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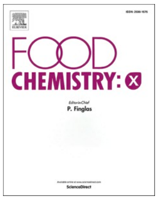

Comparison of milling methods on the properties of common buckwheat flour and the quality of wantuan, a traditional Chinese buckwheat food

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ABSTRACT

The microstructural and techno-functional properties of buckwheat flour and its processability for making wantuan, as affected by different milling methods, were investigated. Results showed that the particle sizes (d (0.5)) of the flours made by stone-milling (SM), hammer-milling (HM), laboratory grinding with steaming pretreatment for 5 min (LG-5) and 10 min (LG-10) were 95.5, 111.5, 35.4 and 41.1 μm, respectively. Moreover, SM and HM flours had less liberated starch granules and 20.84%–24.32% higher relative crystallinity than LG-10 flour. Slurries of laboratory-grinded flours showed excellent suspension stability. LG-10 flour had lowest pasting viscosities but greatest storage modulus and loss modulus. Color differences among the wantuan made from different flours were not visibly perceived ($\Delta E < 5$). Wantuan made from LG-5 flour exhibited highest textual quality due to its greatest resilience (0.376), good springiness (0.933) and accepted chewiness (1093.31). Concluding, steaming prior to grinding could improve the qualities of buckwheat flour for wantuan making.

1. Introduction

Buckwheat, a dicotyledonous herb which belongs to the Polygonaceae family and the Fagopyrum genus, is widely cultivated around the world. Among the buckwheat varieties, two species i.e., common buckwheat (*Fagopyrum esculentum* Moench) and tartary buckwheat (*Fagopyrum tataricum* Gaertn) are widely cultivated for human consumption (Sinkovič, Sinkovič, & Meglič, 2021; Huang et al., 2022). According to the statistical data of the Food and Agriculture Organization of the United Nations (FAOSTAST, 2021), buckwheat is usually planted worldwide including China, Russia, Ukraine, Kazakhstan, the United States, Poland, Japan, France and Lithuania, etc. The total cultivation area of buckwheat in the world is about 1.989 million hm^2, and the total production is about 1.875 million tons. In many nations, buckwheat is a traditional food source that rich in starch, protein, dietary fibers, vitamins, minerals, antioxidants and other bioactive substances (Giménez-Bastida, Piskuła, & Zieliński, 2015). Recently, it has been gaining popularity for the development of functional food or food ingredient due to its unique nutritional profile and health benefits. Besides, buckwheat is attracting more attention for formulating gluten-free food products, particularly for people suffering from celiac disease (Giménez-Bastida et al., 2015).

The milling of shelled buckwheat grains into flour is a common process for buckwheat utilization. As a food ingredient, buckwheat flour has been used in different types of food, such as breads, biscuits, macaroni, noodles, pilafs, pancakes, spaghetti and gel foods (Giménez-Bastida et al., 2015; Thanushree, Sudha, Martin, Vanitha, & Kasar, 2022). In China, buckwheat flour is mainly produced by using stone milling and hammer milling. Stone milling is one of the oldest ways to crush materials using forces including extrusion, shear and friction (Cappelli, Oliva, & Cini, 2020; Sinkovič et al., 2021). Yu et al (2018) reported that stone-milled buckwheat flour had large average particle size, higher retaining of flavonoids, and greater setback and final viscosities than the flour made by high-speed universal grinder or wet-milling. Hammer milling is widely used in flour production due to easy operation, low maintenance cost, time saving and high energy efficiency (Cotabarren, Fernández, Di Battista, & Piña, 2020). According to previous studies, the milling method has been reported to significantly affect the chemical compositions, the particle size, and the techno-functional properties of flours (Kadan, Bryant, & Miller, 2010; Wu et al., 2019; Thanushree et al., 2022). Hence, grain milling plays a crucial role in downstream processing and the quality of the end products (Kim & Shin, 2014; Tian, Sun, Wang, Ma, Li, & Qian, 2020).

In China, there is a traditional buckwheat food called "wantuan",

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which is popularly accepted by the folk especially in Shanxi Province. In the process of wantuan making, buckwheat flour is mixed with drinking water to form a dough, then the dough is diluted with more water to get a slurry. The slurry is placed in a steaming chamber to make a paste. The paste is stirred vigorously while it's hot and then pour into a bowl and cooled for shaping. Eventually, wantuan is made. Structurally, wantuan is a kind of starch-based gel food. To some extent, its typical texture relies on the techno-functional properties of the buckwheat flour. Wantuan is traditionally made from stone-milled buckwheat flour. In recent years, wantuan is more frequently produced by hammer-milled buckwheat flour due to the cost and efficiency in milling. However, there is rare reports that comparing the effect of buckwheat milling methods on wantuan quality.

Recently, the application of different milling methods to produce flours with desirable techno-functional properties has gained increasing attentions. Dayakar Rao, Anis, Kalpana, Sunooj, Patil, and Ganesh (2016) reported that sorghum flour made by a traditional mill had good functional properties for preparing sorghum biscuits. Cappelli et al (2020) concluded that both stone milling and roller milling offered different benefits to wheat flour quality, and it was wise to select either manner according to the end use and the product demand. Furthermore, accumulating evidences proved that thermal pretreatment of grains prior to grinding endowed benefits to the flour performance. Poudel and Rose (2018) pointed out that steaming pretreatment could improve the storage stability of wheat kernels without affecting starch and gluten properties and the flour end-use quality. Chen, Guo, Xing, and Zhu (2020) concluded that tempering with steam changed grinding properties of wheat grains and improved the rheological properties of wheat flour. Park, Oh, Chung, Shin, Choi, and Park (2022) reported that brown rice flour with increased resistant starch content and higher ratio of fine particle size was achieved by steaming the rough rice for 30 min prior to grinding. Sun et al. (2018) indicated that pregelatinization treatment of tartary buckwheat flour was a potential way to improve its properties for use in making noodles. The aim of this research, therefore, was to investigate the effect of the method of milling on the properties of common buckwheat flour and the quality of its product wantuan. Besides two common methods i.e., stone milling and hammer milling, laboratory grinding combined with steaming pretreatment was employed. The physicochemical properties and performances of the obtained common buckwheat flours were studied. Furthermore, the color and texture qualities of wantuan samples were evaluated. The present study could increase the understanding of flour milling methods on buckwheat starch-based gel foods.

2. Materials and methods

2.1. Materials

Dehulled common buckwheat (*Fagopyrum esculentum* Moench) grains were purchased from Dingzhiqiao Agricultural Science and Technology Co., Ltd. (Shanxi, China). The content of moisture, total starch, apparent amylose, protein, lipid and ash was 13.8 g/100 g (wet basis), 67.30 g/100 g (dry basis), 22.65 g/100 g (dry basis), 11.54 g/100 g (dry basis), 0.31 g/100 g (dry basis), and 1.42 g/100 g (dry basis), respectively. Table salt was purchased from China National Salt Industry Group Co., Ltd. Other reagents used in the experiment were of analytical grade and from Sinopharm Chemical Reagent Co., Ltd.

2.2. Preparation of buckwheat flour and isolation of starch

Preparation of stone-milled buckwheat flour (SM): Twenty kg of dehulled common buckwheat grains was milled into flour by a stone mill (Xianlin Stone Mill Machinery Factory, Shandong, China) using the method reported by Yu et al (2018). The roll speed of stone mill was 18 r/min, and the flour was sieved through a 90-mesh sieve and designated as stone-milled buckwheat flour (SM). The buckwheat flour yield rate of stone milling method was about 90% (w/w).

