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Quality deterioration of mashed potatoes during the freeze-thaw cycle: From the perspective of moisture and microstructure

Yingying Yu [a], Wuyin Weng [a], Zhongyang Ren [a], Yucang Zhang [a], Ping Li [b], Linfan Shi [a,*]

[a] College of Ocean Food and Biological Engineering, Jimei University, Xiamen 361021, China

[b] Sericultural & Agri-Food Research Institute Guangdong Academy of Agricultural Sciences, Key Laboratory of Functional Foods, Ministry of Agriculture and Rural Affairs, Guangdong Key Laboratory of Agricultural Products Processing, Guangzhou 510610, China

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ABSTRACT

This study aimed to simulate cold chain sales temperatures to predict the effects of temperature fluctuations on the physicochemical properties, moisture distribution, microstructure, and flavor of mashed potatoes. The results showed a decline in the hardness and chewability of mashed potatoes alongside the migration of water from bound water states to weakly bound states under freeze-thaw cycles (FTC) conditions. Microstructural analysis indicated that the adhesive forces between proteins and starch granules were weakened, and the structure of mashed potatoes particles was destroyed following FTC. The oxidation and degradation of fat induced by FTC increased the content of key compounds such as octanal and nonanal, thereby contributing to an overall deterioration in the flavor of mashed potatoes. This study elucidates the effects of FTC on water migration, microstructure, and flavor characteristics of mashed potatoes, thereby providing a theoretical foundation for improving the quality of prefabricated frozen mashed potatoes dishes.

1. Introduction

The potato (*Solanum tuberosum L.*) is not only the world's fourth-largest food crop, following rice, maize, and wheat, but also an economically significant crop for both vegetable and industrial feedstock (Saidi & Hajibarat, 2021). Potatoes are rich in essential nutrients such as ascorbic acid, vitamins, dietary fiber, and amino acids, and they possess protective properties against cancer, obesity, diabetes, and hypertension (Mishra et al., 2020; Raigond et al., 2023). China boasts the largest potato planting area and yield globally. However, the damage, decay, shrinkage, and germination of potatoes during harvesting, storage, and transportation contribute to post-harvest losses of up to 40 % (Liu et al., 2023). This leads to a significant waste of high-quality potato resources, failing to meet consumer demands. Therefore, increasing the potato processing rate holds great significance for improving the utilization rate of potato resources. As the pace of life accelerates and cold chain infrastructure develops, convenient frozen foods have gained favor among consumers. Consequently, the development of frozen mashed potatoes products exhibits considerable economic value.

Frozen mashed potatoes exhibited good texture and flavor characteristics, achieved through a process involving cleaning, peeling, slicing, steaming, mashing, freezing. The preservation of mashed potatoes primarily relies on frozen storage, which inhibits microbial growth and extends the shelf life. However, limitations persist within the cold chain technology. Throughout the frozen storage duration, repeated freeze-thaw cycles (FTC) occur owing to temperature fluctuations, accelerating water loss and affecting taste and nutritional value. Repeated FTC predominantly affects the water distribution within food, inducing sublimation and recrystallization of ice crystals. Researchers reported that repeated FTCs fracture of hydrogen bonds and damage gluten networks, causing bound water in the dough to migrate to free water (He et al., 2024; Tao et al., 2024). These processes lead to irreversible damage to cells, including the destruction of tissue structure, starch retrogradation, and protein denaturation. Therefore, the moisture overflow and quality of FTC mashed potatoes remain a concern among consumers.

The FTC is a key factor in the assessment of the quality of frozen mashed potatoes, and the increased frequency of FTC fluctuations

Abbreviations: FTC, freeze thaw cycles; LF-NMR, low field nuclear magnetic resonance; SEM, scanning electron microscopy; CLSM, laser scanning confocal microscope; GC-IMS, gas chromatography-ion migration spectroscopy; WHC, water holding capacity; FITC, Fluorescein Isothiocyanate; VOCs, volatile organic compounds; ROAV, relative odor activity value.

* Corresponding author.
E-mail address: linfanshi@jmu.edu.cn (L. Shi).

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exacerbates recrystallization, ultimately leading to the texture deterioration of mashed potatoes (Abedi et al., 2022). During the freezing process, ice crystals form within the mashed potatoes, gradually increasing in volume. This phenomenon results in cell wall separation, reduced water retention, and an increased starch dissolution rate, thereby impacting the quality, stability and structural integrity of mashed potatoes products (Qian et al., 2022). The decline in viscosity and consistency of mashed potatoes post-FTC is associated with structural degradation. Water retention in mashed potatoes decreases following FTC, with structural damage reducing viscosity and elasticity (Alvarez, Fernández, Solas, & Canet, 2011). Ice crystal formation induced by FTC causes surface bulging and folding of potato starch particles, leading to increased pores on the surface of potato starch particles and subsequent deterioration in both texture and flavor of mashed potatoes (Wang et al., 2022). Moreover, the impact of mashed potatoes retrogradation on texture primarily arises from amylose crystallization and cell wall destruction during freezing (Lamberti, Geiselmann, Conde-Petit, & Escher, 2004).

The novelty of this study lies in its focus on the FTC to analyze the effects of temperature fluctuations on the physicochemical properties, moisture distribution, microstructure, and flavor of mashed potatoes in the context of the cold chain sales model. In this study, the texture characteristics, water distribution, microstructure, and flavor of frozen mashed potatoes were analyzed using a chromometer, texture analyzer, rheometer, low-field nuclear magnetic resonance (LF-NMR), scanning electron microscopy (SEM), laser scanning confocal microscopy (CLSM), and gas chromatograph-ion migration spectrometry (GC-IMS). This study aimed to investigate the correlation between FTC and the quality characteristics of mashed potatoes during storage. Our findings establish the theoretical foundations underpinning the storage and quality control of mashed potatoes, thereby fostering advancements in the cold chain preservation of frozen convenience food.

