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# Particle size distribution of wheat starch granules in relation to baking properties of frozen dough

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ABSTRACT

The impact of freezing on the wheat starches with different particle size was studied using a range of characterization methods including X-ray diffraction, differential scanning calorimetry, the Rapid Visco Analyser and a reconstitution dough system. Wheat starches were fractionated into A- and B-type granules, and then subjected to freezing/thawing treatment for 3 cycles. The freezing treatment did not cause apparent damage on A-type granular surface but induced cracked structure on B-type granules. It facilitated materials such as amylose, proteins, and lipids leaching from starch granule and an increase in gelatinization temperatures, melting enthalpy, and pasting viscosities. A smaller bread specific volume was obtained from freezing-treated B-granules while the crumb firmness significantly increased ($p > 0.05$). No marked differences were observed in the counterparts of A-granules after freezing treatment. It seemed that the B-type granules were more sensitive to the freezing/thawing treatment, thus facilitating structural transformations from dough to bread. Results indicated that the deterioration in frozen bread quality derived from starch could be minimized by increasing the A-granules content.



## 1. Introduction

Recently, the effect of freezing on the dough properties was a field of active research with a view to improve the quality of the final thawed product. Frozen dough gradually deteriorated and led to loss of gas retention, poor loaf volume and strong alternation textural properties (Ribotta, León, & Añón, 2001). These were mainly attributed to the loss of yeast fermentative capacity and gluten network integrity (Ribotta, León, & Añón, 2003) by ice-crystallization and re-crystallization (Yadav, Patki, Sharma, & Bawa, 2009).

Starch was an important food ingredient and constituted the largest volume fraction of solids in dough (Colonna et al., 1990). Meziani et al. (2011) reported that low temperature could increase the relative crystallinity of sweet dough as a function of starch retrogradation. Tao et al. (2016) further fractionated the starch granules from frozen dough for elucidating changes on granular structure, and found a loss of chemical components in the starch residues. During freezing, water inside the starch granule expanded channels in the granule envelope. Compression of the starch granule by water matrix formed on freezing treatment caused leaching of the material (Szymonska & Wodnicka, 2005). The surface leaching material resulted in some structure changes and accelerated retrogradation of starch or starchy foods (Tao et al., 2015). As a result, starch granules are probably another cause for deteriorated qualities of frozen dough due to a series of physical phenomena and chemical reactions on starch granules.

In mature wheat grains starch was composed of A- and B-type starch granules according to their shape and size (Lindeboom, Chang, & Tyler, 2004). The A-granules (diameter > 10 mm) were formed first in developing endosperm, whereas the B-granules (diameter < 10 mm) were formed late in kernel development (Park, Wilson, & Seabourn, 2009). Many investigators have shown that starch composition (Bertolini, Souza, Nelson, & Huber, 2003; Pérez & Bertoft, 2010), thermal properties (Chiotelli & Le Meste, 2002; Eliasson & Karlsson, 1983), enzyme susceptibility (de la Hera, Gomez, & Rosell, 2013), and baking characteristics (Rasper & Deman, 1980; Sahlström, Bævre, & Bråthen, 2003) were all affected by granule size. However, there was few literature reported on the effect of particle size distribution in relation to frozen dough quality.

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In this study, the A- and B-type wheat starch granules and their freezing-treated starches were characterized. In order to eventually solve the problem of quality deterioration in frozen doughs, a simple dough system, reconstituting with fractions of the same flour such as starch and gluten, has been used. Because the addition of purified components can be altered to establish the role of each fraction and to find which one is responsible for different baking qualities (Delcour, Vansteelandt, Hythier, & Abecassis, 2000). It could be used to study the relationship between baking properties and basic biochemical examinations of wheat starch.

## 2. Materials and methods

### 2.1. Materials

Wheat starch was provided by Puluoxing Starch Co., Ltd. (Hangzhou, China). Commercial wheat gluten [protein content ($N \times 5.7$) 62.1%] was obtained from Weijing Co., Ltd. (Shanghai, China). Yeast, sugar, and salt were purchased from a local market in Wuxi, China. All other chemicals and reagents were obtained from Sinopharm Chemical Reagent Co., Ltd. (Suzhou, China) and were of analytical grade unless otherwise stated.

### 2.2. Starch isolation and determination of granule size distribution

Aqueous suspensions (30%, w/v) of wheat starches were fractionated into A- and B- granules by centrifugation ($1000 \times g$, 10 min) (Vermeylen, Goderis, Reynaers, & Delcour, 2005) and subsequent pellet separation into top (enriched in B granules) and bottom (enriched in A granules) layers. This procedure was repeated five times on each fraction to be refined and then each fraction was dried in a convection oven at 37 °C for 48 h.

The size distribution of isolated starch granules was determined using a Malvern MasterSizer 2000 (Malvern Instrument, Ltd., UK). The concentration of the measured particles was 0.05 mg/mL within the range of the instrument's specifications. Volumes of all starch granules were calculated on the assumption that all granules were spherical in shape (Zhang et al., 2015).