Preparation of hammer-milled buckwheat flour (HM): Twenty kg of dehulled common buckwheat grains was hammer-milled using a pilot-scale hammer mill (WF-180, Jiangsu Donglan Machinery Manufacturing Co., Ltd, Jiangsu, China) with variable rotation speed, equipped with fixed knife type blades and interchangeable screens. The flour passed through a 90-mesh sieve and designated as the hammer-milled buckwheat flour sample (HM). The buckwheat flour yield rate was about 85% (w/w).

Preparation of laboratory-grinded buckwheat flour with steaming pretreatment for 5 min and 10 min (LG-5 and LG-10): The dehulled common buckwheat grains were pretreated in a steaming chamber (100 ± 0.5 °C) for 5 min and 10 min respectively, then cooled at ambient temperature and dried in an oven at 50 °C for 12 h. The dried buckwheat grains were ground into flour using a high-speed laboratory grinder (Xichu 2000Y, Yongkang Boou Hardware Products Co., Ltd , Zhejiang, China). The obtained flour the above passed through a 90-mesh sieve and sealed in a plastic ziplock bags (polyethylene, 80 μm thick) and stored at a drug cool cabinet until further use.

The buckwheat starch was isolated using a method of Gao et al. (2022) with some modifications. The buckwheat flour was prepared in a suspension by adding distilled water at a 1:3 (w/v) ratio and homogenized for 2 min in a high-speed disperser (XHF-DY, Ningbo Scientz Biotechnology Co., Ltd, Zhejiang, China) and passed through a 120-mesh sieve. The filtrate was centrifuged at 4000 *g* for 3 min at room temperature (25 ± 1 °C). The top layer of the pellet was carefully removed, and the residue was washed several times with distilled water and pooled by centrifugation. The isolated starch was oven-dried at 40 °C for 12 h and stored in a plastic ziplock bags (polyethylene, 80 μm thick) for future use.

2.3. Characterization of buckwheat flour

2.3.1. Optical microscopic analysis

The optical images of buckwheat flour and isolated starch samples were taken under an Olympus BX53 microscope with polarized light accessory (Olympus, Japan). The buckwheat flour was dispersed in distilled water by 1:3 and dropped on the microscopy glass slide. The samples were observed using an optical microscope with a coverslip covered (Chen, Huang, et al., 2020). The observation mode was transformed between bright field and polarized light.

2.3.2. Particle-size distribution

A laser diffraction particle size analyzer (Mastersizer 2000; Malvern, UK) was used to measure the particle size of buckwheat flours. The sample was dispersed in deionized water with a beaker under the stirring of 2000 r/min until the opacity between 10% and 12%. The refractive indices of the samples and deionized water were set at 1.52 and 1.33, respectively. Results were expressed as d (0.1), d (0.5), and d (0.9), (10%, 50%, and 90% of the sample had a smaller particle size than these values, respectively) (Vicente, Villanueva, Caballeroa, Muñozb, & Ronda, 2023). Besides, the surface-weighted mean diameter (D [3,2]), the volume-weighted mean diameter (D [4,3]) and specific surface area were recorded (Yu et al., 2018).

2.3.3. Scanning electron microscopic analysis

Scanning electron microscope (SEM) (JEOL, JCM-6000 Plus, Tokyo, Japan) was employed to study the microstructure of the buckwheat flours prepared by different milling methods. Each sample was sputter-coated with gold in a sputter coater (Structure Probe, West Chester, PA, USA) before being scanned and photographed at various magnifications depending on particle size (Sun et al., 2018). An accelerating potential of 3.0 kV was used during the micrographic image acquisition.

2.3.4. X-ray diffraction characterization

The X-ray diffraction patterns of buckwheat starches were determined with an X-ray diffractometer (PANalytical X'Pert3 Powder XRD system, Almelo, the Netherlands) according to the method of Tao et al. (2021) with some modifications. Prior to analysis, the starch samples were equilibrated at 40 °C hot air oven for 24 h. Diffractograms were obtained in starch samples with CuKα radiation (λ = 0.154 nm) by scanning from 3° to 40° (2θ) with a target voltage of 40 kV, a current of 40 mA and a scan speed of $3^{\circ}\cdot\mathrm{min}^{-1}$. The relative crystallinity was the ratio of the crystallinity area to the total diffraction area with MDI-Jade 6.0 software (Material Data, Inc., Livermore, CA, USA).

2.3.5. Attenuated total reflectance-Fourier transforms infrared spectroscopy (ATR-FTIR)

ATR-FTIR spectra of buckwheat flours were recorded on a PerkinElmer Spectrum 100 with a universal ATR sampling accessory (PerkinElmer, USA). The number of scans were set to 32 with a resolution of 4 cm^{-1} from 4000 cm^{-1} to 600 cm^{-1}. The infrared absorbance ratios of 1045 cm^{-1} to 1022 cm^{-1} ($R_{1045/1042}$) and 1022 cm^{-1} to 995 cm^{-1} ($R_{1022/995}$) were calculated from the deconvolution spectra to evaluate the short-range ordered structure of starch from the buckwheat flour samples (Hu et al., 2018).

2.3.6. Suspension stability and flow properties of buckwheat flour slurry

The suspension stability of buckwheat flour slurry was measured by a sedimentation test. The buckwheat flour slurry was made by mixing the flour and distilled water at a 1:4 (w/v) ratio and homogenized for 1 min in a high-speed disperser (XHF-DY, Ningbo Scientz Biotechnology Co., Ltd, Zhejiang, China). The slurry was poured into a volumetric cylinder and let it stand. The particle matter in the slurry began to settle under the action of gravity. The delamination of the slurry at 0 min, 10 min, 20 min, 30 min, 50 min, 1 h, 2 h, 3 h, 4 h, 5 h and 24 h was recorded by photographing. The volume ratio of liquid to solid part (L/S) in the measuring cylinders was calculated at each time interval as an indication of sedimentation rate.

The flow properties of buckwheat flour slurry was studied by steady shear viscosity measurements, which was performed with a range of shear rate from 0.1 to 500 s^{-1} on a rotational rheometer (Anton Paar MCR 302, Austria) equipped with a 1.016° cone plate (60 mm, Peltier plate Aluminum) having a gap of 0.75 mm. The measuring temperature was kept at 25 °C. The changes in stress (Pa) and viscosity (Pa·s) as a function of shear rate (s^{-1}) were recorded (Ye, Wang, Wang, Zhou, & Liu, 2016).

2.3.7. Pasting properties

The pasting properties of buckwheat flours were determined using a Rapid Visco Analyser (RVA-TecMaster, Perten Instruments, Sweden) according to the RVA profile Standard Analysis. The suspension consisting of the flour and distilled water was transferred to an aluminum canister and stirred with a plastic paddle. It was held at 50 °C for 1 min, heated to 95 °C in 3.5 min and held at 95 °C for 2.5 min. The sample was then cooled to 50 °C in 4 min and held at 50 °C for 2 min. The paddle rotating speed was held at 960 r/min during the first 10 s and then maintained at 160 r/min during the process (Yu et al., 2018). The viscosity parameters were expressed in cP units (Hu et al., 2018).

2.3.8. Dynamic rheological measurements

Dynamic rheological measurements of the buckwheat flour gels were performed on a rotational rheometer (Anton Paar MCR 302, Austria) equipped with a parallel plate geometry (40 mm diameter and 10 mm gap) according to a method of Tao et al. (2021) with some modifications. The gel samples of buckwheat flour were prepared *in situ* in the aluminum canister, as described in the previous section (2.3.7. Pasting properties), and followed by an equilibration at 25 °C before subsequent measurements. The gel samples were placed between the Peltier plates and allowed to rest for 5 min at 25 °C. The strain sweeps were performed from 0.1 to 100% at 1 Hz frequency. The linear viscoelastic (LVE) region was established, and then the frequency sweep was conducted at 25 °C in the range from 0.05 to 250 rad/s under a constant strain of 1% within the LVE region. The traces of storage modulus (G'), loss modulus (G''), and loss tangent ($\tan\delta = G''/G'$) were recorded.

