In addition, the Pearson coefficient of black soybean powder (Table 4) indicated more strong correlation between the pre-treatment temperatures and particle size, Bond's model constants, than soybean powders (Table 3). These results indicated the black soybean would become more breakable by pre-treatment than soybean. In spite of the temperature rise during grinding, the low temperature pre-treatment can clearly affect the physical property of grain concerning to grinding. Ngamnikom and Songsermpong[12] reported similar results. The shape and surface of particle of rice flour from each treatment (soaking and freezing) were significantly different in appearance.

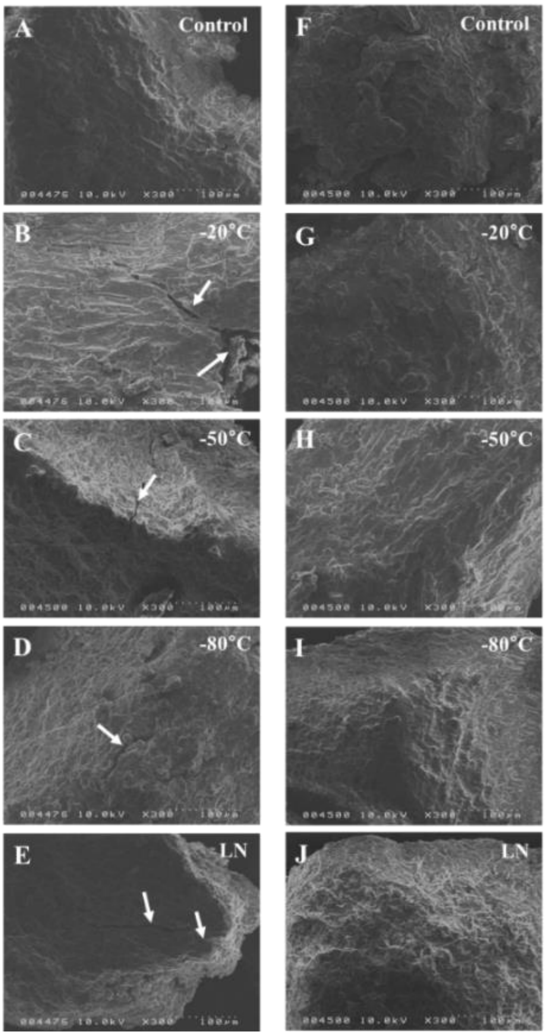

Fig. 4 Scanning electron micrographs of particle of soybean (A-E) and black soybean (F-J) powder at grinding time 30s in magnification: 300 (Control: dry grinding (without pre-treatment); -20°C: Stored in stocker at -20°C for 1day; -50°C: Stored in stocker at -50°C for 1day; -80°C: Stored in stocker at -80°C for 1day; LN: Steeped in liquid nitrogen for 1 min. The arrows exhibit cracks).

4. Conclusion

This study showed that decreasing the pre-treatment temperature of grains (soybean and black soybean) as a pre-treatment prior to grinding was valuable to control their grinding characteristics, including energy consumption and average particle size. The size reduction of grains in the control sample (without pre-treatment) was lower in the range of 72.48-83.48 % than frozen samples.

All of the grain samples, the Bond's constant and Bond's work index showed low values on frozen sample than control (dry grinding). The constant values decreased as pre-treatment temperature decreased. Some fractures were seen on the surface of particles of soybean powder ground with freezing as pre-treatment. On the other hand, the particles of black soybean powder had no cracks but the particle size was decreased instead.

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