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Superheated steam processing improved the qualities of noodles by retarding the deterioration of buckwheat grains during storage

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ABSTRACT

Buckwheat freshness plays a key role in the qualities of noodles. The present study focuses on the protective effects of superheated steam (SS) processing on the quality deterioration of noodles made from stored buckwheat grains. Changes of the volatile compounds, lipid degrading enzymes, pasting properties, farinograph characteristics of buckwheat grains, together with the cooking qualities and texture profiles of buckwheat noodles were investigated. Results suggested that 3-methyl-butyraldehyde and hexanal were the markers of lipid oxidation and flavor change of buckwheat grains during storage. SS treatment inactivated lipase, lipoxygenase and peroxidase and affected the generation of off-flavor volatile compounds of buckwheat grains, thus retarded the flavor deterioration of buckwheat noodles made from stored buckwheat. Storage significantly ($P < 0.05$) decreased the pasting viscosities, water absorption and development time of buckwheat dough. SS treatment maintained the pasting properties of starch stable and enhanced the elasticity and strength of the dough, leading to the improvement of cooking qualities and texture profiles of buckwheat noodles. Therefore, SS processing was an effective way to improve the qualities of buckwheat noodles by stabilizing buckwheat grains.

1. Introduction

Common buckwheat (*Fagopyrum esculentum*) is a gluten-free pseudocereal and widely cultivated all over the world (Sun et al., 2018). Based on its unique nutrients and various health benefits (Fu et al., 2020; Zhu, 2016), common buckwheat is a promising material that is suitable for functional food development and production (Guo, Wei, & Zhu, 2017). Buckwheat noodles are widely consumed in northern China, Japan, Korea and parts of Europe (Wang et al., 2019). A characteristic flavor and taste are favorite for the noodles made from just-harvested buckwheat grains and just-ground buckwheat flour with just-preparation and just-cooking. The flavor of noodles made from buckwheat grains or flour after a long-time storage are always not pleasant because of a rancid taste. However, the mechanism of the quality deterioration of noodles made from stored buckwheat grains is not well studied.

Flavor usually determines the overall sensory characteristics of food, and it plays an important role in the freshness of food during storage (Diez-Simon, Mumm, & Hall, 2019). Volatile compounds of buckwheat were composed of alcohols, aldehydes, ketones, benzene derivatives, terpenoids, alkanes, furans and so on (Prosen, Kokalj, Janeš, & Kreft, 2010; Starowicz, Koutsidis, & Zieliński, 2018). The significance of these volatile compounds for the aroma of buckwheat and its products such as noodles is unknown (Prosen et al., 2010). Volatile compounds were mainly produced through hydrolysis and oxidation of lipids during storage, as well as degradation of proteins, carbohydrates and amino acids (Liu, Li, Chen, & Yong, 2017). Lipid broken down quickly by the hydrolysis of lipase (LIP) results in the release of free fatty acids at the early stage of storage, and free fatty acids are oxidized by lipoxygenase (LOX) or autoxidation to form fatty acid hydroperoxides which are followed by further oxidative degradation by peroxidase (POD) or non-enzymatic reaction to generate off-flavor volatile compounds

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(Doblado-Maldonado, Arndt, & Rose, 2013; Suzuki, Honda, Mukasa, & Kim, 2005). The LIP and POD activities in buckwheat flour are important for the flavor generation of buckwheat noodles (Suzuki et al., 2010). Accordingly, the process of lipid degrading enzymes inactivation is critical to maintain the fresh flavor and quality control of buckwheat grains and noodles.

Superheated steam (SS) is a promising heating method to extend shelf life of cereals grains by inactivating enzyme without degradation of nutrients (Hu, Wang, & Li, 2018; Wu et al., 2016). Our previous study suggested that the hydrolysis rancidity of buckwheat grains was suppressed and the shelf life of the buckwheat grains was extended by SS processing due to its effective lipase inactivation (Wang, Wang, Qiu, & Li, 2020). However, it is still need to be clarified that the effect of SS on the change in volatile compounds of buckwheat grains during storage and its relationship with the qualities of buckwheat noodles, not only flavor but also texture. The texture of noodles is usually affected by flour pasting properties and dough farinograph characteristics. Generally, commercial buckwheat noodles are processed with additional wheat flour, as pure buckwheat noodles are easy to be broken and the soup usually becomes turbid during cooking (Wang et al., 2019). Many pre-gelatinization methods are used to modify the pasting properties of buckwheat flour so as to strengthen the network structure of buckwheat-wheat dough, including roasting, steaming, extrusion, boiling and microwave (Sun et al., 2018). Although SS treatment is reported to increase the viscosities and dough strength of wheat so that improving the firmness, springiness, and chewiness of noodles (Hu, Wang, Zhu, & Li, 2017), there is no report about the effect of SS on the texture of buckwheat noodles. Therefore, the stabilization of SS-treated buckwheat grains during storage may affect the flavor and texture of buckwheat noodles, which is important to the qualities of buckwheat noodles.

The aim of this study was to (1) determine the effects of SS treatment on volatile compounds of buckwheat grains during storage, involved with lipid degrading enzymes, and their correlation with flavor generation; (2) study the effects of SS on pasting and farinograph properties of buckwheat during storage, as well as its effects on the cooking qualities and texture profiles of buckwheat noodles; (3) using Principal component analysis (PCA) to investigate the relationships between qualities of buckwheat noodles with the changes in volatile compounds of buckwheat grains and noodles texture induced by SS treatment, so that proposing the key control points of quality deterioration of noodles made from stored buckwheat.

2. Materials and methods

2.1. Materials

High-protein wheat flour (moisture content 11.89 g/100 g and protein 12.50 g/100 g, respectively) manufactured by Inner Mongolia Hengfeng Food Industry Co. Ltd. (Inner Mongolia Autonomous Region, China) was obtained from the local market. Common buckwheat (Chiqiao 1#, main cultivar of common buckwheat in China) was grown by Chifeng Academy of Agriculture and Animal Husbandry Sciences (Inner Mongolia, China) and harvested in December 2018. Harvested grains were cleaned and dehulled using a buckwheat sheller (model TFQM-300, Qiao Brand Machinery Co., LTD, Liaoning, China), followed by drying naturally in the sun with a moisture content of 12.28 g/100 g. Then the grains were packed in polyethylene film bags per 5 kg for further experiments. All chemicals used were analytical grade and purchased from Sangon Biotechnology (Shanghai, China).