2.4. Preparation of wantuan

Buckwheat flour (200 g) and table salt (16 g) were mixed with distilled water (120 mL, 25 °C) and kneaded for 20 min to obtain a dough. The dough was placed in a tray to stand for 2 h at 25 °C. The dough was then blended with 680 mL distilled water (25 °C) to make a slurry. The slurry was poured into a steaming chamber preheated in advance and steamed for 25 min to obtain a cooked paste. After the completion of the cooking, the paste was taken out immediately and stirred speedily until it became a smooth paste. When it was hot, the paste was transferred to a bowl for cooling to a stiff gel state. Finally, wantuan, the end product, was formed. A video of wantuan preparation could refer to Supplementary materials 1.

2.5. Color analysis of wantuan

The determination of instrumental color parameters was performed by using a colorimeter (UltraScan PRO, HunterLab, Reston, USA) equipped with the CIELAB system. Measurements were conducted at five random parts of the wantuan samples and the corresponding values for lightness L^* ($L^* = 100$ means white; $L^* = 0$ means black), chroma a^* ($+a^*$ means redness; $-a^*$ means greenness) and hue b^* ($+b^*$ means yellowness; $-b^*$ means blueness) were recorded. The colour difference from stone-milled buckwheat flour-made wantuan was calculated by the formula (Huang et al., 2022): $\Delta E = [(L^* - L^*_0)^2 + (a^* - a^*_0)^2 + (b^* - b^*_0)^2]^{1/2}$, where L^*_0, a^*_0, and b^*_0 were the color parameters of wantuan made from stone-milled buckwheat flour.

2.6. Texture profile analysis of wantuan

Textural parameters of the wantuan samples prepared from buckwheat flour made by different milling methods were determined by a Texture Analyzer (TA. XT Plus, Stable Micro Systems, UK) using a P/36R probe (cylindrical probe with 36 mm diameter) according to a method of Wang, Wang, Wang, Qiu, & Li, (2021a) with some modifications. The pre-test, test and post-test speed were set to 2.00 mm/s, 1.00 mm/s, and 1.00 mm/s, respectively. The compression distance was 10.000 mm, triggering force was 5.0 g, and strain was set to 40%. The test was repeated at least seven times for each sample. Textural parameters i.e., hardness, adhesiveness, springiness, cohesiveness, gumminess, chewiness and resilience were recorded for the texture measurements.

2.7. Statistical analysis

All measurements were conducted at least in triplicate. Results were expressed as mean ± standard deviation of replicates. Charts were plotted by using Origin 2019b. Data were analyzed using statistical software IBM SPSS 26. Analysis of variance (ANOVA) followed by the least significant difference (LSD) were used to assess statistical differences between the values ($P < 0.05$).

3. Results and discussion

3.1. Microstructure of buckwheat flours

The optical microscope diagram (Fig. 1A) demonstrated the buckwheat groat was broken into micron-size particles by milling. At a microscopic level, the buckwheat flour consisted of large and small particles. The large particles were buckwheat cell clusters or tissue fragments and the small particles were liberated buckwheat starch

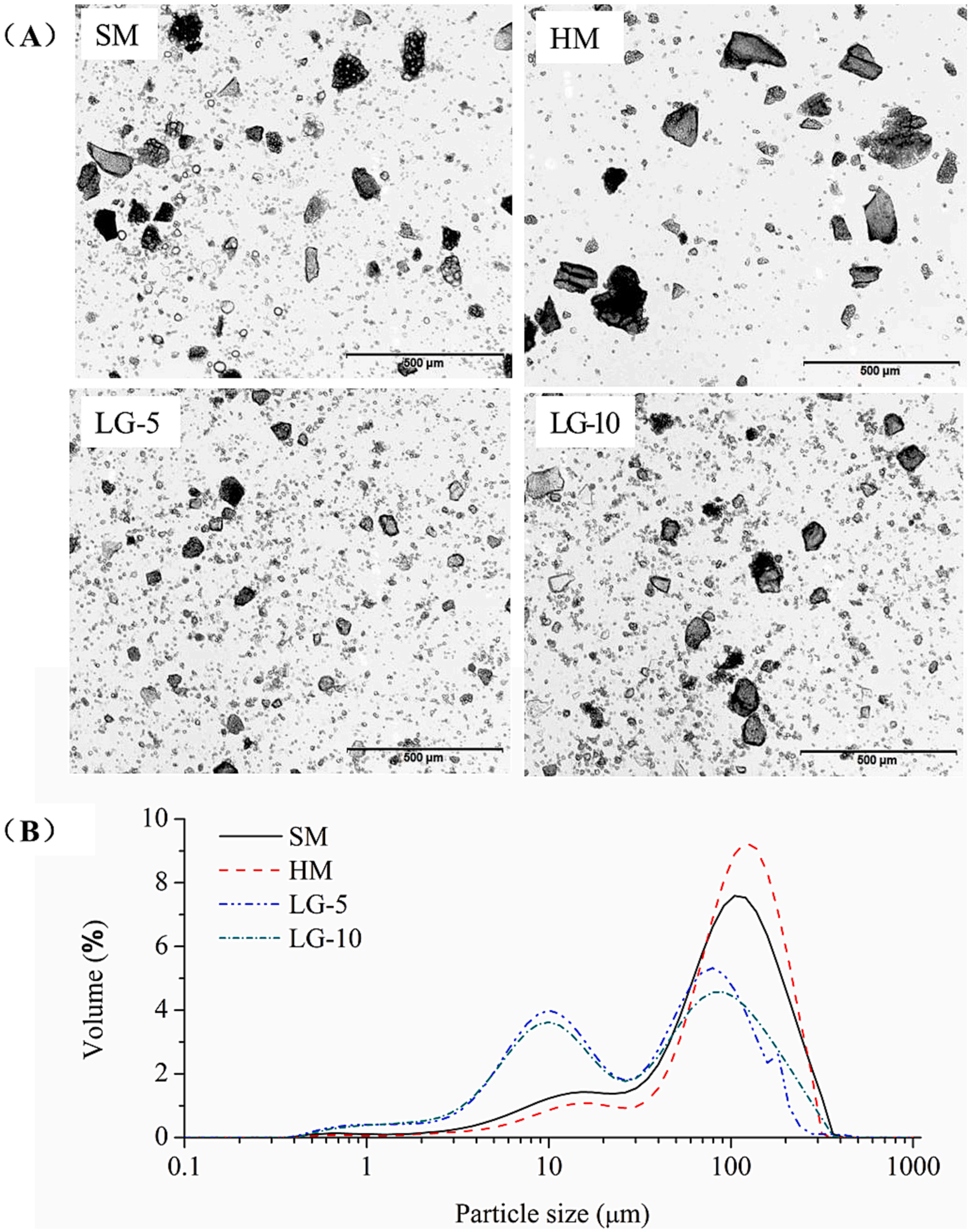

Fig. 1. (A) The morphologies of buckwheat flour under normal light microscope (NLM), scale bar = 500 μm; (B). The particle size distribution of buckwheat flours. SM, stone-milled buckwheat flour; HM, hammer-milled buckwheat flour; LG-5 and LG-10, laboratory-grinded buckwheat flour with 5 min and 10 min steaming prior to grinding, respectively.

granules (Yu et al., 2018; Chen, Huang, et al., 2020). Comparing with different milling methods, SM and HM flour have a greater proportion of larger-sized cell clusters than laboratory-grinded buckwheat flour. It was indicated that the degree of particle size reduction by using stone- and hammer- milling was limited. However, a greater degree of disintegration on the structure of buckwheat groat during the grinding was made, as the buckwheat groats were subjected to laboratory grinding with steaming pretreatment for 5 or 10 min (Yu et al., 2018; Gao et al., 2020). Comparing with stone- and hammer- milled flours, more liberated buckwheat starch granules were observed in laboratory-grinded flours LG-5 and LG-10 (Lazaridou, Vouris, Zoumpoulakis, & Biliaderis, 2018). The possible reason was that the steaming pretreatment might soften and destroy the cell wall of buckwheat grain, and laboratory grinding involed high mechanical impact on the grain during the high-speed operation, making it easier to rupture cleft. More starch granules were liberated from intact cells due to the steaming pretreatment, thus reducing the average particle size of buckwheat flour (Wronkowska & Haros, 2014; Chen, Huang, et al., 2020).