2. Materials and methods

2.1. Materials

Potatoes of uniform size with smooth surfaces and shallow and minimal buds (*Holland No. 15*, Tengzhou, Shandong, 78.54 % moisture, 16.65 % starch, 2.09 % protein, 0.14 % fat, 0.55 % reducing sugar and 1.29 % ash) were selected as the experimental material. Rhodamine B and fluorescein isothiocyanate (FITC) were purchased from Aladdin Reagent Co., Ltd. (Shanghai, China). NaCl and Na_2SO_3 were provided by Xilong Scientific Co., Ltd. (Guangdong, China), while KCl was purchased from Sinopharm Chemical Reagent Co., Ltd. (Suzhou, China).

2.2. Preparation of mashed potatoes

The potato tubers were washed, peeled, and cut, followed by steaming at 100 °C for 30 min before being mashed using an extruder. Subsequently, the prepared samples were placed in Ziplock bags and stored in a refrigerator set to −18 °C for 24 h. They were then transferred to a freezer maintained at 4 °C for 12 h. The samples were alternately stored at −18 °C and 4 °C for a FTC, with zero, one, two, three, four, and five FTC conducted, respectively.

2.3. Color determination

The color of mashed potatoes subjected to varying FTC treatments was assessed using a color difference meter. Calibration was performed using standard black and white plates, while mashed potatoes were uniformly distributed in the test dish for measurement. The color of the mashed potatoes was characterized using the LAB (L^* a^* b^* parameters) color system, as recommended by the International Commission on Illumination (CIE). Standard whiteboards and blackboards were used as benchmarks, with the positive and negative values of L^*, a^*, and b^* denoting white/black, red/green, and yellow/blue, respectively. All measurements were conducted in triplicate and averaged.

2.4. Texture properties

According to the methodology proposed by(Li et al., 2022), the texture characteristics of mashed potatoes were investigated using a texture analyzer (TA-XT Plus, Stable Micro Systems, Godalming, UK). The measurement parameters were configured as follows: employing the P/36R probe, the pre-test and test speeds were set at 2.00 mm/s, with a test distance of 35 mm, an auto-trigger force of 5 g, and a data acquisition rate of 200 pps. The mashed potatoes were compressed twice using the texture analyzer, and the compression at strain of 30 % each time.

2.5. Water holding capacity (WHC)

Mashed potatoes (200 g) was placed in a 500 mL-centrifuge tube and subsequently centrifuged at 4 °C and 9986 ×*g* for 30 min. Following the removal of the supernatant, the mass of the centrifuge tube and the sediment was measured. Water retention was calculated using Eq. (1).

$$WHC = (m_2 - m_0)/(m_1 - m_0) \times 100 \tag{1}$$

In Eq. (1), m_0 represents the mass of the empty centrifuge tube (g); m_1 indicates the mass of the centrifuge tube and sample (g); and m_2 denotes the mass of the centrifuge tube and sediment (g).

2.6. Solubility of potato starch and protein

2.6.1. Determination of soluble starch

Potato starch was extracted according to the method described by Wang et al., with minor modifications (Wang et al., 2023). Mashed potatoes (90–100 g) were washed employing a 100-mesh sieve and distilled water. Subsequently, excessive 1 % (*w/v*) NaCl solution and a 0.2 % (w/v) sodium sulfite solution were added to the precipitate, which was then allowed to stand for 3 h at 4 °C. After the removal of the supernatant, the precipitate was washed three times with distilled water until a neutral supernatant was obtained. The resulting wet starch was dried in an oven at 50 °C, subsequently crushed, sieved through an 80-mesh sieve, and stored for subsequent experiments. Starch weighing 2.80 g was combined with 140 mL of deionized water and incubated in a water bath at 50 °C for 30 min. Subsequently, centrifugation was performed at 4 °C and 9986 ×g for 15 min. The supernatant and precipitate were collected separately and dried to a constant weight in a drying oven set at 105 °C. Solubility and swelling power were calculated using Eqs. (2) and (3), respectively.

$$S = m_s/m \times 100 \tag{2}$$

$$C = m_c/[m \times (100 - S)] \times 100 \tag{3}$$

In Eqs. (2) and (3), S denotes solubility (%); C represents swelling power (g/g); m_s indicates the mass of dried supernatant after centrifugation (g); m_c indicates the mass of dried precipitate after centrifugation (g); and m denotes the mass of dry potato powder (g).

2.6.2. Determination of soluble protein

In this experiment, soluble protein was extracted from 10.00 g of mashed potatoes. The mashed potatoes were homogenized using a 0.6 mol/L KCl aqueous solution for 2 min, followed by stirring in a water bath for 1 h and centrifugation at 4 °C and 9986 ×*g* for 15 min. The protein concentration in the supernatant was determined using the Lowry method employing an LS55 fluorescence spectrophotometer. The 25 μL of extracted protein solution was measured, utilizing a spectrophotometer set at a wavelength of 750 nm.