2.2. SS processing and storage experiment of buckwheat grains

Buckwheat grains were treated in a SS processing system developed by the Laboratory of Cereal Science at China Agricultural University (Beijing, China) based on our previous methods (Hu, Nie, Hu, & Li, 2016; Wang et al., 2020). Buckwheat grains (about 300 g) were scattered on the metal mesh sample tray in a uniform thin layer (2–3 mm) and conveyed into the SS processing chamber. The optimized SS processing condition (170 °C for 5 min) was used according to our previous study on the inhibition of enzymatic reactions and the rate of hydrolysis rancidity of buckwheat during storage (Wang et al., 2020). After SS treatment, the buckwheat grains were cooled down to room temperature by spreading out and ziplock-packed per 500 g (polyethylene, 80 μm in thickness). Then the packed buckwheat grains were stored at 50 °C and 60% relative humidity (Aghababaei, Maftoonazad, Elhamirad, & Badii, 2017; Wang et al., 2020; Zhang et al., 2017) in temperature-controlled incubator (BPS-50CA, Yiheng Inc., Shanghai, China) for an accelerated aging of 3 months. All the experiments were conducted in triplicate. Buckwheat grains were taken off from the incubator at intervals of one month, ground using a mill (HY-04A, Beijing Huanyatianyuan Instrument Co., Ltd, Beijing, China) and passed through a 60-mesh sieve to obtain buckwheat flour. The summary of the present study was shown in Fig. 1.

2.3. Volatile compounds analysis of buckwheat grains by HS-GC–IMS

The changes in volatile compounds of buckwheat grains during storage were analyzed according to the method of Li et al. (2019) with some modifications. Analysis were completed by an Agilent 490 gas chromatograph (Agilent Technologies, Palo Alto, CA, USA) and IMS instrument (FlavourSpec®, Gesellschaft für Analytische Sensorsysteme mbH, Dortmund, Germany), equipped with an autosampler (CTC Analytics AG, Zwingen, Switzerland) that directly sampled from the headspace by using a 1 mL airtight heated syringe.

Buckwheat flour (1.0 g) were placed into a 20 mL headspace glass sampling vial and incubated at 80 °C for 20 min. After incubation, 500 μL of headspace was automatically injected into the injector under splitless mode with a heated syringe at 85 °C. Then the samples were driven into a FS-SE-54-CB capillary column (15 m × 0.53 mm, 60 °C isothermal conditions) by nitrogen at a programmed flow as follows: 2 mL/min for 2 min, 10 mL/min for 8 min, 150 mL/min for 20 min. The analytes were ionized in an IMS ionization chamber at 45 °C. The drift gas (nitrogen gas) was set at 150 mL/min. The analysis conditions are listed in Table S1. All analyses were performed in triplicate. The retention index (RI) of volatile compounds was calculated using N-ketones C4–C9 (Sinopharm Chemical Reagent Beijing Co., Ltd, China) as external references. Volatile compounds were identified by comparing the retention index (RI) and drift time of standards in the GC–IMS library (Gesellschaft für Analytische Sensorsysteme mbH, Dortmund, Germany).

2.4. Assays of endogenous lipid degrading enzymes activities of buckwheat grains

The LIP activity was determined according to the titrimetric procedure (Wang et al., 2020) by using glycerol trioleate as a substrate and phenolphthalein as an indicator. LIP activity was quantified in terms of milligrams of 0.01M KOH required to neutralize the acid in per gram of sample. The LOX and POD activities were assayed using an ultraviolet spectrophotometry with a LOX activity detection kit (BC0320, Solarbio Science & Technology Co., Ltd, Beijing, China) and a POD assay kit (Nanjing Jiancheng Bioengineering Institute, Nanjing, China), respectively. One unit of LOX activity was defined as an increase in absorbance of 0.01 unit at 234 nm per minute per gram of sample (Zhang, Hua, Li, Kong, & Chen, 2020). POD activity was measured according to the change of absorbance at 420 nm by catalyzing H_2O_2. One unit of POD activity was defined as the amount of enzyme which was catalyzed and generated per micrograms of substrate by per gram of sample in the reaction system at 37 °C (Li et al., 2013).

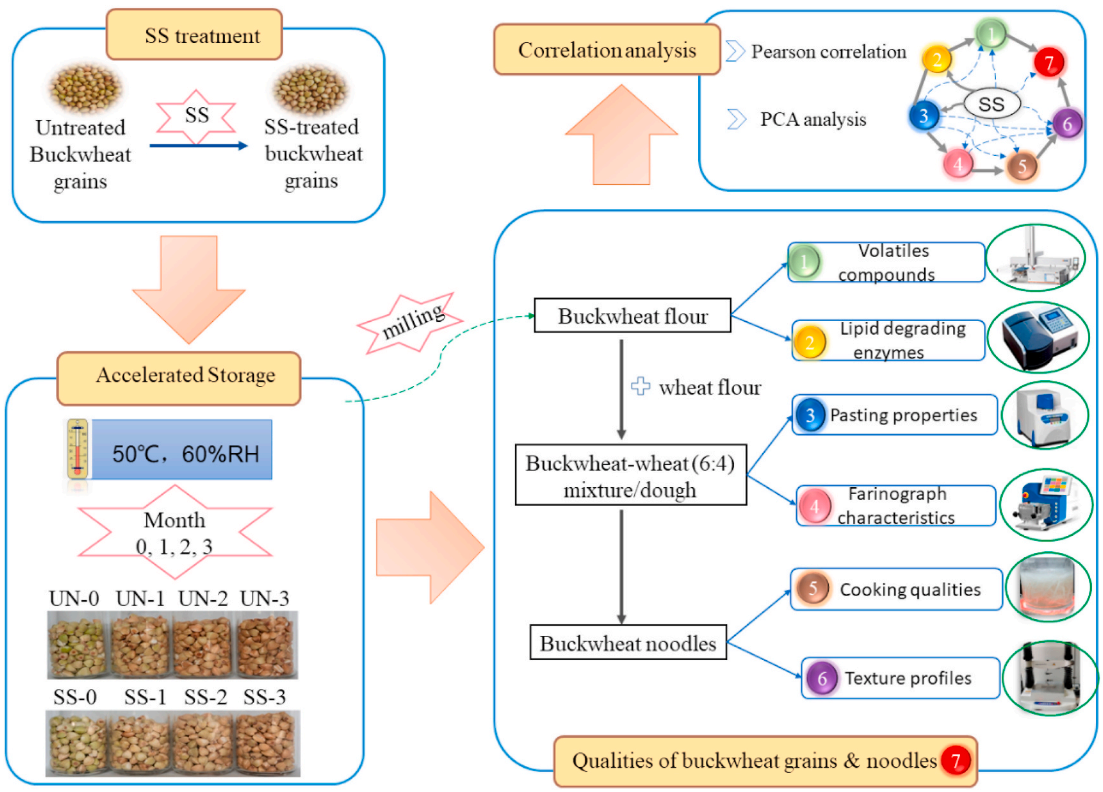

Fig. 1. Schematic diagram of the present study.

2.5. Pasting properties of buckwheat-wheat mixtures

Buckwheat flour was mixed with wheat flour at the ratio of 40: 60 to gain buckwheat-wheat flour mixtures, according to JAS 0911 "Dried Japanese noodles" (Japanese Agricultural Standard, 2019). The pasting properties of mixtures were determined by a Rapid Visco-Analyzer (RVA-4, Newport Scientific Pty. Ltd, Australia) according to the method of Sun et al. (2018). The pasting temperature, peak viscosity, trough viscosity, final viscosity, breakdown, and setback were determined. The viscosity was expressed in cP units.