As shown in Fig. 1B, the particle size distribution of buckwheat flours presented a clear bimodal distribution, with the lower peak representing free starch granule fraction and the higher peak representing buckwheat cell clusters fraction which contained packed starch aggregates (Vicente et al., 2023). On the whole, the volume proportion of free starch granules was less than that of starch aggregates. Stone- and hammer-milled buckwheat flours had lower ratio of free starch granule fractions, which was consistent with the results showed in Fig. 1A. The particle size (d (0.5)) of stone- and hammer-milled buckwheat flours was far larger than that of the laboratory grinding samples, as listed in Supplementary materials 2 Table S1. The d(0.5) of laboratory-grinded flour was reduced from 111.5 μm to 35.4 μm (LG-5) and 41.1 μm (LG-10), respectively. Apparently, more free starch granules and looser cell clusters were made as the milling conditions become harsher (i.e., laboratory grinding with steaming pretreatment), making the flour with higher polydispersity index (calculated as (d(0.9) − d(0.1))/ d(0.5)). Stone- and hammer-milled buckwheat flours showed the lower specific surface area (0.237 and 0.164 m^2/g) among the four types of flours. As we compared the laboratory-grinded flour with different pre-steaming time, LG-10 showed greater d(0.5) value and larger particle size distribution range than LG-5. This may be due to partial gelatinization of starch during a longer period of steaming pretreatment, leading to particle agglomeration (Kim, Rahman, Lee, & Choi, 2021). Rordprapat, Nathakaranakule, Tia, and Soponronnarit (2005) and Soponronnarit, Nathakaranakule, Jirajindalert, and Taechapairoj (2006) both proved that the hydrothermal treatment could lead to the tight accumulation of starch granules, and concluded the longer the hydrothermal treatment time, the greater the gelatinization degree and the expansion degree of starch granules. Besides, the lowest D[3,2] and D[4,3] value for LG-5 indicated the highest particle shape regularity and lowest ratio of cell clusters fraction among the samples.

3.2. Structure of buckwheat starch

The microscopic structure of buckwheat starch was analyzed using optical microscopy under normal and polarized light, respectively. Under normal light (Fig. 2 BO), the isolated buckwheat starch showed polygonal, spherical or ovoid shape. It was also observed that the buckwheat starch granules were occurred in different sizes. Under polarized light (Fig. 2 PO), the buckwheat starch granules presented a typical birefringence pattern with a Maltese cross at the umbilical point, indicating that the structure of the starch granules was intact (Hu et al., 2018).

Fig. 2 also showed the micrographs of the buckwheat flour samples by scanning electron microscopy (SEM). As disclosed by SEM (Fig. 2, magnitude 1000×), buckwheat starch granules in cell clusters were packed aggregates (Huang et al., 2022). For the stone-milled flour (SM), it was observed that the aggregated starch granules were almost in spheriacal shape and adhered to cell matrix of the cell clusters, indicating the presence of broken cells in the stone-milled flour. Compared to SM, the hammer-milled flour (HM) contained intact cells with tightly packed starch granules in the cell matrix. The laboratory-grinded flours also consisted of particles in which starch granules were embedded. As examined more closely (Fig. 2, magnitude 10000×), the surface of

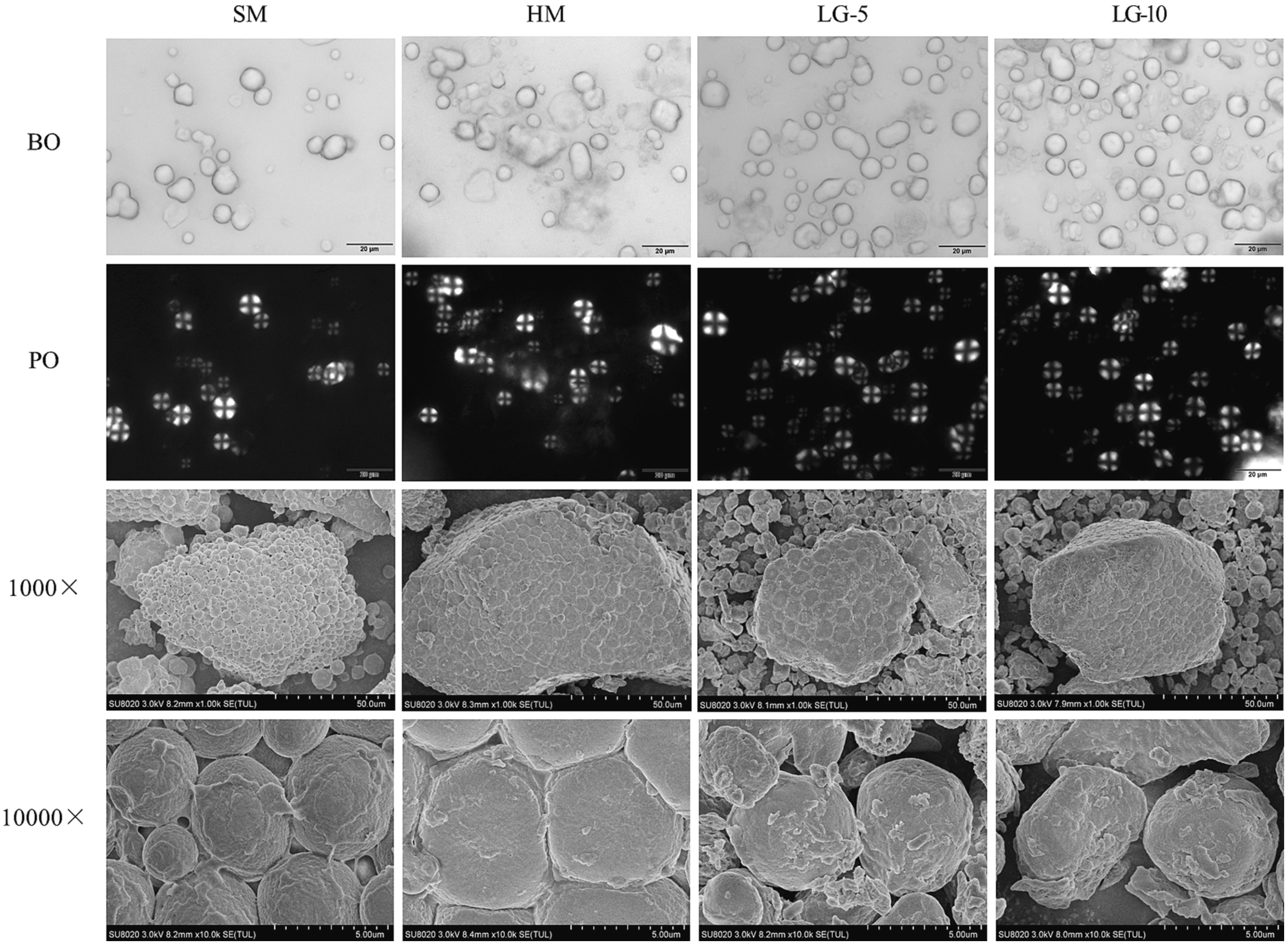

Fig. 2. The morphologies of buckwheat starch under normal light microscope (NLM), Scale bar = 20 μm (BO); and polarized 20 μm (PO), and scanning electron microscope (SEM), Scale bar = 50.0 μm (1000 ×) and 5.00 μm (10000 ×). SM, stone-milled buckwheat flour; HM, hammer-milled buckwheat flour; LG-5 and LG-10, laboratory-grinded buckwheat flour with 5 min and 10 min steaming prior to grinding, respectively.