2.7. Rheological properties

Rheological properties were assessed using the method outlined by Zhang et al., with minor modifications (Zhang et al., 2023). A plate-to-plate sensor system with a 35-mm diameter was used for dynamic rheometer measurements. Following calibration to 25 °C, the gap between the bottom plate and the parallel plate was set at 2 mm. The mashed potatoes were balanced between the bottom and parallel plates for 1 min, after which excess mashed potatoes was removed. To mitigate water loss from the sample, we applied methylsilicone oil around the flat cylinder, and the sample was capped before rheological analysis commenced. To determine the linear viscoelastic region, we performed an initial stress scan at a constant frequency (ω) of 1 rad s^{-1} within a shear stress range of 1–100 Pa. Conditions for frequency scanning were as follows: stress was maintained at 0.1 %, temperature was set to 25 °C, and frequency varied between 0.1 and 20 Hz. Rheological properties were analyzed based on the elastic modulus (G') and viscous modulus (G").

2.8. LF-NMR

The transverse relaxation time (T_2) of mashed potatoes was assessed at 24 MHz utilizing a nuclear magnetic resonance analyzer and imaging system (HT-MRSI20-60 A, Shanghai Huantong Science and Education Equipment Co., Ltd., Shanghai, China). The mashed potatoes were tightly packed in Ziplock bags to mitigate moisture loss before placement in magnet chambers. The transverse relaxation time (T_2) was derived employing the Carr-Purcell-Meiboom-Gill (CPMG) pulse train method. The pulse durations for 90° (P_1) and 180° (P_2) were 89 and 196 μs, respectively. The echo interval was 3.0 ms, with 2048 echoes acquired, a repetition time of 2.0 TR/S, and a cumulative number of 20. The relaxation curves from LF-NMR were subjected to fitting using the Huantong nuclear magnetic T_2 spectrum acquisition and inversion software. The recorded parameters encompassed the relaxation times T_{21}, T_{22}, and T_{23}.

2.9. SEM

The microstructure of mashed potatoes was examined according to the method outlined by Alvarez et al. (Alvarez, Fernández, & Canet, 2011). Mashed potatoes subjected to varying FTC conditions were freeze-dried, crushed, sifted, and affixed to the sample table using double-sided tape. Following chemical fixation with osmium tetroxide, and observations were made after ion sputtering and gold spraying.

2.10. CLSM

The distribution of starch and protein in mashed potatoes was examined utilizing TCSSP8 CLSM (Leica, Germany). Initially, 5 g of mashed potatoes was supplemented with 10 μL of a mixed dye solution comprising 1.0 g/L Rhodamine B and 1.0 g/L FITC for staining purposes. Subsequently, the mixture was homogenized for 5 min after dyeing, and the sample was stored in a beaker in the dark. A stained aliquot (2 μL) was placed onto a microscope slide and examined under an inverted microscope (10 × magnification). A confocal laser scanning microscope was employed in fluorescence mode to visualize the starch and protein, stained respectively with FITC and Rhodamine B, utilizing excitation wavelengths of 488 and 543 nm, respectively. CLSM images were subsequently acquired (Lu et al., 2022).

2.11. GC-IMS

The volatile organic compounds (VOCs) present in mashed potatoes were analyzed via GC-IMS. After thawing, the 10.00 g of mashed potatoes were weighed, placed in a 20mL headspace bottle, and then incubated in an oven at 80 °C for 30 min. Subsequently, the volatile components were separated employing a GC-IMS protocol at 45 °C, with the injection pulse width set to 100 μs and the duration at 1680 s. High-purity nitrogen (≥99.999 % purity) served as the carrier gas, while the oven program followed the following sequence: initially, the column temperature was maintained at 40 °C for 5 min; thereafter, it was ramped up to 120 °C at a rate of 10 °C/min. Subsequently the temperature was increased to 210 °C at increments of 20 °C/min, followed by a further increase to 230 °C at a rate of 2 °C/min. Each sample was tested in triplicate, and the retention indices (RI) along with drift time were compared against standard compounds to identify VOCs (Xi et al., 2024).

2.12. Relative odor activity value (ROAV)

The contribution of an individual volatile compound to the overall flavor is contingent upon both its concentration and odor threshold, with this contribution being determined by its ROAV (Dai et al., 2024). The volatile compounds that contributed the most to the overall flavor of the sample was defined as 100, and the ROAV of the remaining volatile compounds were calculated using Eq. (4).

$$ROAV \approx C_i/C_{st} \times T_{st}/T_i \times 100 \tag{4}$$

In Eq. (4), C_i represents the relative percentage (%) of the concentration of the flavor compound being analyzed; C_{st} denotes the relative percentage (%) of the concentration of volatile compound that contribute the most to the overall flavor; T_i denotes the sensory threshold of the flavor compound being analyzed (μg/kg); and T_{st} indicates the sensory threshold of volatile compound that contribute the most to the overall flavor (μg/kg).

2.13. Statistical analysis

The data were recorded as mean ± standard deviation and analyzed using SPSS software (SPSS, Inc., Chicago, IL, USA). One-way analysis of variance (ANOVA) and the Duncan test were employed to examine the difference between the mean values, with $p < 0.05$ indicating statistical significance.