2.6. Buckwheat-wheat dough farinograph characteristics

Farinograph characteristics of buckwheat-wheat mixture were determined using a Farinograph (HZF-150, Brabender, Germany) following the method of Guo et al. (2017). Water absorption, dough development time (DDT), dough stability time (DST) and degree of softening (DS) were measured.

2.7. Preparation of buckwheat noodles

Based on the method of Li et al. (2012), the dough made of 100 g flour mixture and 50 mL distilled water was rested in a plastic bag for 20 min and then passed through a roll machine (Ohtake Noodle Machine MFG. Co. Ltd, Tokyo, Japan) for 6 times to get the dough sheet. The dimensions of the resultant noodle strands were 2 mm in width, 1.25 mm in thickness and 200 mm in length. These noodles were air-dried at 30 °C and 60% relative humidity for 48 h. Dried noodles were stored in sealed polyethylene bags for further measurements.

2.8. Cooking qualities of noodles

The water absorption and cooking loss of buckwheat noodles were determined according to a reported method of Guo et al. (2017) with some modifications. 25 g of buckwheat noodles were cooked in 500 mL distilled water for 5.5 min when the core of the noodle strands disappeared. The cooking water was collected into the volumetric flask and adjusted to 500 mL with deionized water. An aliquot of 100 mL was added to the beaker and evaporated in an air oven at 105 °C until dryness. The residue was weighed and the cooking loss was calculated as the percentage of the raw noodles. The water absorption was expressed as the mass ratio after and before cooking. All experiments were conducted in triplicate.

2.9. Texture profiles of noodles

Texture evaluation of cooked noodles was conducted with a TA-XT2i Texture Analyzer (Stable Micro System Ltd., London, England) according to the published method of Guo et al. (2017) with some modifications. The cooked buckwheat noodles were immersed in cold water for 1 min. For texture profile analysis (TPA), five cooked noodle strands were placed on a metal platform and compressed by a P/50R probe at the speed of 1.0 mm/s with 50% compression ratio and 5 g triggering force. For tensile test, the A/SPR probe was used at the test speed of 0.5 mm/s and distance of 80 mm. The maximum force and distance at the extension limit were obtained. Analyses were repeated at six times for each set of noodles.

2.10. Statistical analysis

The volatile compounds were analyzed by different perspectives using Laboratory Analytical Viewer (LAV, G.A.S., Dortmund, Germany) and three plug-ins (G.A.S., Dortmund, Germany) as well as GC × IMS Library Search. The significant differences ($P < 0.05$) in means were analyzed statistically by one-way analysis of variance (ANOVA) and Duncan's multiple range tests by using SPSS software of version 18.0 (SPSS Institute, Chicago, USA). The correlations among the studied variables in the samples were determined by two-tailed Pearson correlation analysis ($P < 0.01$). PCA was performed using Origin software (version 2019b, Microcal Inc., MA, USA).

3. Results and discussion

3.1. Changes in volatile compounds of untreated and SS-treated buckwheat grains during storage

The flavor deterioration of buckwheat grains during storage could seriously affect the flavor of buckwheat noodles and then affect their sensory properties. The volatile compounds in buckwheat grains at different stage of storage were visualized in the form of topographic plots (Fig. 2. A & B). It could be seen that most of the signals appeared in the retention time of 100–900 s and the drift time of 1.0–1.8. Red dots increased gradually with prolonged storage, especially in UN-2 and UN-3, indicating that the variety and content of volatile compounds increased after storage, which might attribute to lipid oxidation. After SS treatment, the signal intensities of some volatile compounds disappeared or decreased, while those of other volatile compounds increased. Meanwhile, the red spot areas in SS-treated buckwheat were distinctly smaller than those in untreated buckwheat, which suggested that SS treatment might suppress the change in buckwheat flavor during storage.

A total of 53 target compounds from topographic plots were identified by the GC × IMS Library including 25 aldehydes, 14 alcohols, 11 esters, 2 ketones and 1 furan (Table 1). The formation of these volatile compounds is related to the degradation of lipids through both non-enzymatic and enzymatic reactions (Tian et al., 2020). Moreover, the difference in volatile compounds and the dynamic changes in each compound during storage were revealed intuitively through Gallery Plot (Fig. 2 C & D). N-nonanal, hexanal, 1-hexanol, 3-methylbutanol, ethanol, gamma-butyrolactone and acetone were the main volatile compounds of buckwheat. Before storage, the signal intensities of butyl propionate and ethyl hexanoate were stronger in UN-0, and those of 2-methylbutanal, hexanal and 6-methyl-5-hepten-2-one were stronger in SS-0. The ester derivatives, hexanal, 2-methylbutanal, and 6-methyl-5-hepten-2-one was known to have a green, grassy and sweet fruity flavor (Garrido-Delgado, Dobao-Prieto, Arce, & Valcárcel, 2015), which might be associated with the fresh flavor of buckwheat. This difference in volatile compounds between UN-0 and SS-0 indicated that SS treatment changed the flavor of buckwheat before storage, which might be associated with the slight hydrolysis of glycerophospholipids induced by SS (Wang et al., 2020).

During the first two months of storage, the signal intensities of most alcohols (2-propanol, 2-methylpropanol, 3-methylbutanol, 1-hexanol, 1-heptanol and pentanol) and part of aldehydes (pentanal, octanal and heptanal) in untreated buckwheat increased gradually. However, the signal intensities of most esters (butyl acetate, propyl bytanoate, butyl propionate, ethyl acetate and ethyl hexanoate) and part of alcohols (2-propanol, ethanol and 2-methylpropanol) in SS-treated buckwheat increased. After three months of storage, the signal intensities of alcohol volatile compounds in untreated buckwheat decreased, while that of aldehyde substances still increased continuously. The aldehyde volatile compounds, including E−2-hexanal, E−2-heptanal, 3-methylbutanal, benzaldehyde, furfural, 2-methylbutanal, and E−2-octenal, showed strongest signal intensities in UN-3 of all untreated samples. The changes of alcohols and aldehydes might be related to the oxidation of polyunsaturated fatty acids such as linoleic acid and linolenic acid, which probably accelerated by lipid degrading enzymes in the buckwheat during storage (Klensporf & Jeleń, 2008; Lampi et al., 2015; Suzuki et al., 2005). However, SS treatment could suppress this flavor deterioration probably by its inactivation of LIP, LOX and POD, which was explained in 3.2. Moreover, with the extension of storage time, the signal intensities of 3-methylbutanal dimer and hexanal dimer increased continuously, while SS treatment could significantly delay their changes, indicating that 3-methylbutantal and hexanal could be used as markers of lipid oxidation and flavor change during buckwheat storage.