starch granules of buckwheat flour was rough, unlike some extracted buckwheat starch granules which had smooth surface (Chen, Huang, et al., 2020; Wang et al., 2021a). It seemed like some substances adhering to the surface of the granules. Previous studies proved that a variety of adhesive substances could appear on the surface of buckwheat starch granules, including cell walls, proteins and other non-starch elements (Wang et al., 2021a; Ma, Zhang, Jin, Xu, & Xu, 2022). The surface characteristics of these starch granules from different flours were differed from each other. The surface of starch granules in laboratory-grinded flour was rougher than stone- and hammer-milled flours, which may be related to the endogenous protein denaturation and partial starch gelatinization during steaming (Sun et al., 2018; Yu et al., 2018). Besides, starch in SM showed the most granular integrity, while starch in HM was to some extent deformed. The integrity of starch granules in LG-5 and LG-10 was partly destroyed due to the steaming pretreatment.

As shown in Fig. 3A, buckwheat starch showed typical A-type diffraction pattern, with a strong peak at (2θ) 15° and 23°, and a double peak at 17° and 18° (Li et al., 2014). The same pattern was observed in starches of all buckwheat flours, indicating that different milling methods had little effect on the crystal type. Besides, at 20° (2θ) a peak with moderate intensity was found in all buckwheat flour samples, which was associated with V-type crystallinity (Vicente et al., 2023). The relative crystallinity of the starch in SM and HM showed no significant difference, as presented in Supplementary materials 2 Table S2. However, the relative crystallinity of the starch in buckwheat flours LG-5 and LG-10 was significantly lower than that in SM and HM. The decrease of relative crystallinity indicated that steaming pretreatment may induce greater fluidity of amylose and amylopectin molecules, and break partial hydrogen bonds within and between the molecules, leading to distortion and destruction of crystal structure (Hu et al., 2018; Zhong et al., 2022). The relative crystallinity of buckwheat starch in LG-10 was the lowest due to 10 min of pre-steaming.

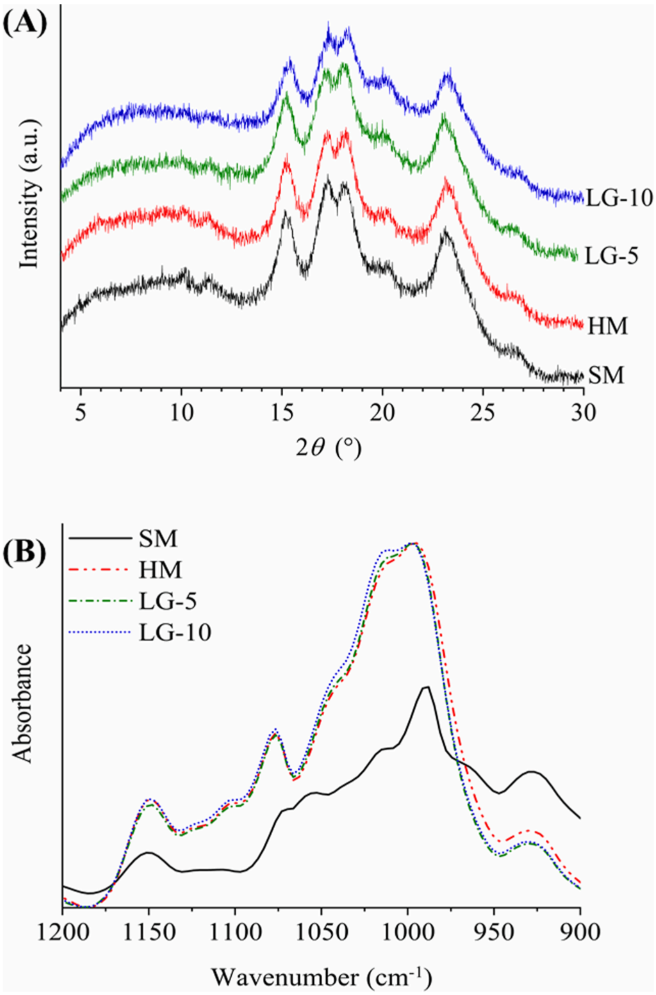

Fig. 3. X-ray diffractograms (A) and FT-IR spectra (B) of buckwheat flours made by different milling methods. SM, stone-milled buckwheat flour; HM, hammer-milled buckwheat flour; LG-5 and LG-10, laboratory-grinded buckwheat flour with 5 and 10 min steaming prior to grinding, respectively.

The ATR-FTIR spectra of the buckwheat flours were shown in Fig. 3B. The analysis was focused in the region from 900 cm^{-1} to 1200 cm^{-1}, where the most remarkable changes for starches were found. The intensity of absorbance at 995, 1022, and 1045 cm^{-1} was sensitive to changes in starch conformation. The absorbance ratio of 1045/1022 cm^{-1} ($R_{1045/1022}$) were used to quantify the degree of molecular order in the starch, and absorbance ratio of 1022/995 cm^{-1} ($R_{1022/995}$) were employed as a measure of the proportion of amorphous to ordered hydrated structure in the starch (Wang, Wang, Wang, Qiu, & Li, 2021b). As listed in Supplementary materials 2 Table S2, the $R_{1022/995}$ value of the flours followed an ascending order of HM < LG-5 < SM < LG-10. Due to the lowest $R_{1022/995}$ value, HM had the lowest portion of ordered hydrated starch structure among the flours. LG-10 had the highest $R_{1022/995}$ value, indicating steaming pretreatment for 10 min markedly maintained the interaction of water and starch molecules (Wang et al., 2021b). Furthermore, the $R_{1045/1022}$ of pre-steamed flours (LG-5 and LG-10) was lower than that of the flours SM and HM. This phenomenon was attributed to the increasing mobility of starch chains during the steaming pretreatment, which disrupted the most unstable ordered structures at a short-range scale (Wang et al., 2021b).

