Grinding characteristics and powder properties of the grains pre-treated with subzero temperature

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論文題目 Grinding characteristics and powder properties of the grains pre-treated with subzero temperature （氷点下温度で前処理した穀物の粉砕特性と粉の性状に関する研究）		

学位論文の要約（サマリー）

1. Introduction

Pre-treatment at subzero temperature used for grain on the grinding process. Grinding to break cereal grains into flour can cause damage to starch granules. In addition, some grinding energy turns into heat in the grinder that can make the grain rubbery and difficult to break. Therefore, the pre-treatment at subzero temperature for powder production is useful to prevent heat damage from mechanical energy and size reduction can be easier than dry grinding. From the perspective of practical use, pre-treatment at subzero temperature is more convenient but no previous study presented the effect of low temperature pre-treatment on grinding grains.

2. Objectives

The objectives of this study were to investigate the effect of pre-treatment at subzero temperature on the grinding characteristics of cereal grain, to determine the physical properties of particle and bulk powder, to evaluate the powder quality as processing raw material.

3. Materials and methods

3.1. Sample

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

3.2. 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, -50°C and -80°C. 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.

3.3. Grinding process and characteristics

The frozen samples were ground with a 250W cutter type domestic grinder. 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. The particle size of bulk powder was analyzed by particle size analyzer. Particle shape of bulk powder was evaluated by fractal dimension, an image processing technique was used. The images of each particle were obtained from scanning electron microscopy images.

3.4. Determination of starch damage

The starch damage of buckwheat powder was determined by microscopic method and chemical method. The samples prepared for microscopic observation had particle size in range of 0.43-0.60 mm. 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 field emission scanning electron microscope operated at an acceleration voltage of 10 kV. All of the images were obtained with magnification of 300× and 500×. The powder samples were prepared by sieving with size range of 150-250 μm. Starch damage of buckwheat powder was determined by the American Association of Cereal Chemists method 76-31 using a starch damage assay kit.

3.5. Flow behavior of bulk powder

Each grinding process for buckwheat powders was carried out on 200 g of buckwheat for 10, 30, 60, 90, and 120 s using a 250 W dry grinder. After grinding, the powder samples put in plastic bag, and then stored for 24 h at room temperature. The flowability of buckwheat powder were measured using powder flow tester.

3.6. Flavor component analysis

A gas chromatography was used for analysis of flavor component on buckwheat powders. The samples were prepared just before each analysis. Solid-phase microextraction method was applied.

3.7. Determination of gelatinization characteristics

Gelatinization characteristics of buckwheat powder were measured using a differential scanning calorimeter. 5 mL of distilled water was mixed to 1.5 g of buckwheat powder sample (grinding time for 90 s). All samples were scanned from 40°C to 100°C at 5 °C/min. The onset temperature (To), maximum peak temperature (Tp), conclusion temperature (Tc), and enthalpy (ΔH, J/g) of the changes were recorded.

3.8. Dough properties

Dynamic oscillatory measurement was conducted with a dynamic rheometer. Frequency sweeps test was performed from 1 to 10 Hz at a strain 0.01 % to determine the storage modulus (G'), loss modulus (G") as a function of frequency. Texture quality of boiled buckwheat dough was measured by using texture analyzer. Buckwheat dough made with 12 g of buckwheat powder (grinding time for 90 s) added 7 mL of distilled water.

4. Results and discussion

The average particle size of soybean, black soybean and buckwheat powders decreased as pre-treatment temperature decreased. The size reduction is most effective on black soybean, it reduced in the range of 78.55-90.95% from initial size. The buckwheat size changed in the range of 72.48-83.46% from initial size. These results might be due to the different moisture content, initial grain size, and hardness. The Bond's law could describe the energy requirement for grinding of grains to specific particle size. The constants derived from Bond's law decreased as pre-treatment temperature decreased. The scanning electron microscopy was used for observation of surface damages on the particles by grinding process. The cracks 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 showed no fractures. The buckwheat powder showed the sharp breakage surfaces, especially particles made by dry grinding without pre-treatment had the damaged starch granules structure. The freezing as pre-treatment of grains prior to grinding process was found to be effective to control their grinding characteristics and microstructure damages. The results showed that decreasing the temperature of soybean, black soybeans and buckwheat was valuable to control their grinding characteristics, including energy consumption and average particle size.

The bulk buckwheat powder properties were investigated as flowability and particle size distribution. The average particle size of powders made from pre-treated buckwheat seed was smaller than that of sample without pre-treatment. Bulk buckwheat powder showed a general flow function and the flow became cohesive with increasing grinding time. Buckwheat powder made from pre-treated seed was found to approach closer to the cohesive flow. It was found the pre-treatment on the buckwheat powder was effective to size reduction significantly. The ratio of change in bulk density increased as the particle size increased with grinding time increased. Therefore, the grinding process with pre-treatment tended to produce flour with finer particle size and good flowability.

The starch granules damage of buckwheat powder were observed by microscopic image and were determined by chemical method. The control powder (without pre-treatment) showed the damaged starch granules on the surfaces. On the other hand, the particle made by grinding with pre-treatment showed smooth surface of particles with less broken part. In addition, the degree of damaged starch was determined by chemical method on the particle made from pre-treated sample was less than control sample without pre-treatment. The flavor compounds abounded more in buckwheat powder ground with freezing as pre-treatment. After the 6 months storage, buckwheat powder with pre-treated at subzero temperature showed more flavor compounds retained than control. The results demonstrated that the grinding process of buckwheat with pre-treated at low temperature is useful for reducing their microstructural damage and retaining flavor component after the storage.

The control sample without pre-treatment had lower values of thermal properties were determined by differential scanning calorimeter. The viscoelastic behavior of buckwheat dough was investigated by dynamic rheometer. The storage modulus and the loss modulus of the dough made of the buckwheat powder ground with pre-treatment at subzero temperature showed lower levels than control made of powder ground without pre-treatment. However, the texture property of boiled buckwheat dough was not changed by pre-treatment. It was confirmed the pre-treatment at subzero temperature resulted in a lower content of damaged starch that could improve the quality of final product.

5. Conclusions

This study demonstrated the effect of pre-treatment with subzero temperature on the grain grinding process and powder properties. The quality of powder was evaluated and the bulk powder properties were determined. The pre-treatment at subzero temperature significantly affected the physical property of grains. It also was effective in terms of energy consumption during grinding. The grain pre-treated with subzero temperature enabled less damage during grinding. The particle size, shape, and bulk powder flowability were affected by pre-treatment of grain. In addition, the final product qualities made of the powder were expected to be improved. Consequently, this study provided important information to apply the pre-treatment at subzero temperature for grain powder processing.