3.2. Effect of SS on lipid degrading enzymes of buckwheat grains during storage

Many studies indicated that the lipid degradation and oxidation induced by LIP, LOX, and POD are the main causes of the flavor deterioration of cereal. The protective effect of SS treatment on buckwheat fresh flavor observed above was closely relevant to the changes in LIP, LOX and POD activities of buckwheat during storage (Fig. 3A-C). Before storage, SS treatment significantly ($P < 0.05$) decreased the LIP, LOX and POD activities by 50.06%, 97.18% and 71.86%, respectively. The LIP, LOX and POD activities of untreated buckwheat decreased rapidly during storage. However, the SS-treated buckwheat had lower LIP, LOX and POD activities during storage, and the slopes of SS-treated buckwheat was lower than those of untreated buckwheat. The lower level of lipid degrading enzymes variation in SS-treated buckwheat during storage indicated that SS inactivated the lipid degrading enzymes, suppressed the rate of lipid hydrolysis and oxidation, changed the metabolic pathways of volatile compounds, and thus inhibited the flavor deterioration of buckwheat. The suppress effect of SS treatment on lipid hydrolysis and oxidation has also been evidenced in lightly milled rice and wheat bran (Hu et al., 2017; Wu et al., 2016). The unsaturated fatty acids accounted for a large proportion of fatty acids (75.28%) in buckwheat (Wang et al., 2020), and the changes in alcohols and aldehydes volatile compounds were related to the enzymatic oxidation of unsaturated fatty acids such as linoleic acid and linolenic acid. Therefore, the correlations between lipid degrading enzymes and volatile compounds were analyzed using Pearson correlation analysis (Fig. 3. D). The results showed that LIP activity was significantly positively ($P < 0.05$) correlated with the signal intensities of 6-methyl-5-hepten-2-one, propyl bytanoate, and ethanol, while negatively ($P < 0.05$) correlated with ethyl acetate, 3-methylbutanal, 2-methylbutanal, pentanal, hexanal, (E)-2-heptanal, benzaldehyde, octanal, (E)-2-octenal, n-nonanal, 3-methylbutanol, 1-pentanol, and 1-octen-3-ol. The activity of POX had significant positive ($P < 0.05$) correlations with the signal intensities of ethanol and 6-methyl-5-hepten-2-one, while negative ($P < 0.05$) correlations with ethyl acetate, (E)-2-heptenal, octanal, pentanal, hexanal and furfurol. The activity of LOX had no significant correlation with most volatile compounds. It indicated that LIP and POX were important for generation of volatile compounds in buckwheat, which was in accordance with the findings of Suzuki et al. (2005) that buckwheat flours with high contents of LIP and POX tended to deteriorate quickly. These findings suggested that SS could change the generation of volatile compounds of buckwheat flour by the inactivation of the lipid degrading enzymes, and thus inhibited the flavor deterioration of buckwheat noodles.

3.3. Changes in pasting properties of buckwheat mixture during storage

The characterization of RVA paste viscosities was used to evaluate the improvement of SS treatment on the noodle qualities made from buckwheat-wheat mixture (Table 2). Compared with the untreated buckwheat (UN-0) before storage, a significant decrease in peak viscosity and breakdown of SS-0 ($P < 0.05$) showed that there was partial starch gelatinization in SS-treated buckwheat. It was consistent with the previous finding that SS decreased the peak viscosity and breakdown of lightly milled rice (Wu et al., 2016). The pasting properties including peak, trough and final viscosities and setback of untreated and SS-treated buckwheat significantly decreased during three months of storage, which agreed with the previous study of rice (Ziegler et al., 2017). However, the decreases in peak, trough and final viscosities of SS-treated buckwheat were slower than those of untreated one. It indicated that SS slowed down the changes in pasting properties during storage. The reductions in pasting viscosities might be due to the reduced ability of the starch granules to absorb water prior to physical