3. Results and discussion

3.1. Effect of FTC on the color of mashed potatoes

The appearance of mashed potatoes is directly influenced by their color, which is a crucial factor affecting consumer preference. The impact of the number of FTC on the color of mashed potatoes is shown in Fig. S1 and Table 1. Compared with mashed potatoes samples not subjected to FTC, the brightness and yellowness values of mashed potatoes increased significantly with increasing number of FTC ($p < 0.05$). The L^* value of mashed potatoes increased by 16 % after five FTC. This increase in brightness is often attributed to juice loss caused by cell rupture and the melting of ice crystals. After five FTC, a substantial amount of free water appeared on the surface of mashed potatoes, intensifying light reflection (Yang et al., 2024). Previous studies have reported significant increases in the L^* value of *Pseudosciaena crocea*, beef, and egg yolk due to increased free water during FTC (Chen et al., 2023). The b^* value (yellowness) of mashed potatoes decreased significantly with increasing number of FTC, while the a^* value (redness) initially increased and then decreased. This is due to the non-enzymatic browning of the mashed potatoes caused by the increased temperature during the thawing process. The browning reaction was aggravated by the increased of thawing times, resulting in a decrease in the b^* value and an increase in the a^* value. Jha's study showed that the reduction in yellowness value and the increase in redness value of potatoes were affected by FTCs, attributed to browning reactions during the thawing process (Jha et al., 2019). Consequently, mashed potatoes are affected by FTC, exhibiting a yellowish and white appearance, which significantly impacts their

Table 1
Color and texture characteristics of mashed potato during freeze-thaw cycle.

Freeze-thaw cycles (times)	L^*	a^*	b^*	Hardness (N)	Adhesiveness (N)	Springiness (mm)	Cohesiveness	Gumminess (N)	Chewiness (mj)	Resilience
0	71.81 ± 1.83^{e}	−4.51 ± 0.41^{c}	32.22 ± 0.70^{a}	367.31 ± 11.99^{a}	−3.12 ± 0.78^{a}	0.95 ± 0.05^{a}	0.78 ± 0.01^{a}	293.90 ± 15.80^{a}	264.10 ± 17.11^{a}	0.42 ± 0.04^{a}
1	74.14 ± 0.97^{d}	−4.48 ± 0.31^{c}	30.12 ± 1.04^{a}	324.34 ± 10.10^{b}	−3.45 ± 0.81^{a}	0.92 ± 0.07^{a}	0.79 ± 0.01^{a}	287.23 ± 44.01^{a}	260.73 ± 21.18^{a}	0.42 ± 0.02^{a}
2	74.53 ± 1.02^{cd}	−3.84 ± 0.25^{ab}	29.52 ± 2.03^{a}	305.71 ± 16.13^{bc}	−4.41 ± 2.46^{a}	0.89 ± 0.09^{a}	0.77 ± 0.04^{a}	210.86 ± 6.32^{b}	227.97 ± 10.42^{b}	0.42 ± 0.02^{a}
3	76.53 ± 1.20^{bc}	−3.93 ± 0.35^{ab}	26.31 ± 2.61^{b}	239.56 ± 58.99^{cd}	−7.39 ± 1.54^{b}	0.85 ± 0.07^{a}	0.72 ± 0.02^{a}	171.73 ± 11.93^{c}	172.98 ± 16.16^{c}	0.36 ± 0.02^{b}
4	77.96 ± 1.15^{ab}	−3.67 ± 0.21^{a}	23.18 ± 0.80^{c}	188.27 ± 22.19^{de}	−8.14 ± 0.53^{b}	0.86 ± 0.12^{a}	0.77 ± 0.08^{a}	134.87 ± 19.64^{cd}	115.02 ± 26.04^{cd}	0.40 ± 0.04^{a}
5	79.29 ± 0.87^{a}	−4.34 ± 0.04^{bc}	19.86 ± 1.56^{d}	144.82 ± 18.17^{e}	−8.69 ± 0.98^{b}	0.88 ± 0.05^{a}	0.76 ± 0.03^{a}	110.36 ± 11.23^{d}	104.81 ± 11.83^{d}	0.35 ± 0.02^{b}

Different letters within the same group indicate significant differences ($p < 0.05$).

aesthetic appeal and consumer preference.

3.2. Effect of FTC on the texture characteristics of mashed potatoes

In the TPA (Texture profile analysis) test, the sample was compressed twice using a texture analyzer to simulate oral movements, and the sensory quality of mashed potatoes was evaluated using texture curves and parameters. The impact of FTC on the texture of mashed potatoes is shown in Table 1. The hardness, adhesiveness, gumminess, and chewiness of mashed potatoes were significantly decreased with increasing number of FTC ($p < 0.05$), whereas the alterations in springiness, cohesiveness, and resilience were not significant ($p > 0.05$).

The hardness of mashed potatoes were affected by starch interaction and swelling behavior, which were closely related to the pasting characteristics and swelling power. Compared with untreated mashed potatoes, those subjected to five FTC exhibited a decrease in hardness from 367.31 to 144.82 N and in viscosity from −3.12 to −8.69 N, respectively. This may be attributed to the mechanical damage caused by ice crystals to starch granules during FTC. Due to the fact that the amorphous region and crystal structure of potato starch were damaged seriously by ice crystals, more channels though which water permeates were formed inside the starch granule and internal ice crystals grew. The expansion of internal ice crystals promoted the amorphization of ordered molecular structure, leading to the breakage of the starch chain and the leaching of amylose and amylopectin in the amorphous region. This caused a decrease in the substances that participate in the formation of gel network after starch gelatinization and a reduction in the crosslinking degree between starch molecules, which in turn resulted in the lower hardness of starch gels. The stickiness and chewability of mashed potatoes decreased from 293.90 to 110.36 N and from 264.10 to 104.81 mj, respectively, which could be attributed to reductions in starch content and weakening of protein-starch molecular interactions. Olivera et al. similarly observed decreased hardness and chewability in frozen noodles compared with fresh noodles, validating structural impairment resulting from freezing (Olivera & Salvadori, 2009).

