



Multi-scale structures and pasting characteristics of starch in whole-wheat flour treated by superfine grinding



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ABSTRACT

The multi-scale structures and pasting properties of starch in WWF were investigated after superfine grinding. Five particle size distributions of WWF and their corresponding starch were obtained. The grinding process reduced the particle size of WWF and starch. However, a slight increase of fragments from starch granules was observed with enhanced grinding strength because of the small decrease in starch particle size and the existence of other WWF components that undertook some of shearing force and friction during grinding. A prominent reduction in starch crystallinity was resulted due to the destruction of crystalline structure by grinding. Small-angle X-ray scattering analyses indicated the disordering in starch semi-crystalline lamellae with thinner lamellae thickness. Additionally, the ^{13}C Nuclear Magnetic Resonance spectra demonstrated the alterations in starch chain conformation by varying peak areas of starch carbons (C1 and C4). Along with these structural changes, Starch pasting characteristics showed substantial variations, indicating decreased viscosities and higher pasting stability. The results suggest that the grinding treatments influenced the structures and pasting properties of starch even at a non-separated state, the changes in starch structures were related to the variations in starch gelatinization characteristics.

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1. Introduction

Whole-wheat flour (WWF), as one of the most important whole grains, is a rich source of fiber, vitamins, minerals, and phytochemicals [1,2]. Long-term intake of whole-wheat products is associated with reduced risks of various types of chronic diseases such as obesity, diabetes, and cancer [3,4]. The physiological function and synergistic effects of the biologically active components are responsible for the beneficial effects that whole-wheat products supply with [5]. Nevertheless, the presence of bran and germ physically interferes with dough development and interrupts the formation of gluten network, leading to undesirable texture and sensory qualities of bread and noodle made from WWF [6,7]. Hence, much effort is being made to improve the qualities of whole-wheat products. Among the existing methods, milling technique is regarded one of the effective ways to reduce the destructive effects of bran and germ on end-use products.

There have been a number of milling techniques used for grinding wheat bran and WWF, such as stone mill, hammer mill, and

roller mill [8,9]. Alternatively, superfine/ultrafine grinding techniques, as emerging milling technologies that can produce fine powders on micro- and submicron-scales, are being applied to reduce bran/WWF particle size and modify the properties related to technological and physiological functionalities [10,11]. Superfine grinding effectively reduced the particle size of bran fiber and enhanced the water holding capacity, water retention capacity, and swelling capacity [12]. Ultrafine grinding could increase the specific surface of wheat bran and promote its radical scavenging activity [11]. Hemery [13] noted that ultrafine grinding was useful to develop bran-contained products and improved the bio-accessibility of phenolic acids in bran-rich breads. Additionally, Liu et al. [14] reported that entire grain grinding by an ultrafine mill increased farinograph water absorption and stability of WWF, and provided a larger height/diameter ratio and specific volume of Chinese steamed bread than the grinding process by a roller mill. In our previous study, the influences of entire kernel superfine grinding on the properties of WWF and its noodle qualities were investigated, reduced WWF particle size showed beneficial effects on the quality improvements of whole-wheat noodle [15].

Starch, as a major component in wheat flour, has prominent impacts on the quality attributes of flour-based products. The structural characteristics and physicochemical properties of starch, such

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as granule size, semi-crystalline structure, amylose/amylopectin ratio, swelling behaviors, and pasting properties, are highly related with the thermodynamic characteristics of wheat flour and rheological properties of wheat dough [16]. A higher proportion of small starch granules in noodle flour induced lower elasticity due to more dense packing of starch granules in gluten network and less open air space in dough sheet [17]. The varying starch pasting properties after freezing/thawing treatments were associated with the changes in the dynamic modulus of wheat dough [18]. Grinding processes have been reported to modify starch structural and physicochemical characteristics, and thus influence the properties of wheat flour and its products. A fine fraction from wheat flour obtained by a jet mill indicated a higher water holding capacity, faster granules swelling, and greater soluble solids leakages, which were resulted from the increased damaged starch, loosened granular structure, and enhanced leakage of amylose upon heating by the jet-milling process [19]. The lower glass transition temperature (T_g) of starch due to the decreased crystallinity and greater plasticization after ball-milling treatments was partially responsible for the reduced T_g of wheat flour [20]. A higher damaged starch and lower ordering degree of starch structure by a planetary ball-milling caused the reductions in gluten strength and thus decreased the hardness, gumminess, and chewiness of steamed bread [21].