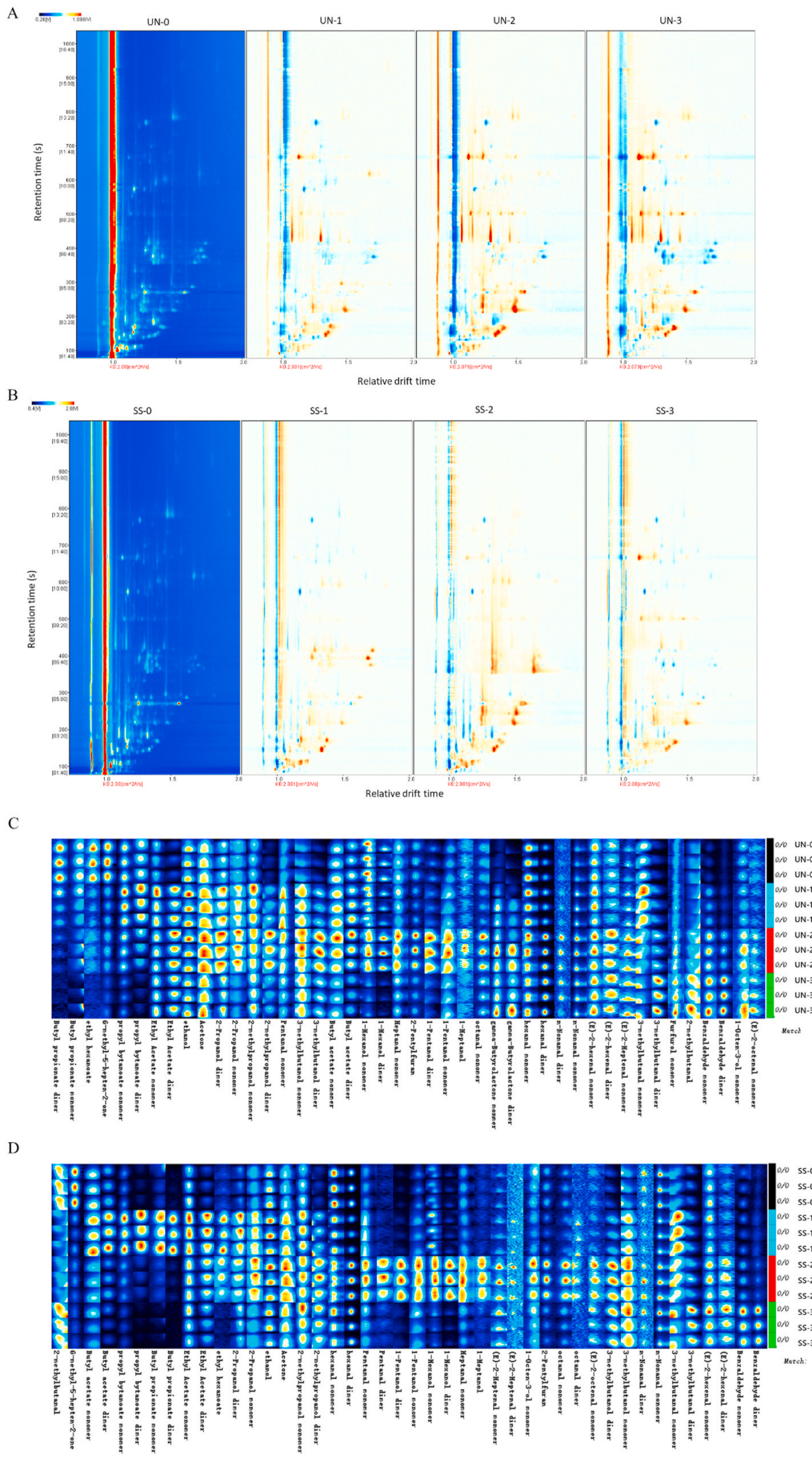

Fig. 2. 2D topographic (A & B) and gallery plots (C & D) of volatile compounds in untreated and SS-treated buckwheat flour during storage. UN: untreated buckwheat, SS: superheated steam treated buckwheat. 0–3: buckwheat stored for 0, 1, 2, 3 months.

Table 1
HS–GC–IMS integration parameters of volatile compounds of buckwheat.

No.	Compound	CAS	Formula	MW	RI	Rt (sec)	Dt (RIP relative)	Comment
1	ethanol	C64175	C2H6O	46.1	501.9	103.217	1.04844	
2	Acetone	C67641	C3H6O	58.1	512.9	108.296	1.11794	
3	2-Propanol	C67630	C3H8O	60.1	516.6	110.022	1.0922	monomer
4	2-Propanol	C67630	C3H8O	60.1	516.6	110.022	1.17551	dimer
5	Ethyl Acetate	C141786	C4H8O2	88.1	595.4	146.309	1.09977	monomer
6	Ethyl Acetate	C141786	C4H8O2	88.1	596.3	146.721	1.33997	dimer
7	2-methylpropanol	C78831	C4H10O	74.1	613.3	154.556	1.17226	monomer
8	2-methylpropanol	C78831	C4H10O	74.1	618.6	157.03	1.36378	dimer
9	3-methylbutanal	C590863	C5H10O	86.1	640.2	166.972	1.1804	monomer
10	3-methylbutanal	C590863	C5H10O	86.1	640.2	166.972	1.40842	dimer
11	2-methylbutanal	C96173	C5H10O	86.1	651.7	172.29	1.16083	
12	Pentanal	C110623	C5H10O	86.1	685.9	188.037	1.19402	monomer
13	Pentanal	C110623	C5H10O	86.1	686.3	188.242	1.42459	dimer
14	3-methylbutanol	C123513	C5H12O	88.1	725.1	221.374	1.2458	monomer
15	3-methylbutanol	C123513	C5H12O	88.1	724.6	220.911	1.48879	dimer
16	1-Pentanol	C71410	C5H12O	88.1	753	245.859	1.25146	monomer
17	1-Pentanol	C71410	C5H12O	88.1	752.4	245.333	1.50966	dimer
18	hexanal	C66251	C6H12O	100.2	784.1	273.177	1.25818	monomer
19	hexanal	C66251	C6H12O	100.2	782.4	271.684	1.56329	dimer
20	Butyl acetate	C123864	C6H12O2	116.2	799.9	291.104	1.23638	monomer
21	Butyl acetate	C123864	C6H12O2	116.2	797.7	288.614	1.62322	dimer
22	Furfurol	C98011	C5H4O2	96.1	816.1	309.524	1.08329	monomer
23	Furfurol	C98011	C5H4O2	96.1	814.8	308.071	1.33242	dimer
24	(E)-2-hexenal	C6728263	C6H10O	98.1	838.2	334.701	1.18234	monomer
25	(E)-2-hexenal	C6728263	C6H10O	98.1	836.5	332.764	1.51552	dimer
26	1-Hexanol	C111273	C6H14O	102.2	871.4	372.466	1.32492	monomer
27	1-Hexanol	C111273	C6H14O	102.2	865	365.204	1.64159	dimer
28	propyl bytanoate	C105668	C7H14O2	130.2	891.8	395.706	1.26639	monomer
29	propyl bytanoate	C105668	C7H14O2	130.2	889.7	393.285	1.68962	dimer
30	Heptanal	C111717	C7H14O	114.2	894.3	400.064	1.33693	monomer
31	Heptanal	C111717	C7H14O	114.2	895.6	402.485	1.69862	dimer
32	Butyl propionate	C590012	C7H14O2	130.2	902.7	415.557	1.2844	monomer
33	Butyl propionate	C590012	C7H14O2	130.2	902.7	415.557	1.72414	dimer
34	gamma-Butyrolactone	C96480	C4H6O2	86.1	912.2	432.987	1.08179	monomer
35	gamma-Butyrolactone	C96480	C4H6O2	86.1	909.8	428.63	1.30241	dimer
36	Benzaldehyde	C100527	C7H6O	106.1	950	502.51	1.1455	monomer
37	Benzaldehyde	C100527	C7H6O	106.1	949.2	501.041	1.47189	dimer
38	(E)-2-Heptenal	C18829555	C7H12O	112.2	951.1	504.47	1.2543	monomer
39	(E)-2-Heptenal	C18829555	C7H12O	112.2	949.7	502.02	1.66803	dimer
40	1-Heptanol	C111706	C7H16O	116.2	970	539.251	1.39681	
41	1-Octen-3-ol	C3391864	C8H16O	128.2	977.7	553.458	1.1547	monomer
42	1-Octen-3-ol	C3391864	C8H16O	128.2	977.7	553.458	1.59755	dimer
43	6-methyl-5-hepten-2-one	C110930	C8H14O	126.2	989.7	575.502	1.17615	
44	2-Pentylfuran	C3777693	C9H14O	138.2	988.9	574.032	1.25277	
45	octanal	C124130	C8H16O	128.2	1005.5	605.385	1.40294	monomer
46	octanal	C124130	C8H16O	128.2	1005	604.405	1.82893	dimer
47	ethyl hexanoate	C123660	C8H16O2	144.2	1001.9	598.526	1.34228	
48	n-Nonanal	C124196	C9H18O	142.2	1098.6	784.816	1.47492	monomer
49	n-Nonanal	C124196	C9H18O	142.2	1099.5	786.647	1.94607	dimer
50	(E)-2-octenal	C2548870	C8H14O	126.2	1055.4	701.501	1.33502	monomer
51	(E)-2-octenal	C2548870	C8H14O	126.2	1055.8	702.416	1.82708	dimer
52	(E)-2-nonenal	C18829566	C9H16O	140.2	1152.7	889.188	1.41703	
53	decanal	C112312	C10H20O	156.3	1212.1	1003.632	1.54246	

MW: Molecular weight, RI: the retention index calculated using n-ketones C4–C9 as external standard on FS-SE-54-CB capillary column, Rt: the retention time in the capillary GC column, Dt: the drift time in the drift tube.

collapse, which was demonstrated in 3.4. Storage also significantly increased the pasting temperature and breakdown of buckwheat ($P < 0.05$), but the SS-treated buckwheat showed the lower breakdown than untreated buckwheat at each storage stage ($P < 0.05$), which indicated that SS treatment kept starch stable during the continuous heating and shearing process.