3.3. WHC

The impact of FTC on the WHC of mashed potatoes is shown in Table 2. WHC, defined as the capacity of mashed potatoes to bind water molecules, is indicative of the maximum amount of water that mashed potatoes can absorb and retain (Liu et al., 2019). Mashed potatoes subjected to five FTC exhibited a significant reduction in WHC compared to those subjected to zero FTC ($p < 0.05$). This can be attributed to the reduced water-binding capacity of mashed potatoes resulting from FTC, thereby leading to a decrease in WHC. The easy separation of water from mashed potatoes was attributed to the reduction in WHC. The melting and freezing of ice crystals caused water to move from the inside of the cell to the outside during FTC, and the cell's ability to reabsorb water back into the cell was impaired by repeated FTC. The binding ability of starch and water molecules in mashed potatoes was related mainly to the number of hydroxyl groups in starch. The molecular structure of starch was destroyed by the ice crystals produced during the freeze-thaw cycle, which caused the pores inside the starch to enlarge. The hydroxyl groups' ability to bind to water molecules was reduced, resulting in decreased WHC. Schmitz et al. demonstrated that the WHC of potato decreases due to the collapse of the fiber structure during the refrigeration process (Schmitz et al., 2021).

3.4. Solubility of potato starch and protein

The hydration capacity of starch manifests through its solubility and swelling power, with the solubility of starch being closely linked to the extent of damage incurred by starch granules (Liu et al., 2022). As

Table 2
Changes of T_{22} time, width, peak area, water retention and solubility characteristics of mashed potato in freeze-thaw cycle.

Freeze-thaw cycles (times)	T_{22} time (ms)	T_{22} width	T_{22} peak area	Specific retention (%)	Starch solubility (%)	Starch swelling force (%)	Protein solubility (%)
0	65.22 ± 3.20^{bc}	191.24 ± 1.01^{ab}	363,223.29 ± 27.48^{f}	93.72 ± 0.07^{a}	0.29 ± 0.04^{bc}	3.97 ± 0.29^{a}	0.18 ± 0.70^{c}
1	80.32 ± 3.94^{bc}	198.43 ± 16.34^{ab}	525,173.89 ± 20.87^{e}	93.15 ± 0.06^{a}	0.26 ± 0.08^{c}	3.73 ± 0.22^{ab}	0.19 ± 1.40^{bc}
2	63.56 ± 12.40^{c}	129.34 ± 49.98^{b}	525,283.73 ± 21.70^{d}	83.41 ± 0.06^{ab}	0.36 ± 0.00^{bc}	3.45 ± 0.18^{b}	0.20 ± 0.70^{bc}
3	69.91 ± 3.43^{bc}	150.52 ± 17.15^{ab}	657,249.83 ± 49.68^{c}	82.27 ± 0.05^{ab}	0.39 ± 0.04^{b}	3.38 ± 0.22^{bc}	0.20 ± 0.70^{bc}
4	86.51 ± 12.69^{ab}	172.06 ± 86.20^{ab}	1,129,585.14 ± 14.51^{b}	77.31 ± 0.01^{b}	0.64 ± 0.01^{a}	2.93 ± 0.08^{cd}	0.21 ± 1.41^{ab}
5	102.59 ± 10.06^{a}	242.04 ± 10.93^{a}	1,182,037.46 ± 24.90^{a}	75.94 ± 0.01^{b}	0.73 ± 0.05^{a}	2.83 ± 0.06^{d}	0.23 ± 0.70^{a}

Different letters within the same group indicate significant differences ($p < 0.05$).

shown in Table 2, the dissolution rate of potato starch exhibited a significant increase with increasing number of FTC. This likely stems from the structural impairment of starch granules induced by FTC, thereby augmenting the likelihood of starch molecules leaching from within the starch granules. This observation parallels findings reported by Liu et al., who observed a trend wherein the solubility of wheat starch increased in proportion to the number of FTC applied (Liu et al., 2022).

The swelling force is defined as the capacity of starch to absorb water at a certain temperature, primarily attributed to the binding affinity between starch molecular chains and water molecules (Hasjim et al., 2012). The swelling of starch granules is primarily influenced by amylopectin, while amylose inhibits this process to maintain the integrity of the granules. As presented in Table 2, the swelling force of mashed potatoes decreased with increasing FTC. The original structure of starch granules is destroyed by FTC, leading to a reduction in the swelling power casused by inhibition of amylopectin (Hasjim et al., 2012).

The XRD pattern of mashed potatoes during FTC is shown in Fig. S2. The mashed potatoes starch showed a B-type crystal structure with 2θ located at 14°, 17° and 22°. The mashed potatoes starch was partially gelatinized during the cooking process, and part of the B-type crystal structure was transformed into an amorphous structure, with the relative crystallinity decreasing from 20.49 % to 15.19 % with 5 FTC. This suggested that ice crystal growth and recrystallization within the potato starch granules were promoted by FTC, resulting in the breakdown of the hydrogen bonds within the starch and the destruction of the double helix structure. This further proves that the crystal structure of potato starch was destroyed by FTC and the swelling force of starch was decreased.