3.3. Suspension stability and flow properties of buckwheat slurry

In the process of wantuan making, buckwheat flour and water were mixed in a dough mixer and then kneaded to form buckwheat dough. The buckwheat dough was placed in a bowl for resting. Then, the dough was diluted with excessive water and disintegrated into slurry by kneading. The slurry was then cooked to be a paste. For the wantuan making in industrial scale, the slurry was usually piped to a steaming chamber for cooking. In this situation, the flow properties and suspension stability of the slurry were key techno-functional properties for wantuan making. If the particle matter in the slurry settled too fast, the slurry would be transformed to packed sediment layers with supernatant before cooking. Subsequently, the process of cooking showed the uneven and limited gelatinization of starch, and ultimately reflected damage to the starch gel structure and wantuan quality. Empirically, well dispersed state of the particle matter in the slurry was required for wantuan making, which was a premise to form a smooth paste when the slurry was cooking. The buckwheat flour-made slurry consisted of aqueous phase and particle matter including free starch granules, cell clusters, and ruptured cell walls. According to the Stokes settlement law, the settling rate of particle matter in the slurry depended on few factors such as the characteristics of the particle matter, the flow rate, water content and its viscosity. When the slurry was fixed with same water content and kept still, the settlement of the slurry was related to the characteristics of the particle matter and rheological properties of the slurry. It can be seen from Fig. 4A that, by 1 h or 5 h of settlement, the slurry with the same mass concentration underwent varied sedimentation i.e., in order of HM > SM > LG-5 > LG-10. The curves presented in Fig. 4B demonstrated the settling rate of particle matter from different flours during 0 ~ 5 h. Among them, the settlement of particle matter in LG-10 slurry was very slowly. However, the settlement for the slurries made of SM, HM and LG-5 flours displayed a biphasic trend, which a rapid rate within the first 1 or 2 h following a progressively decreased rate thereafter. The slurries that settled for 24 h (Fig. 4A) could be used to assess the compactness of sediment or the swelling power of buckwheat flour. It was observed that the sediment of SM slurry was tightly packed, followed by HM, LG-5 and

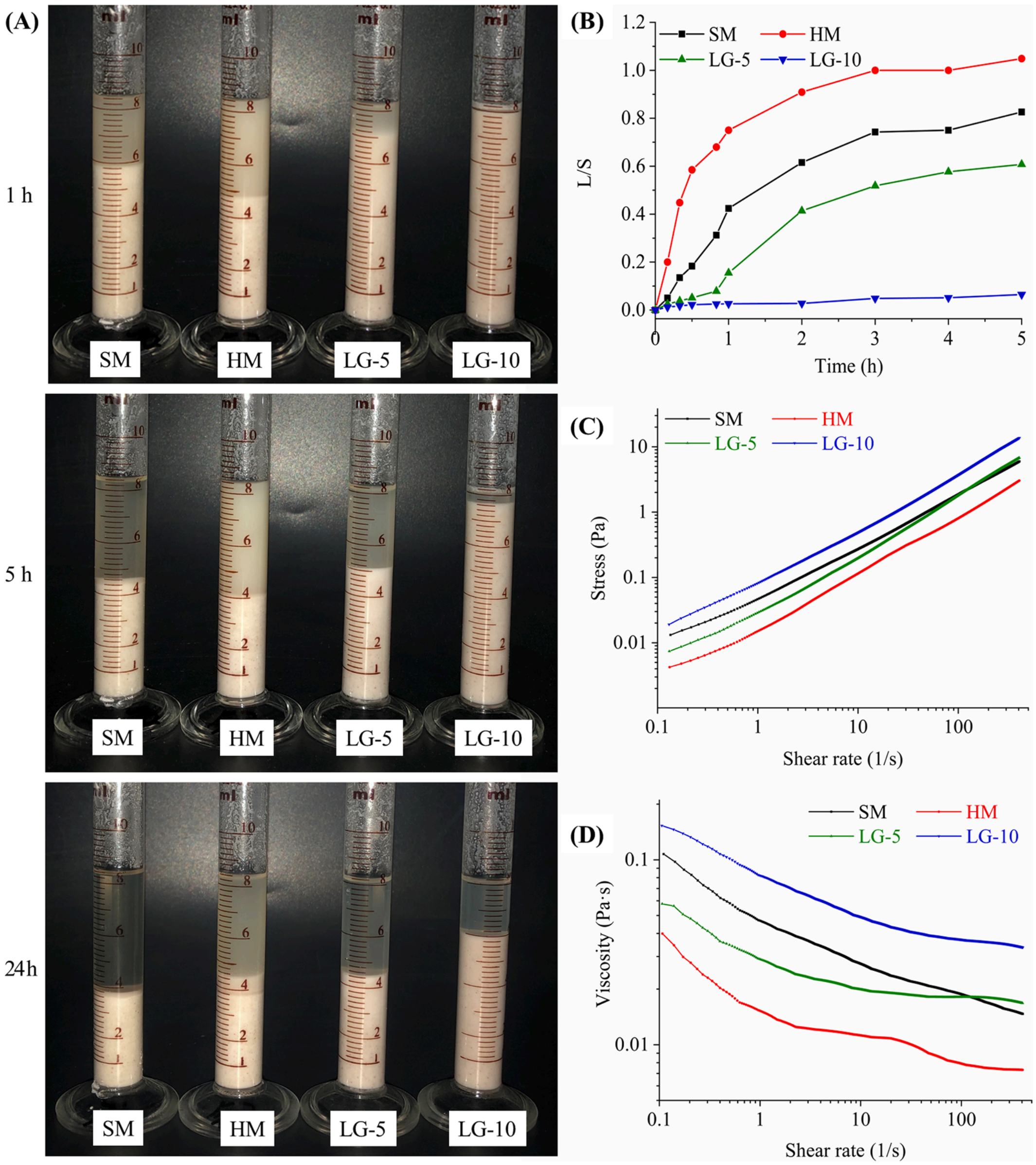

Fig. 4. Digital photographs of the slurries that settled for 1 h, 5 h and 24 h, respectively (A); flow rate of settling slurries under the action of gravity (B); relationship between stress and shear rate (C); and relationship between viscosity and shear rate (D) of buckwheat flour slurry. SM, stone-milled buckwheat flour; HM, hammer-milled buckwheat flour; LG-5 and LG-10, laboratory-grinded buckwheat flour with 5 min and 10 min steaming prior to grinding, respectively; L/S, volume ratio of upper layer (supernatant) to lower layer (precipitate) in the measuring cylinders.

LG-10. The buckwheat flour SM had the least swelling power, which was probably due to the cell structure of buckwheat retaining relatively intact in the stone milling process. By hammer milling, the tissue or cells were more fragmented, led to looser particle and more damaged starch in HM. As a result, the HM slurry showed a higher volume of solid layer than the SM slurry. Furthermore, the upper liquid of the HM slurry was relatively turbid after 24 h of settlement, indicating that the producing of ultra-fine particle matters in the hammer milling process. Larger volume of solid layer was showed in LG-5 and LG-10, indicated greater swelling power of laboratory-grinded buckwheat flours. The reasons were probably due to finer particles, higher degree of destruction to cell tissues, and the decomposition of cell walls and the gelatinization of the out-layer of starch granules under high temperature during the steaming pretreatment (Wang, Liu, & Ai, 2022). Therefore, in the perspective of suspension stability of the slurry, laboratory grinding with steaming pretreatment enhanced the processing suitability of the buckwheat flour slurry.

The buckwheat flour slurry could be regarded as a dispersed system consisting a continuous phase of soluble populations of buckwheat components and a dispersed phase of solid matters (Ye et al., 2016). Therefore, rheological property was one of the vital flow properties of the flour slurry, which played a key role in the wantuan processing. Fig. 4C showed shear stress *versus* shear rate for the slurries. The shear stress of the slurries increased as the increase of the shear rate. In range of shear rate (0.1–500 s^{-1}), the curve monotonically changed and platform trends did not appear, indicating the absence of yield or plasticity in the slurry to some extent. The shear viscosity *vs.* shear rate was presented in Fig. 4D. The shear viscosity of the slurry decreased gradually with the increase of shear rate, indicating the slurry having typical shear thinning behavior. The viscosity at low shear rate indicated the stability of the slurry. At a shear rate of 1.0 s^{-1}, the viscosity of slurry followed an ascending order of HM < LG-5 < SM < LG-10. At the same shear rate, the viscosity of slurry made of stone-milled flour was higher than that of hammer-milled flour. The results indicated that SM slurry showed greater suspension stability than HM slurry. When comparing the settlement observation results between the slurries made of LG-5 and LG-10, it was indicated that an increase in the duration of steaming pretreatment led to a more viscous slurry. This phenomenon corresponded to the starch gelatinization by steaming pretreatment, which led to improved water binding and the formation of slurry with enhanced viscosity (Wu et al., 2022). In addition, the viscosity of SM slurry was higher than that of LG-5 slurry at lower shear rate, but it eventually down to a similar viscosity toward LG-5 slurry at higher shear rate. Low viscosity at high shear rate was an advantageous feature, which made the slurry more uniform and of excellent liquidity, and consequently, a premise of good processability.