3.4. Changes in farinograph characteristics of buckwheat mixture during storage

Our subsequent findings about farinograph characteristics explained the reductions in pasting viscosities and breakdown of buckwheat-wheat mixtures induced by storage and SS, respectively (Table 2). Before storage, the water absorption and DDT of buckwheat mixtures did not significantly ($P > 0.05$) change after SS treatment, while the DST increased and DS decreased significantly ($P < 0.05$). It supported that the dough made from SS-treated buckwheat was more resistant to the shear stress mentioned above. The enhanced dough strength might be closely associated with the starch gelatinization and retrogradation phenomena caused by SS, which contributed to a forceful starch network structure (Hu et al., 2017). SS treatment also caused the possible cross-linking of starch-lipid complexes or starch-protein networks, which might form network during dough formation (Guo, Wu, & Zhu, 2020). During storage, a decrease in water absorption and DDT of untreated buckwheat dough were observed, supporting the decrease in ability of the starch granules to absorb water, and explaining the reduced pasting viscosities mentioned above. The increase in DS and the decrease in DST of buckwheat dough during storage indicated the decreased dough strength and stability. However, these changes of SS-treated buckwheat induced by storage were lower than that of

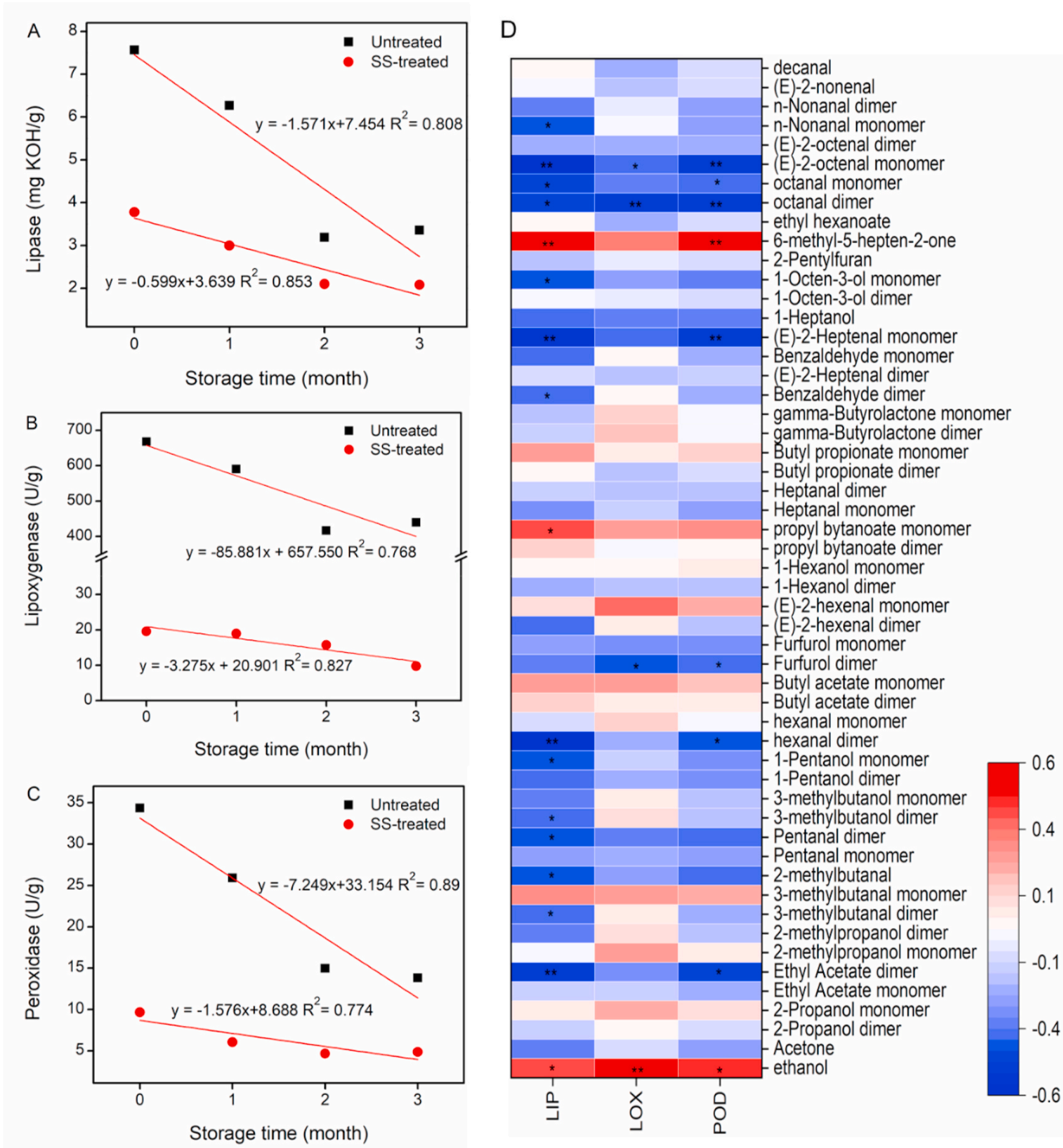

Fig. 3. Changes of lipid degrading enzymes activities in buckwheat during storage (A–C) and its relationship with volatile compounds (D). LIP: lipase, LOX: lipoxygenase, POD: peroxidase. Data with single asterisk (*) and double asterisk (**) are statistically significant at $P < 0.05$ and $P < 0.01$, respectively.

untreated one, suggesting that SS enhanced the elasticity and strength of the dough which was more resistant to continuous mixing shear and the increase of shear stress. Based on these results, it could be predicted that the noodles made from SS-treated buckwheat might have improved textural properties and higher qualities.

3.5. *Effects of SS on cooking qualities of noodles made from stored buckwheat*

Cooking quality is one of the most important factors to determine the qualities of noodles, and is evaluated by water absorption and cooking loss. As shown in Table 3, the water absorption of noodles made from fresh SS-treated buckwheat was significantly lower than that of noodles made from fresh untreated buckwheat (SS-0 vs. UN-0, $P < 0.05$). Compared with the noodles made from fresh buckwheat, the water absorption of noodles made from stored buckwheat significantly decreased (UN-3 vs. UN-0, SS-3 vs. SS-0, $P < 0.05$), and SS-treated buckwheat could slow down the decrease in water absorption of noodles. The water absorption of noodles was significantly ($P < 0.05$) positively correlated with the pasting viscosities (peak, trough and final viscosities) of buckwheat flour and the water absorption of dough (Fig. 4. A). That is also supported by the finding that the decreased water absorption of starch granules was retarded by SS treatment.