3.5. Rheological properties

The storage modulus (G') signifies the elastic nature of a substance, representing the energy restored following a sinusoidal deformation during a vibration period. The loss modulus (G") represents the viscous nature of a substance, denoting the energy consumed after each cycle of sinusoidal deformation (Zhou et al., 2022). Both G' and G" values exhibited an increase with increasing angular frequency, and the dominance of G' over G" values indicated the prevalence of elasticity over viscosity in mashed potatoes (Fig. 1). Moreover, the G' and G" values decreased with increasing number of FTC at identical frequencies, suggesting a reduction in the viscoelasticity of mashed potatoes due to FTC treatment. The result was consistent with the observed trend of decreased viscosity and elasticity in determining mashed potatoes texture (Section 3.2) with increasing number of FTC. The loss of soluble starch and protein in mashed potatoes due to structural damage (Table 2) impacts the interaction between mashed potatoes granules, consequently reducing G' and G". Freezing treatment induces the breakage of hydrogen bonds in starch and crystal structures. The starch molecular arrangement becomes relaxed, leading to a decrease in viscosity. Additionally, ice crystal formation compresses the gluten network during FTC, reducing gluten crosslinking and thereby weakening the dough's water-binding capacity, leading to decreased stickiness and elasticity (Mohsen et al., 2022).

3.6. Water distribution in mashed potatoes

The migration characteristics of water molecules in mashed potatoes were assessed using the LF-NMR technique, which elucidated the relaxation behavior and amplitude of hydrogen protons. The impact of FTC treatment on the lateral relaxation time of mashed potatoes is shown in Fig. 2. The three distinct states of water within mashed potatoes were represented by three corresponding peaks in the transverse relaxation time distribution (T_2) map. The first peak (T_{21}) corresponds to tightly bound water in mashed potatoes (1–10 ms), the second peak (T_{22}) represents weakly bound water (10–100 ms), and the third peak

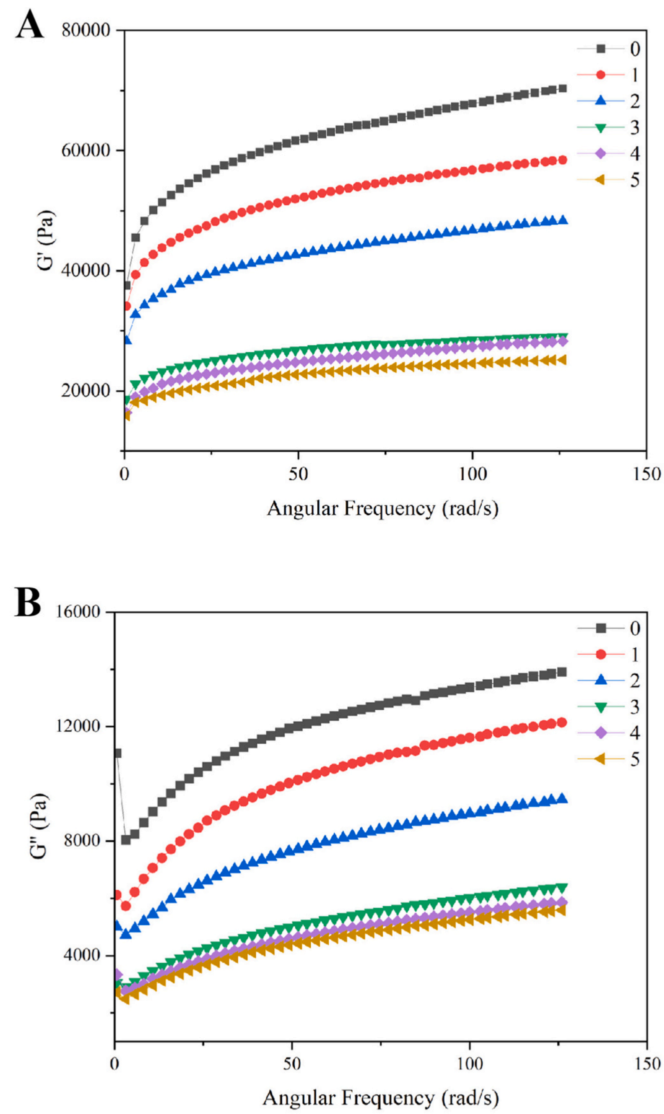

Fig. 1. Changes in storage modulus (A) and loss modulus (B) of mashed potato with different freeze-thaw cycles.

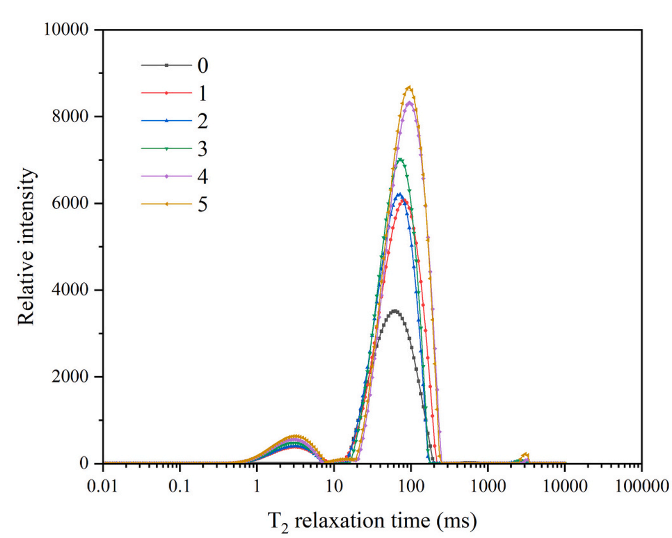

Fig. 2. Lateral relaxation time distribution of mashed potato during freeze-thaw cycle.