### 2.3. Preparation of multiple freezing/thawing-treated starch

Fractioned wheat starch (30 g, dry basis) was soaked with 45 mL distilled water for 3 h at room temperature (25 °C). Then the starch suspension were transferred to low temperature (−34 °C) for 22 h, as described by Tao et al. (2015). The samples were then thawed to equilibrate at 25 °C for 3 h. This freezing/thawing (F/T) treatment was repeated for 3 cycles before removing the supernatants by centrifugation at $2200 \times g$ for 20 min. The starch residues were collected after being freeze-dried with Labconco FreeZone (Labconco, USA). Control samples (native A- and B-type granules) were stirred at 25 °C for 3 h and then directly centrifuged at the above conditions.

### 2.4. Chemical composition contents

The apparent amylose content was determined by the iodine binding colorimetric method (Wang et al., 2014). The protein content of centrifuged pellets was measured by the kjeldahl method (Kjeldahl, 1883). Lipid content of starches was determined by gravimetrically after extraction with Ether at 70 °C for 8 h.

### 2.5. X-ray diffraction (XRD) of starch

XRD analysis was performed using a Bunker D8 Advance X-ray Diffractometer (Bruker AXS Inc., Karlsruhe, Germany) equipped with Ni-filtered radiation Cu-$K\alpha$ (wavelength of 1.5405 Å). The scan was operated at 40 kV and 30 mA. Data were recorded over an angular range of 4° to 40° as a function of ($2\theta$) with a scanning speed of 4°/min. Prior to XRD test, native and treated starch samples were milled to powers (200 mesh) and hydrated at 75% relative humidity in a sealed vessel using saturated sodium chloride for a week (Wei et al., 2013). The XRD diffractograms were analyzed using Jade 5.0 software (Materials Data Inc., Livermore, CA, USA) and relative crystallinity degree (%) of starch was estimated from the ratio of the peak area to the total area of a X-ray diffractogram (Komiya & Nara, 1986). All experiments were performed in triplicate.

### 2.6. Granule morphology

Images of native and freezing/thawing-treated starch granules were performed using a Hitachi S-4800 (Hitachi, Japan)at an acceleration voltage of 5 kV with ×3000 magnification. The freeze-dried starch samples were placed on aluminum specimen stubs with double-sided adhesive tape and coated with gold for observation.

### 2.7. Differential scanning calorimetry (DSC)

Thermal properties of starch samples were analyzed by a differential scanning calorimetry 7000 instrument (Seiko Instruments Inc., Chiba, Japan) according to the method of Tao et al. (2015). A total weight of 3.0 mg samples (dry basis) and distilled water (6 μL) were placed in pre-weighed aluminum sample pans. The pans were sealed hermetically to prevent moisture loss and kept overnight. For all DSC runs, a sealed empty aluminum pan was used as a reference. All pans were allowed to equilibrate at 4 °C for 24 h and then heated from 30 °C to 90 °C at a constant rate of 10 °C/min using nitrogen gas (80 mL/min). The gelatinization onset, peak temperatures and gelatinization enthalpy ($\Delta H$, J/g) of starch granule were calculated by TA Thermal System Software (Muse version 1.6, SIINT, Japan, 2012).

### 2.8. Pasting properties

The pasting profiles were analyzed using a rapid visco-analyzer (Model RVA-4C, Newport Scientific Pty. Ltd., Warrriewood, Australia). The starch concentration used in the present study was 8% (Dry weight, 28 g total weight). The suspension was stirred manually using the plastic paddle before the RVA run. Test profile was programmed according to the general pasting method (Standard 2). The slurries were first held at 50 °C for 1 min, heated at a rate of 6.0 °C/min to 95 °C, maintained at that temperature for 5 min, cooled to 50 °C at a rate of 6.0 °C/min and held at 50 °C for 2 min. Constant paddle rotating speed (160 rpm) was used throughout the entire analysis except for a speed of 960 rpm for the first 10 s to disperse the samples. The average values for peak viscosity (PV) (cP), trough viscosity (TV) (cP), final viscosity (FV) (cP), pasting temperature (PT) (°C), breakdown (cP), and setback viscosity (cP) were obtained for each sample from triplicate measurements.

### 2.9. Model reconstituted bread procedure

Model breads were prepared as described by Tao et al. (2015) using a Brabender Farinograph-E (Brabender, OHG, Duisberg, Germany). The reconstituted flour consisted of wheat starch and gluten in a ratio of 86/14 calculated on dry basis content. All fractions (300 g reconstituted flours + 4.5 g yeast + 10.5 g sugar + 4.5 g salt) were pre-mixed in a 300 g pin for 5 min to improve the homogeneity of the reconstituted products. Then the reconstituted flours were hydrated to 55% (dry matter base) for 6.5 min and molded into 60 g pieces. Then they were proofed at 37 °C with 80% relative humidity for 90 min before baking (15 min, 210 °C). To evaluate