Cooking loss is dependent on the starch gelatinization and the network structure of starch-proteins (Hu et al., 2020). The cooking loss of noodles made from the fresh untreated buckwheat was significantly higher than that from fresh SS-treated buckwheat (UN-0 vs. SS-0, $P < 0.05$). A significant increase in the cooking loss of noodles made from the stored untreated buckwheat noodles (UN-3 vs. UN-0, $P < 0.05$), but not in the noodles made from stored SS-treated buckwheat (SS-3 vs. SS-0, $P > 0.05$). The less cooking loss of buckwheat noodles induced by SS treatment could be attributed to its improvement of starch stability based on RVA results, and its promotion of the elasticity and strength of dough based on farinograph results (Table 2). That was the reason why cooking loss showed significantly ($P < 0.05$) positive correlations with the breakdown and DS, while had significantly ($P < 0.05$) negative correlations with DDT and DST (Fig. 4. A).

Table 2
Pasting properties and farinograph characteristics of untreated and SS-treated buckwheat flour mixture during storage.

	Pasting properties						Farinograph characteristics			
	Pasting Temperature (°C)	Peak Viscosity (cP)	Trough Viscosity (cP)	Breakdown (cP)	Final Viscosity (cP)	Setback (cP)	Water absorption (%)	DDT (min)	DST (min)	DS (BU)
UN-0	68.2 ± 0.6bc	2712 ± 5a	1987 ± 14a	725 ± 19b	3399 ± 13a	1412 ± 1a	59.0 ± 0.1a	3.5 ± 0.1a	4.7 ± 0.3a	103 ± 4.2de
UN-1	68.6 ± 0.1 ab	2639 ± 28b	1965 ± 116a	674 ± 16c	3356 ± 45a	1391 ± 34a	56.8 ± 0.5b	4.0 ± 0.1 ab	4.2 ± 0.1abc	116 ± 1.4bc
UN-2	68.3 ± 0.6bc	2518 ± 14c	1803 ± 9c	716 ± 5b	3044 ± 19c	1241 ± 10c	56.3 ± 0.1b	3.5 ± 0.6 ab	3.7 ± 0.4c	122 ± 1.4b
UN-3	69.4 ± 0.1a	2417 ± 13d	1616 ± 5f	802 ± 8a	2847 ± 6d	1232 ± 1c	56.4 ± 0.3b	3.0 ± 0.1b	2.9 ± 0.1d	132 ± 7.0a
SS-0	67.8 ± 0.2c	2652 ± 26b	1989 ± 20a	663 ± 6c	3413 ± 62a	1424 ± 42a	58.5 ± 0.1a	3.5 ± 0.3 ab	4.8 ± 0.3a	99 ± 2.8e
SS-1	68.6 ± 0.1 ab	2528 ± 35c	1899 ± 32b	630 ± 4d	3196 ± 59b	1298 ± 28b	57.2 ± 0.3b	3.4 ± 0.4 ab	4.6 ± 0.3a	104 ± 1.4de
SS-2	68.7 ± 0.1 ab	2430 ± 2d	1759 ± 1d	671 ± 3c	2984 ± 17c	1225 ± 18c	56.9 ± 0.9b	3.3 ± 0.1 ab	4.3 ± 0.1 ab	114 ± 4.2bc
SS-3	69.4 ± 0.5a	2379 ± 23d	1658 ± 4e	722 ± 19b	2888 ± 29d	1230 ± 25c	56.3 ± 0.2b	3.3 ± 0.1 ab	3.9 ± 0.1bc	111 ± 2.8cd

All data represent the mean of triplicate determinations ± standard deviation. Means with different lowercase letter in the same column are significantly different ($P < 0.05$). UN: untreated buckwheat. SS: superheated steam treated buckwheat. 0–3: buckwheat stored for 0, 1, 2, 3 months. DDT: dough development time, DST: dough stability time, DS: degree of softening.

Table 3
Cooking properties and texture profiles of noodles made from different buckwheat samples.

	UN-0	SS-0	UN-3	SS-3
Cooking properties				
Water absorption (%)	170.23 ± 1.28a	161.45 ± 3.73b	150.02 ± 2.67c	154.46 ± 1.42c
Cooking loss (%)	8.49 ± 0.03b	8.22 ± 0.06c	9.20 ± 0.10a	7.99 ± 0.11c
Texture profiles				
Tension force (g)	13.70 ± 0.46a	13.77 ± 0.38a	12.04 ± 0.85b	13.23 ± 1.18a
Tension distance (mm)	35.15 ± 1.97a	32.38 ± 1.32 ab	26.17 ± 2.87c	29.42 ± 2.93b
Hardness (g)	3474 ± 57b	3694 ± 27a	3165 ± 52c	3630 ± 49a
Springiness (%)	91.18 ± 1.80a	89.93 ± 0.78a	87.96 ± 2.01b	91.04 ± 1.28a
Chewiness	2562 ± 153b	2610 ± 105 ab	2410 ± 108c	2733 ± 106a
Adhesiveness (g.s)	70.49 ± 7.32b	79.55 ± 6.94a	60.48 ± 6.14c	68.91 ± 6.44b

All data represent the mean ± standard deviation (n = 3 for cooking properties, n = 6 for texture profiles). Means with different lowercase letter in the same row are significantly different ($P < 0.05$). UN: untreated buckwheat. SS: superheated steam treated buckwheat. 0 & 3: buckwheat stored for 0 & 3 months.

3.6. Effect of SS on texture profiles of noodles made from stored buckwheat

Textural properties are the paramount concern to consumers, because textural attributes are also one of the most important properties of noodles (Guo et al., 2017). The noodles made from fresh SS-treated buckwheat displayed significantly (P˂0.05) higher hardness, chewiness and adhesiveness than that from untreated buckwheat, while the tension force, tension distance and springiness of SS-treated ones were not significantly different ($P > 0.05$) from the untreated ones (Table 3). All the texture properties including tension force, tension distance hardness, springiness, chewiness, adhesiveness of noodles made from stored untreated buckwheat significantly decreased (UN-3 vs. UN-0, $P < 0.05$), while SS treatment significantly ($P < 0.05$) retarded these texture changes of noodles. The tension force and tension distance showed significantly positive correlations with the DDT and DST, while was negatively correlated with DS of buckwheat dough, which indicated that the enhancement of elasticity and strength of the dough caused by SS treatment had a great potential for producing high quality noodles. Hardness of noodles was related to the gelatinization of starch and mixing stability of the dough (Guo et al., 2020). That was the reason why hardness of noodles showed significantly ($P < 0.05$) negative correlations with breakdown and DS observed in this study (Fig. 4. A).