(T_{23}) denotes free water with high mobility that can freely move through the system (100–10,000 ms) (Zhuang et al., 2023). The relative intensity of the peak corresponding to weakly bound water, T_{22}, significantly increased with the magnify of FTC, which was attributed to the increased content of weakly bound water in mashed potatoes. The main reasons for the high content of weakly bound water were the fracture of hydrogen bonds and the damage to the protein network. This may lead to the more starch granule channel pores, which reduced the degree of protein-water interaction and promoted water loss (He et al., 2024; Tao et al., 2024). Potato cell walls were destroyed due to the formation of larger ice crystals during the freezing process. In fact, the damaged cell walls were incapable of retaining water, causing the migration of water from the intracellular space to the extracellular space. The moisture loss post-thawing could not be re-absorbed by the cells, thus redistributing within the intercellular space and resulting in the occurrence of juice loss in mashed potatoes. This phenomenon parallels the water dynamics observed in potatoes during FTC, as reported by Zhang et al. (Zhang et al., 2022). Tan et al. similarly observed that FTC treatment destroyed the tissue structure of sea cucumber, accompanied by a significant increase in the fluidity of weakly bound water (Tan et al., 2018).

The variations in peak time, width, and area of T_{22} corresponding to mashed potatoes subjected to various FTC treatments are shown in Table 2. The T_{22} peak time for mashed potatoes shifted from 65.22 to 102.59 ms with increasing number of FTC. The phenomenon of mashed potatoes loosely binding to water was attributed to cellular destruction during the freezing process, leading to water spillage from the mashed potatoes. Furthermore, the T_{22} peak width and area of mashed potatoes exhibited a significant increase with an increasing number of FTC ($p < 0.05$). This indicates FTC weakens the bond between mashed potatoes and water, thereby a higher propensity for water molecules to freeze (Zhuang et al., 2023). The migration of bound water to weakly bound water contributes to the enlarged permeability of mashed potatoes cell walls because of freezing-induced damage, consequently increasing the T_{22} peak area. In a previous study, they calculated changes in the amount of freezable water during FTC and found that the water in the dough migrated as the FTC increased, leading to an increase in the content of frozen water (Wei, Sun, et al., 2024). Compared with the previous study, this study was used to detect the migration of bound water to weakly bound water in mashed potatoes due to ice crystal growth and recrystallization.

3.7. Changes in microstructure of mashed potatoes

3.7.1. SEM

The impact of FTC treatment on the microstructure of mashed potatoes granules is shown in Fig. 3A. Mashed potatoes granules not subjected to FTC exhibited an elliptical or round shape with smooth and intact surfaces. Conversely, after two to three FTC, the mashed potatoes granules displayed a rough surface and an increased presence of irregular polygonal holes, accompanied by slight shrinkage. The grooves, holes, and cracks on the surface of potato granules because the potato granule was severely damaged structure following four to five FTC. The formation of large ice crystals within the cells has been shown to disrupt the structural integrity of the mashed potatoes granules during FTC (Hasjim et al., 2012). Previous studies have shown that the interactions between potato starch chains are perturbed by the formation of ice crystals during FTC, resulting in the destruction of the network structure of starch chains and the microstructure of potato starch (Abedi et al., 2022). Furthermore, FTC exacerbated the presence of holes and cracks on the surface of potato granules. The enlargement of internal channels in potato granules was attributed to the unique mechanical force exerted on starch granules during successive FTC and ice crystal melting. The mechanical force generated during FTC treatments enlarged the internal channels within potato granules, leading to the dissolution of more soluble substances and the redistribution of water in the pores. Upon freeze-drying, moisture sublimation occurs, resulting in a more concave surface of the granules (Martínez et al., 2022). Researchers suggest that the holes on the surface of starch granules are a consequence of the disruption caused by the melting of ice crystals during FTC (Liu et al., 2019; Martínez et al., 2022).

3.7.2. CLSM

The impact of FTC treatment on the microstructure of mashed potatoes was further examined through CLSM (Fig. 3). Protein was stained red using Rhodamine B, while starch was stained green using FITC. The cross-linking of protein and starch was visualized by the manifestation of yellow hues, while areas of black denoted pores or voids induced by structural deterioration from ice crystal formation (Tao et al., 2023).

In the absence of FTC treatment, starch and protein exhibited uniform distribution, with the protein embedded in the starch granules to establish a continuous starch-protein matrix, indicating a dense and intact mashed potatoes structure. However, the CLSM depiction of mashed potatoes exhibited a transition from a green-yellow alternating pattern to a predominance of red areas with increasing number of FTC (Fig. 3B). The structure of mashed potatoes is disrupted by temperature fluctuations and ice crystal growth during FTC (Wei, Zhang, & Xie, 2024). The red areas in mashed potatoes were caused by ice crystals breaked up the mashed potatoes cells, allowing proteins and starches to separate. Proteins stained red by Rhodamine B float outside the cells to form red areas. We observed that the starch granules in mashed potatoes transitioned from elliptical to irregular polygonal shapes, accompanied by gradual protein depletion and separation from starch (Fig. 3C). The molecular structure of mashed potatoes was disrupted by ice crystal formation during FTC, resulting in diminished adhesive forces between protein and starch granules, consequently expediting moisture loss from the mashed potatoes (Lu et al., 2022). These results were consistent with a decrease in WHC and an increase in the protein solubility of mashed potatoes. It is noteworthy that structural damage induced by ice crystal formation during FTC led to an increase in pores between starch granules. Previous studies have reported alterations in the number, shape, and size of ice crystals during FTC, leading to a substantial enlargement of pores in dough owing to starch and protein separation (Cui et al., 2023; Wei et al., 2023). Zhou et al. found that the dehydration efficiency, moisture status, microstructure and nutrient retention of potato tubers were affected by freeze-thaw treatments with different freezing rates (Zhou et al., 2022). In this study, we found that the sensory quality, water distribution and microstructure of mashed potatoes were affected by FTC. This helps us to elucidate the relationship between the FTC and the structure of mashed potatoes, and lays a solid foundation for future improvements in the quality of mashed potatoes.