3.4. *Pasting properties of buckwheat flour*

Rapid Visco Analyser (RVA) is frequently used to record changes in the viscosity of starch aqueous suspensions when they are heated and cooled in a controlled manner. The pasting curves and derived parameters of the buckwheat flour prepared by different milling methods were shown in Fig. 5A and Supplementary materials 2 Table S3, respectively. As depicted in Fig. 5A, the pasting viscosities *versus* time curves of SM, HM and LG-5 were similar from each other, while the pasting viscosities *versus* time curve of LG-10 was much lower than other curves. The peak viscosity refers to the maximum viscosity when starch granules are expanded to the maximum extent. The RVA amylogram of HM displayed the greatest peak viscosity, followed by that of SM and LG-5. However, LG-10 had the lowest peak viscosity. Results indicated that the buckwheat starch of HM swelled to a maximum size and highest extent of starch leaching, which led to produce the greatest peak viscosity. The breakdown viscosity of the starch of HM was the greatest, indicating the worst paste stability among all four flours. Interestingly, the viscosity curves of LG-10 continued to increase during the programmed heating and cooling periods. Thus, it had no trough viscosities, no breakdown values, and no setback values. It was assumed that the swollen starch granules were not totally destroyed after pasting (Vicente et al., 2023). Less granule swelling and less leaching of amylose populations led to lower gelatinization parameters for LG-10 (Gao et al., 2022). Actually, the steaming pretreatment promoted the thermal stability of the starch in the flours. Both LG-5 and LG-10 showed higher pasting temperature than SM and HM. Karanam, Theertha, Kumar, Inamdar and Sakhare (2020) reported that an increase in pasting temperature of semolina while a decrease in viscosity was achieved by extending the hydrothermal treatment time. Furthermore, the final viscosity is an index reflecting the ability of starch pastes to form networks, and its value largely depends on the re-arrangement of soluble amylose during cooling (Kaushal, Kumar, & Sharma, 2012). In addition, the having lowest final viscosity, and setback viscosity of LG-10 was beneficial for shaping the paste in wantuan making process, and it also made a distinct texture of forming gel.

3.5. *Dynamic rheological properties of the gels made from buckwheat flours*

The curves of storage modulus (G') and loss modulus (G'') of the gels made from buckwheat flours at test frequency were depicted in Fig. 5B and C, respectively. G' represented the elastic energy of the gel, while G'' represented the energy loss by the gel to resist shear (Tao et al., 2021). As shown in Fig. 5B and C, there was a slight upward trend in G' and G'' for different samples with the increase of frequency, respectively, which exhibited slight frequency dependence. The storage modulus (G') of the gels made from four types of buckwheat flours was higher than the loss modulus (G''), indicating that the elastic characteristics of the gels were higher than their viscosity characteristics (Gao et al., 2022). Besides, SM had the lowest G' and G'', indicating that gels made from stone-milled

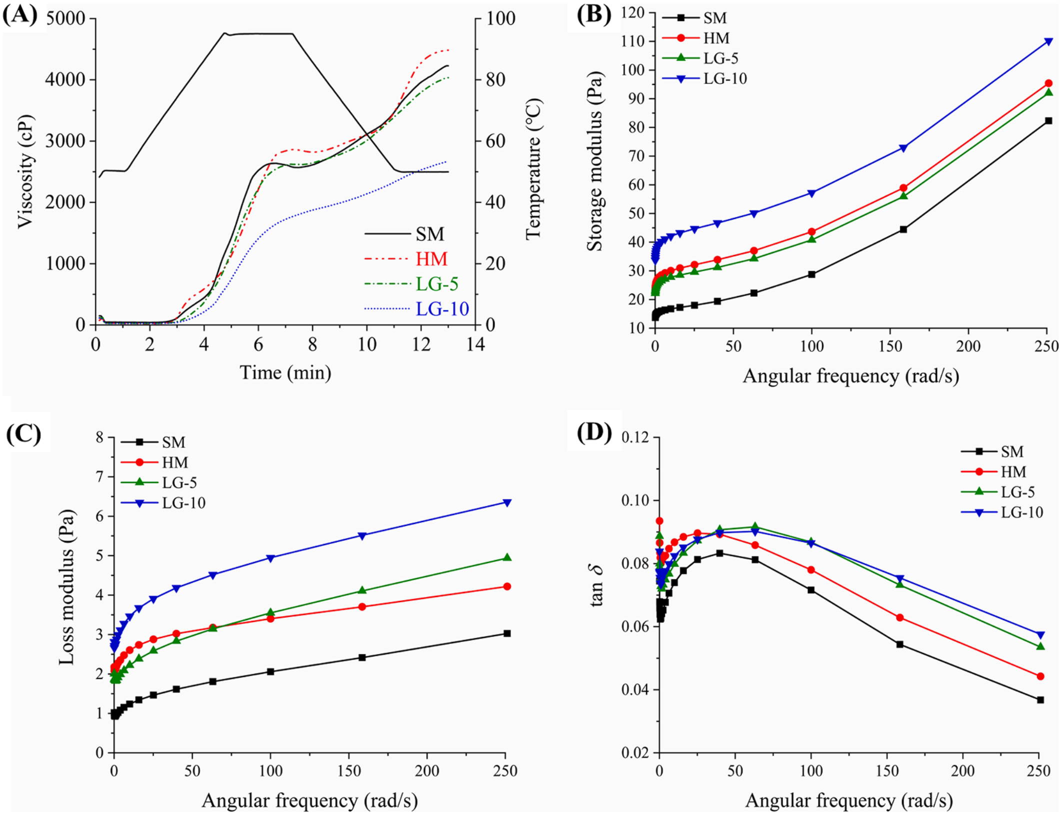

Fig. 5. Pasting curves by RVA (A); the relationship between G' and angular frequency (B); the relationship between G'' and angular frequency (C); and the relationship between tan δ and angular frequency (D) of the gels made from buckwheat flours. SM represents stone milled buckwheat flour; HM represents hammer milled buckwheat flour; LG-5 and LG-10, laboratory-grinded buckwheat flour with 5 min and 10 min steaming prior to grinding, respectively.

buckwheat flour had softer texture among them. G' and G'' were comparable and similar trend for HM and LG-5. However, it could be seen that the G' and G'' of LG-10 gel were significantly higher than those of other gels at the same frequency and exhibited better elasticity and viscosity, suggesting that 10 min of steaming pretreatment could significantly improve the structural strength of the gel. It was difficult to interpret the phenomenon with a single factor. The buckwheat flours were made up of tissue fragments, clusters of starch granules and liberated starch granules (Fig. 1A and Fig. 2). When heating the flour-made slurry, the water availability for different types of solid matter of the slurry varied, which isolated starch granules and starch granules tightly packed in the cell clusters underwent a quite different gelatinization process. Due to the protection effect of cell walls, the granule swelling and amylose leaching was limited for clusters of starch granules and tissue fragments. The paste was roughly composed of leached amylose populations, swollen and ruptured granules and disintegrated tissue fragments. Upon cooling the paste, the junction zone of a gel formed. The extent of the formation junction zone mainly depended on the state of gelatinized starch molecules. Compared to the flour SM, the flour LG-10 composed of smaller size of tissue fragments and more isolated starch granules, which was favorable to form a more uniform paste. As a matter of fact, LG-10 paste had lower viscosity than SM paste (Fig. 5A). Consequently, LG-10 paste formed gel with better three-dimensional network structures. Similar phenomenon was observed by Wu et al (2022), who investigated effect of superheated steam treatment on pasting and rheological properties kudzu starch and found that superheated steam treatment at 30% moisture content markedly lowered the pasting viscosities of kudzu starch but increased the G' and G'' values of the paste. As shown in Fig. 5D, obvious frequency dependence was observed in tan δ. At lower frequency ranges, the tan δ values increased as increasing the frequency. However, a decrease of tan δ values occurred at higher frequency ranges, indicating that the gel changed to be more visco-elastic solid at higher angular frequency.

