



Effect of multiple freezing-thawing cycles on structural and functional properties of starch granules isolated from soft and hard wheat

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ABSTRACT

Properties of starches isolated from soft and hard wheat dough after freezing/thawing (F/T) treatment were investigated. Significance of results was observed between isolated hard wheat starch (HWS) and soft wheat starch (SWS), but both cultivars showed an increase in the amounts of damaged starch and leaching proteins, lipids, and amylose with F/T cycles. The freezing-treated HWS exhibited a higher swelling power and peak, trough, breakdown and final viscosity than SWS after F/T treatment. The onset, peak and conclusion gelatinization temperatures and the enthalpy of the isolated HWS determined by differential scanning calorimetry, decreased throughout F/T cycles. Concomitantly, the bread containing freezing-treated HWS exhibited a lower bread specific volume and harder crumb firmness, which might be associated with its significant structural changes induced by F/T treatment.

1. Introduction

The refrigerated/frozen dough products market across the globe is expected to show a substantial growth in Compound Annual Growth Rate over 2015–2020. Freezing consists of three stages, i.e., cooling the product to its freezing point (pre-cooling or chilling stage), removing the latent heat of crystallization (phase transition stage) and finally cooling the product to the final storage temperature (tempering stage) (Kiani & Sun, 2011). The phase transition part of the freezing process involves the conversion of water to ice through the crystallization process and is the key step determining the efficiency of the process and the quality of the frozen product (Hagiwara, Wang, Toru Suzuki, & Takai, 2002). In the freezing of tissue foods, formation of large ice crystals which is mostly extracellular, results in a decrease in the number of viable yeast cells (Meziani et al., 2012), reduces gas production (Le-Bail, Nicolitch, & Vuillod, 2010), and promotes a weak gluten network (Wang, Jin, & Xu, 2015). This is reflected in a loss of dough strength and poor baking quality of frozen doughs. Several researchers have studied the effects of storage time and temperature on yeast viability (Ribotta, León, & Anón, 2003), gluten structure (Wang, Zou, Gu, & Yang, 2018; Wang, Zou, Liu, Gu, & Yang, 2018) and starch (Meziani et al., 2011). However, few of those authors focused on the study of starch in related to the wheat variety.

Wheat variety is a key determinant factor that affects wheat plant development and starch accumulation. Wheat cultivars can be classified and traded as hard wheat and soft wheat according to the endosperm hardness (Yu et al., 2015). It is one of the most important determinants of milling, baking, and end-use quality. Soft wheat requires less energy to mill, having higher break flour yield, smaller flour particle size and less damaged starch compared to hard wheat (Martin, Meyer, Morris, & Giroux, 2007). The comparatively higher proportion of intact starch granules in soft wheat flours together with the lower protein content results in lower water absorption compared to hard wheat flours (Li et al., 2013). Flours from soft wheat are used in making pastries and cookies while those from hard wheat are used for bread and other leavened products (Quayson, Atwell, Morris, & Marti, 2016). In order to reduce starch deteriorations in frozen dough, consideration about both the wheat cultivars and their starch properties are required.

This study was performed to compare the sensitivity of soft and hard wheat starch to freezing treatment. For this purpose, the effects of freezing on structural, functional (swelling behavior, pasting and thermal properties) and baking properties (specific volume and bread crumb texture) of soft and hard wheat starch were evaluated.

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2. Materials and methods

2.1. Materials

The wheat variety Aomai (soft wheat) and Zhengmai 366 (hard wheat) were used in this study. The wheat grains were milled on a pilot-scale Miag Multomat mill (Buhler, Inc., Braunschweig, Germany). The soft wheat flour composition was: moisture content 13.37%, ash content 0.68%, gluten content 7.25% (wet basis), protein content 9.58% (dry basis), and total starch content 81% (dry basis). The hard wheat flour composition was: moisture content 14.24%, ash content 0.53%, gluten content 11.70% (wet basis), protein content 11.70% (dry basis), and total starch content 75% (dry basis). Commercial wheat gluten (62.1% protein content) was obtained from Weijing Co., Ltd. (Shanghai, China). All other chemicals and reagents were obtained from Sinopharm Chemical Reagent Co., Ltd. (Suzhou, China) and were analytical grade unless stated otherwise.

2.2. Preparation of frozen dough

Frozen dough samples were prepared according to the method reported by Tao, Xiao, Wu, and Xu (2018). Briefly, the bread formula included 300 g wheat flour, 60 g sugar, 6 g salt and 180 g water. All ingredients were mixed in a Farinograph (Brabender, Duisburg, Germany) for 8 min and divided into 60 g pieces. Both dough sample sets were frozen at $-34\text{ }^{\circ}\text{C}$ for 22 h and then thawed in a $25\text{ }^{\circ}\text{C}$ water bath for 2 h. The freezing and thawing process was repeated for 0, 3 and 7 times. To evaluate the impacts on soft and hard wheat starches, the flour fraction was substituted with soft wheat flour and hard wheat flour, respectively.