The modifications in starch structures and physicochemical properties induced by grinding have been shown to affect the characteristics of refined flour and its product qualities. In whole-wheat system, starch is also the major constituent and occupies the largest volume fraction of solids in dough as in refined flour system. However, the changes in starch structural and pasting characteristics when WWF is subjected to grinding treatment have not been examined yet. The objective of the study is to investigate the impacts of superfine grinding on the granule morphology, crystalline structure, lamellar structure, short-range scale orders, and pasting properties of starch in WWF. This study was expected to illuminate the starch changes in WWF after superfine grinding and provide valuable information to explain the variations in WWF properties during grinding process.

2. Materials and methods

2.1. Materials

Wheat samples, Yumai 58 cultivar, were obtained from Henan Dacheng Grain and Oil Group Co., Ltd (Zhumadian, Henan, China). The moisture, starch, protein, and ash contents of the wheat samples were 11.5%, 67.4%, 14.6%, and 1.7%, respectively. All reagents used in the study were purchased from local chemical companies.

2.2. WWF preparation by superfine grinding and starch extraction

Whole-wheat flour (WWF) was first produced by milling wheat kernels in a disc-mill, and then the resulting coarse samples were re-milled for one to five times using a ZM 200 superfine centrifugal mill (Retsch, Hann, Germany) to achieve five particle size distributions. The starch in each WWF group was isolated according to the method of Singh, Singh, Isono, & Noda [22]. Total starch and amylose contents were analyzed according to the AACC International Approved Method 76-13.01 [23], and the iodine binding colorimetric method [24], respectively. The purity of isolated starch was around 99% and amylose content was 24.3% (based on total starch). The particle size distributions of WWF and starch were determined by a Mastersizer 2000 particle analyzer (Malvern, Worcestershire, UK) using the wet method. Damaged starch was determined according to the AACC International Approved Method

76-30A [23]. The resulted WWF groups and corresponding starch groups were labeled with each median diameter (MD), packed in airtight plastic bags, and stored at room temperature for further use.

2.3. Scanning electron microscopy (SEM)

Granule morphology was evaluated using a Quanta 3D scanning electron microscope (FEI Co, Tokyo, Japan). All samples were mounted on an aluminum stub using a double-sided tape, then earthed with conductive carbon paint and coated with 50 nm of gold using an ion coater. Granule morphology was observed at an accelerating voltage of 3.0 kV and the micrographs were taken at $\times 500$ magnification. During sample preparation, the procedures of mounting and coating were performed as quickly as possible to avoid possible contamination from operating environment.

2.4. X-ray diffraction (XRD)

X-ray diffraction patterns were measured by a JDX-IOP 3A diffractometer (Japan Electron Optics Laboratory, Tokyo, Japan) with copper K_{α} radiation (0.1542 nm, 40 kV, 50 mA). The diffraction angle (2θ) scanning was from 5 to 40° with a scanning speed of 10°/min. The PeakFit 4.0 software was used to calculate the relative crystallinity according to the method of Lopez-Rubio, Flanagan, Gilbert, & Gidley [25].

2.5. Small-angle X-ray scattering (SAXS)

SAXS measurements were performed by using a NanoSTAR small-angle X-ray scattering system (Bruker-AXS, Germany). The experiments were operated at 50 mA and 40 kV using copper K_{α} radiation with a wavelength of 0.1542 nm. The samples (around 60% moisture content) were equilibrated at 20 °C for 24 h before measurements. After being exposed at an incident X-ray monochromatic beam for 5 min, the data of each sample was recorded and collected. All data was normalized and the background intensity and smeared intensity were removed for further analyses.

Based on Woolf-Bragg's equation ($d_{\text{bragg}} = 2\pi/q$), d_{bragg} (the average repeat distance of semi-crystalline lamellae) was calculated by using the peak position value (q). The other parameters, d (the thickness of semi-crystalline lamellae), d_a (the average thickness of amorphous lamellae), d_c (the average thickness of crystalline lamellae), were calculated according to the methods of Goderis, Reynaers, Koch, & Mathot [26] and Wang, Liao, & Cheng [27].

2.6. ^{13}C CP/MAS nuclear magnetic resonance (NMR) spectroscopy

The solid-state ^{13}C CP/MAS NMR was performed on a Bruker Advance AV spectrometer (Bruker, Germany) equipped with a 4 mm double-resonance MAS probe. Starch sample (500 mg) was placed into the spinner and inserted into the center of magic field. The NMR spectrum was recorded at 100.613 MHz and a total of 6000 scans were accumulated with a recycle delay of 2s. The PeakFit 4.0 software was used to decompose each spectrum into several peaks through deconvolution, according to the method of Tan, Flanagan, Halley, Whittaker, & Gidley [28].

2.7. Pasting properties

The pasting properties of WWF and starch were determined by a Super 3 rapid visco analyzer (Newport Scientific, Maryland, Australia) using the AACC International Approved Method 76-21 [23]. A sample of 3.5 g (14% mb) and 25 mL of distilled water were

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