3.6. *Color characteristics of wantuan*

Colour characteristics influence the consumer acceptability of wantuan. As indicated in Table 1, the wantuan samples made from different buckwheat flours showed some differences in the measured color parameters (L^* (lightness), a^* (±, redness/greenness), and b^* (±, yellowness/blueness)). Compared to the L^* value of wantuan made from stone-milled flour (SM), lower L^* values of wantuan made from laboratory-grinded flour (LG-5 and LG-10) indicated darker in color, as a consequence of Maillard reaction during the steaming pretreatment. To the contrary, HM wantuan showed greater L^* value than SM wantuan, probably due to higher intensity of light reflected from the wantuan surface. Furthermore, the positive values for a^* and b^* coordinates implied that each sample included variable amounts of red and yellow color. The a^* value of the four types of wantuan fluctuated from 2.61 to 4.01, followed an ascending order of SM $<$ HM $<$ LG-5 $<$ LG-10. Concerning b^* value, SM wantuan showed the highest b^* value among all four samples. This could be due to the retention of flavonoids in the stone milling process (Yu et al., 2018). Overall, color differences with respect to the wantuan made from stone milled buckwheat flour (SM) were not visibly differentiated ($\Delta E < 5$) for wantuan samples made from the flours HM, LG-5 and LG-10 (Vicente et al., 2023).

3.7. *Textural properties of wantuan*

The effects of milling methods on the textural characteristics of wantuan were evaluated, as noted in Table 1. The hardness, gumminess, and chewiness of wantuan made from LG-5 and LG-10 were significant higher than those made from SM and HM. Furthermore, the adhesiveness of wantuan made from LG-5 and LG-10 was close to that of SM ($P > 0.05$) but higher than that of HM ($P < 0.05$). The resilience of all wantuan samples showed an ascending order of HM $<$ LG-10 $\approx$ SM $<$ LG-5. Overall, the texture of wantuan was expected to be elastic and chewy. In this context, wantuan made from hammer-milled buckwheat flour was not as good as that made from stone-milled buckwheat flour. Compared with SM, ten minutes of steaming (LG-10) promoted the hardness, springiness, gumminess, and chewiness, but it had marginal effect on the resilience. However, five minutes of steaming pretreatment prior to grinding could significantly improve the wantuan processability of the flour, as reflected by the texture quality of wantuan in terms of hardness, cohesiveness, gumminess, chewiness and resilience ($P < 0.05$). Wang et al (2021b) had proved that superheated steam (170 °C, 5 min) treatment of buckwheat grain prior to grinding into flour improved the texture characteristics of buckwheat noodles. Ma, Sang, Xu, Jin, Chen, and Xu (2021) suggested that superheated steam treatment had great potential as a wheat flour modification technique for cake quality improvement.

4. Conclusions

Milling is a required process of producing buckwheat flours. The milling method has been proved to affect the characteristics of buckwheat flour and its processability for wantuan making. The findings of this study indicated that the buckwheat flour made by different milling methods varied in particle size distribution, starch multi-scale structures and techno-functional properties. Laboratory grinding combined with steaming pretreatment greatly reduced the particle size of the flours and

Table 1
Color characteristics and textural properties of wantuan made from buckwheat flours by different milling methods.

		SM	HM	LG-5	LG-10
	Color values				
	L^*	61.51 ± 0.47^{b}	62.67 ± 0.46^{a}	57.64 ± 0.28^{d}	58.64 ± 1.15^{c}
	a^*	2.61 ± 0.61^{d}	3.18 ± 0.14^{c}	3.79 ± 0.04^{b}	4.01 ± 0.10^{a}
	b^*	6.73 ± 0.19^{a}	4.86 ± 1.18^{c}	5.39 ± 0.37^{b}	5.50 ± 0.33^{b}
	ΔE	—	2.31 ± 0.28^{b}	4.29 ± 0.15^{a}	3.48 ± 1.00^{a}
Textural properties					
	Hardness (g)	565.62 ± 129.37^{c}	619.92 ± 88.19^{c}	1428.58 ± 151.35^{b}	1661.32 ± 204.71^{a}
	Adhesiveness (g·*sec*)	-181.23 ± 94.62^{a}	-319.78 ± 133.99^{b}	-172.46 ± 61.99^{a}	-174.90 ± 70.00^{a}
	Springiness	0.904 ± 0.017^{b}	0.929 ± 0.029^{ab}	0.933 ± 0.022^{ab}	0.951 ± 0.032^{a}
	Cohesiveness	0.687 ± 0.022^{b}	0.447 ± 0.031^{c}	0.754 ± 0.015^{a}	0.692 ± 0.021^{b}
	Gumminess	387.88 ± 85.69^{b}	274.95 ± 22.95^{b}	1075.46 ± 91.71^{a}	1150.23 ± 156.39^{a}
	Chewiness	350.19 ± 76.43^{b}	255.40 ± 24.14^{b}	1003.49 ± 90.95^{a}	1093.31 ± 142.38^{a}
	Resilience	0.311 ± 0.038^{b}	0.137 ± 0.017^{c}	0.376 ± 0.005^{a}	0.295 ± 0.016^{b}

Note: Data were means ± standard deviations, values with different letters for the same parameter indicate significant difference at $P < 0.05$. L^*, lightness; a^*, redness/greenness; b^*, yellowness/blueness; ΔE, the colour difference from the wantuan made from stone-milled buckwheat flour. SM represents stone-milled buckwheat flour; HM represents hammer-milled buckwheat flour; LG-5 and LG-10 represent laboratory-grinded buckwheat flour with 5 min and 10 min steaming prior to grinding, respectively.

lowered the relative crystallinity of the starch. Meanwhile, it enhanced the suspension stability and flow properties of the flour-made slurries, which is good for wantuan making. Besides, the milling method markedly affected the pasting and gel properties of the buckwheat flours. The wantuan samples made from the laboratory-grinded flour with steaming pretreatment were darker in color and stronger in gel strength. Nevertheless, color differences with respect to the wantuan made from the flours by different milling methods were not visibly differentiated ($\Delta E <$ 5). Texture test indicated that the buckwheat flour with 5 min of steaming pretreatment prior to grinding was more suitable for wantuan making than stone- and hammer-milled buckwheat flours.

CRediT authorship contribution statement

Lei Zhang: Investigation, Data curation, Formal analysis, Writing – original draft. **Qifan Meng:** Visualization, Writing – review & editing. **Guohua Zhao:** Methodology, Software, Validation, Funding acquisition. **Fayin Ye:** Resources, Conceptualization, Funding acquisition, Supervision, Project administration, Writing – review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.fochx.2023.100845.

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