2.3. Starch isolation

Native and freezing-treated starch granules were isolated from the prepared fresh dough and frozen dough, respectively, according to the method of Singh, Singh, Isono, and Noda (2010). Briefly, stiff dough was prepared by mixing 100 g flour and 50 mL water in a pan, and the dough ball was subsequently kept at $30\text{ }^{\circ}\text{C}$ for 1 h. The dough ball was then kneaded by hand in a NaCl solution (0.4 M, 150 mL), and the starch slurry was collected. Starch slurry passed through a 100-mesh nylon cloth for twice to remove bran and endosperm cell-wall impurities. The material retained on the cloth was discarded. Starch slurry was then centrifuged at $5000\times g$ for 10 min. The upper pigmented layer was carefully removed, and decanted from any more starch, which had settled after 30 min. The starch fraction along with starch from decanting step was purified by resuspending in NaCl solution and centrifuging before drying in an oven at $40\text{ }^{\circ}\text{C}$ for 48 h.

2.4. Starch compositions

The apparent amylose content was determined by the iodine binding colorimetric method. The nitrogen content of starch pellets was determined by micro-Kjeldahl methodology (Kjeldahl, 1883). The protein content (%) was calculated from protein (%) = nitrogen (%) \times 6.25. The lipid content of starches was determined gravimetrically after extraction with ether at $60\text{--}70\text{ }^{\circ}\text{C}$ for 8 h. Analyses were performed in triplicate.

The values of damaged starch (%) were obtained according to AACC 76-30A method (AACC, 2000). A fungal enzyme from *Aspergillusoryzae* (10065, Sigma Chemical Co., St. Louis, MO, USA) was used in this analysis. Analyses of all samples were performed in triplicate.

2.5. Swelling power

Swelling power of starch was measured by the method of Lv et al. (2018) with some modifications. The isolated wheat starch (1% w/v) in

volume-calibrated sealed tubes was heated at $80\text{ }^{\circ}\text{C}$ in a shaking water bath for 30 min, cooled for 5 min, and centrifuged at $3000\times g$ for 15 min. The supernatant was separated and swollen starch sediment was weighted. Swelling power (SP, g/g) was calculated as follows: $\text{SP} = \frac{\text{the weight of sediment}}{\text{the dry weight of starch}}$.

2.6. Pasting properties

Pasting properties of starch were measured by a rapid visco-analyzer (RVA-4500, Newport Scientific Pty. Ltd., Australia) using standard 1 method (Chen et al., 2018; Huang, Zhou, Jin, Xu, & Chen, 2016). Starch slurries containing 8% (w/w) starch were transferred into an aluminum canister and equilibrated at $50\text{ }^{\circ}\text{C}$ for 1 min, heated to $95\text{ }^{\circ}\text{C}$ within 3.75 min, and then maintained at that temperature for 2.5 min. The hot paste was subsequently cooled to $50\text{ }^{\circ}\text{C}$ within 3.75 min. Paddle speed was 960 rpm for the beginning 10 s to disperse the sample, and then the speed was set at 160 rpm during the measurement. The average values for peak viscosity (PV), trough viscosity (TV), final viscosity (FV), breakdown viscosity (BV = PV-TV), and setback viscosity (SV = FV-TV) were obtained for each sample from triplicate measurements.

2.7. Differential scanning calorimetry (DSC)

Thermal properties were determined by a SIINT instrument (X-DSC 7000 model; Japan). Starch (approximately 3 mg) was weighed accurately into an aluminum sample pan. Distilled water was added with a pipette to obtain starch:water ratios of 1:2 (w/v) in the DSC pans, and equilibrated at $4\text{ }^{\circ}\text{C}$ for 24 h. An empty pan was used as a reference; the sample pans were heated at a range of $20\text{ }^{\circ}\text{C}\text{--}90\text{ }^{\circ}\text{C}$ with a constant rate of $10\text{ }^{\circ}\text{C}/\text{min}$ using nitrogen gas at a flow rate of 80 mL/min (Wang, et al., 2018). The onset (T_o), peak (T_p), conclusion (T_c) temperature, and enthalpy (ΔH) of gelatinization were obtained by TA Rheology System Software Muse, version 1.6 (SIINT, Japan, 2012). Each sample was run in triplicate.

2.8. Reconstituted bread preparation

Wheat starch and gluten in a ratio of 86/14 calculated on dry basis content were blended to prepare the reconstituted flour. To evaluate the impacts of freezing, the starch fraction in the reconstituted flours was substituted with the isolated starch from frozen dough. The reconstituted flour (300 g, 14% moisture content), fresh yeast (4.5 g), sugar (10.5 g), and salt (4.5 g) were pre-mixed in a 300 g pin mixer by Brabender Farinograph-E (Brabender, OHG, Duisberg, Germany) for 5 min. 180 mL of water was added to the mixtures and hydrated for 6.5 min. After mixing, 60 g of dough was placed into an aluminum pan and fermented for 90 min at $37\text{ }^{\circ}\text{C}$ and 80% relative humidity. The samples were then baked at $210\text{ }^{\circ}\text{C}$ for 15 min and cooled at room temperature for 2 h. Specific volume of breads was determined by the rapeseed displacement.

Bread texture was measured using a TA-XT plus analyzer (Perten Instruments, Hägersten, Sweden) with a 25 mm diameter cylindrical probe (Zhang et al., 2018). Other parameters used in this study were: pre-test speed of 3 mm/s, test speed of 1 mm/s, post-test speed of 5 mm/s and 50% compression. Analyses were done using a 10-mm-thick slice from the center of bread, taking the average of three measurements made. Firmness was recorded as the peak force of the first compression for bread texture analysis.

2.9. Statistical analysis

The data were expressed as means of triplicate determinations. Statistical significance was assessed with one-way analysis of variance using SPSS 20.0 (SPSS Inc., Chicago, USA) for windows program. $p < 0.05$ was considered to be statistically significant throughout the

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