3.7. PCA analysis

PCA was applied to analyze storage stability of buckwheat and investigate the potential relationships between all variables observed in this study. The cumulative variance contribution rate of the first part PC1 (63.5%) and the second part PC2 (18.9%) was 82.4% (Fig. 4. B). All buckwheat samples were well distinguished in the distribution map, indicating that the qualities of buckwheat changed significantly after SS treatment and/or storage. The loading plot clearly showed the changes in untreated and SS treated buckwheat qualities during storage were related to many factors (Fig. 4. C). On the positive sides of PC1 and PC2, UN-0 was characterized with higher lipid degrading enzymes, pasting viscosities, setback, and dough water absorption. On the positive side of PC1 and the negative side of PC2, SS-0 was separated from the other samples by their higher hardness, chewiness, tension force, DDT and DST. In terms of negative PC1 and positive PC2, UN-3 was predominantly affected by cooking loss, breakdown, DS, aldehydes, and total volatile compounds content. Relatively close to SS-0 distribution, SS-3 was less influenced by cooking loss, breakdown, DS, and total volatile compounds content. These results indicated that the quality deterioration of noodles was attributed to the increased off-flavor volatile compounds of buckwheat during storage and the decreased texture profiles of noodles. However, SS could inactivate lipid degrading enzyme, inhibit the lipid oxidation and generation of off-flavor volatile compounds in buckwheat grains, facilitate the formation of starch network structure, together with the improvement of the flavor and firmness of noodles, and thus retard the decrease of overall acceptability of noodles.

4. Conclusion

In this study, the effects of SS stabilized buckwheat grains on the qualities of noodles made from stored buckwheat were investigated. The volatile compounds of aldehydes continuously increased during buckwheat storage, in which hexanal and 3-methyl-butyraldehyde could be

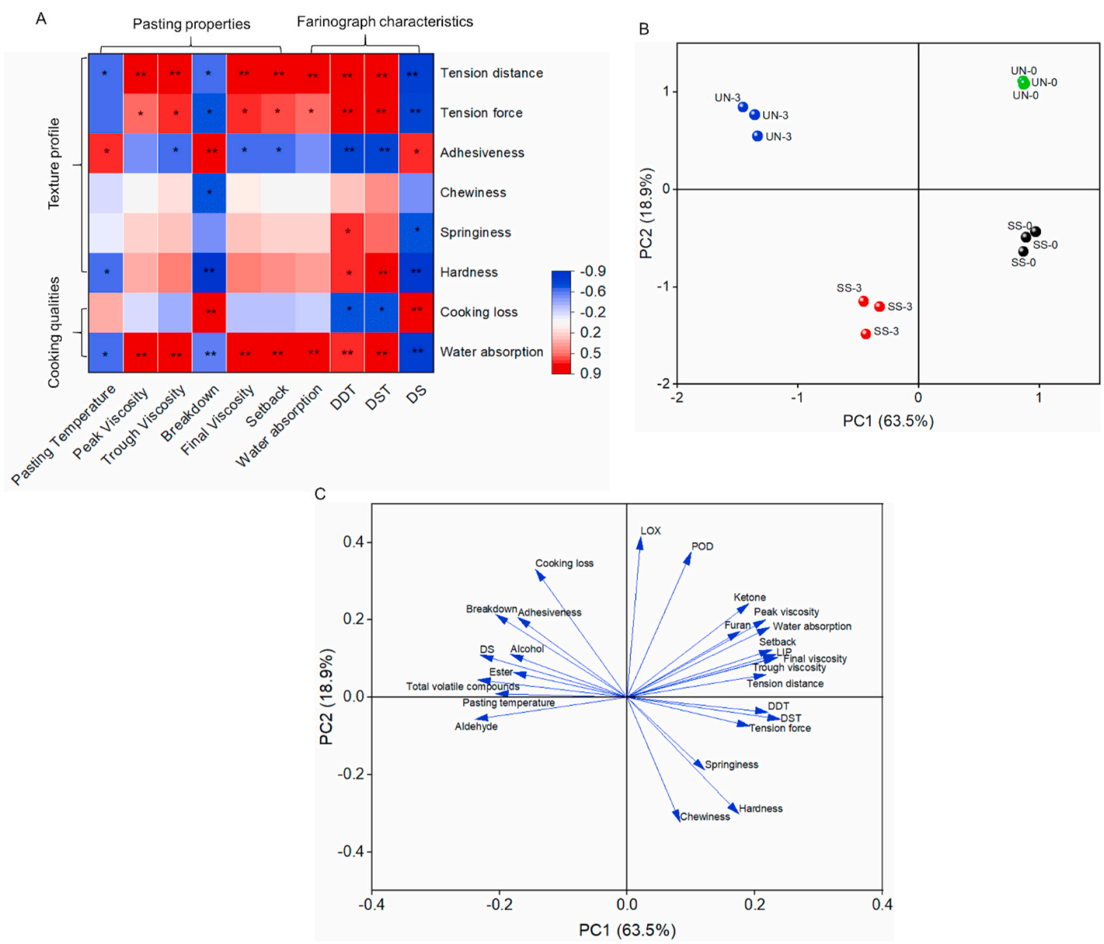

Fig. 4. Pearson's correlation and PCA analyses. Correlation between the pasting properties and farinograph characteristics of buckwheat mixture and texture profiles and cooking qualities of noodles (A); Score plot of the different buckwheat samples (B); Loading plot of the selected variables of buckwheat (C). LIP: lipase, LOX: lipoxygenase, POD: peroxidase, DDT: dough development time, DST: dough stability time, DS: degree of softening. UN: untreated buckwheat, SS: superheated steam treated buckwheat. 0 & 3: buckwheat stored for 0 & 3 months. Data with single asterisk (*) and double asterisk (**) are statistically significant at $P < 0.05$ and $P < 0.01$, respectively.

used as markers of lipid oxidation and flavor change. LIP and POX were important to promote the generation of volatile compounds in buckwheat grains during storage. The decrease in pasting and farinograph characteristics of buckwheat dough and the deterioration of cooking and texture characteristics of buckwheat noodles were observed. SS changed the generation of off-flavor volatile compounds of buckwheat grains during storage by inactivating the lipid degrading enzyme, retarded the texture profiles of buckwheat noodles and suppressed the cooking loss by keeping the pasting properties of starch stable and enhancing the dough strength, thus suppressed the quality deterioration of buckwheat noodles. Therefore, SS processing might be a novel and efficient method to improve the qualities of buckwheat noodles by stabilization of buckwheat grains quality during storage.

CRediT authorship contribution statement

Lijuan Wang: Conceptualization, Methodology, Software, Investigation, Writing - original draft. **Libo Wang:** Validation, Formal analysis, Visualization, Software. **Aili Wang:** Validation, Writing - review & editing. **Ju Qiu:** Resources, Writing - review & editing, Supervision, Project administration. **Zaigui Li:** Resources, Writing - review & editing, Supervision, Project administration, Funding acquisition.

Declaration of competing interest

The authors declare no conflicts of interest.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.lwt.2020.110746.

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