3.8. GC-IMS

The qualitative analysis of volatile compounds in mashed potatoes was conducted through GC-IMS. The changes in volatile compounds were visualized through three-dimensional morphology at varying FTC conditions (Fig. 4A). The X-axis, Y-axis, and *Z*-axis correspond to retention time, ion migration time, and peak intensity, respectively (Huang et al., 2023). The content of volatile compounds significantly increased with increasing number of FTC (refer to red circle for details). Further comparative analysis was conducted employing two-dimensional chromatograms (Fig. 4B) and subtraction graphs (Fig. 4C) of mashed potatoes to clarify the changes in volatile compounds. The characteristic peaks of VOCs in mashed potatoes were concentrated between 400 and 1200 s, with drift times distributed between 1.0 and 1.75 s. Upon subtracting the chromatogram after FTC from the reference chromatogram, we obtained a comparative chromatogram, with red and blue dots representing compounds with higher or lower content, respectively, compared to the untreated mashed potatoes used as a reference (Fig. 4C) (Deng et al., 2024). Following one to three FTC, the peak intensity was low within the retention time range of 800–1200 s, and the VOC drift time was 1.0–1.5 s. The peak intensity increased

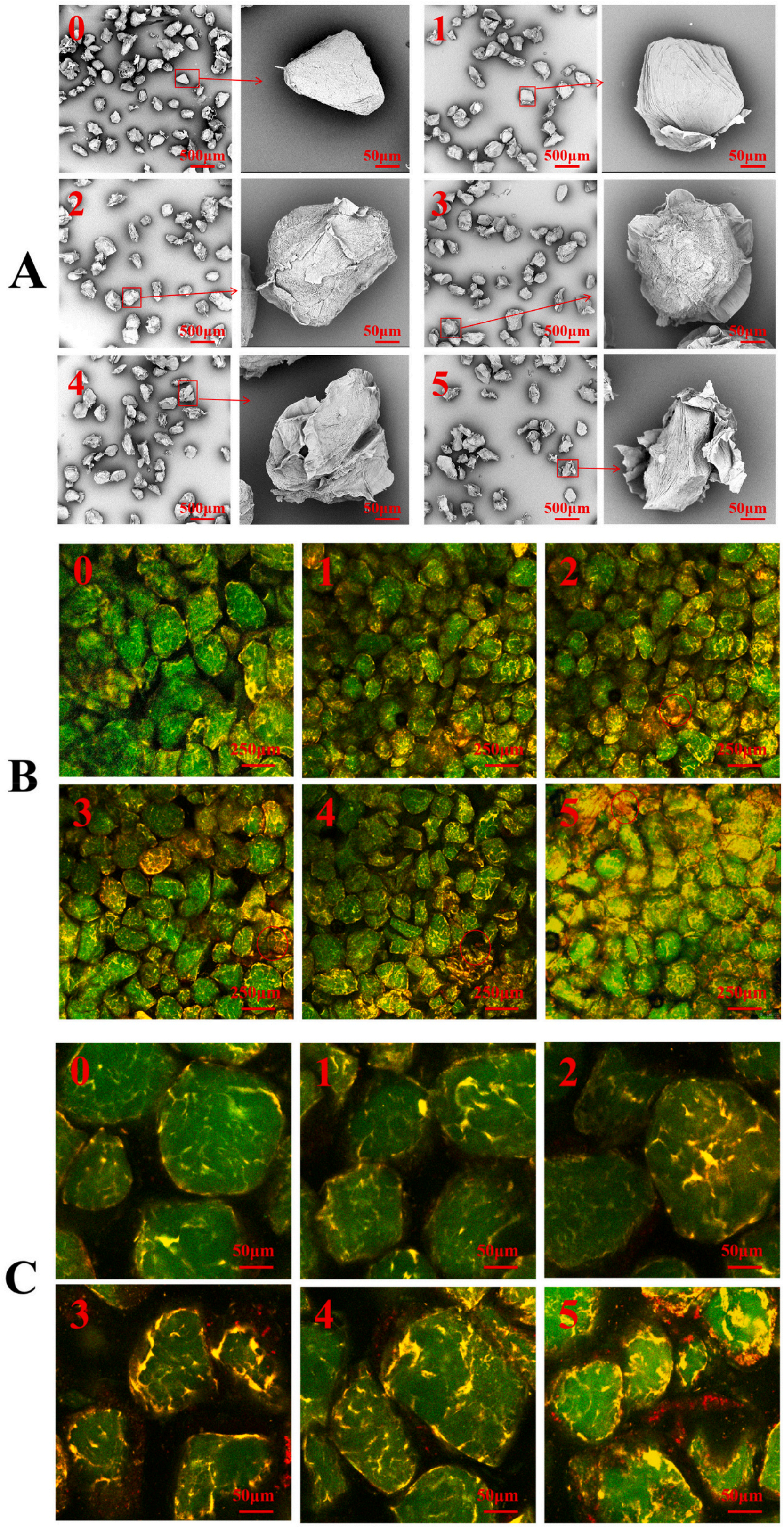

Fig. 3. SEM (A) and CLSM (B and C) micrographs of mashed potato during freeze-thaw cycle (CLSM: green for starch, red for protein, and yellow for cross-linking of the two macromolecules).

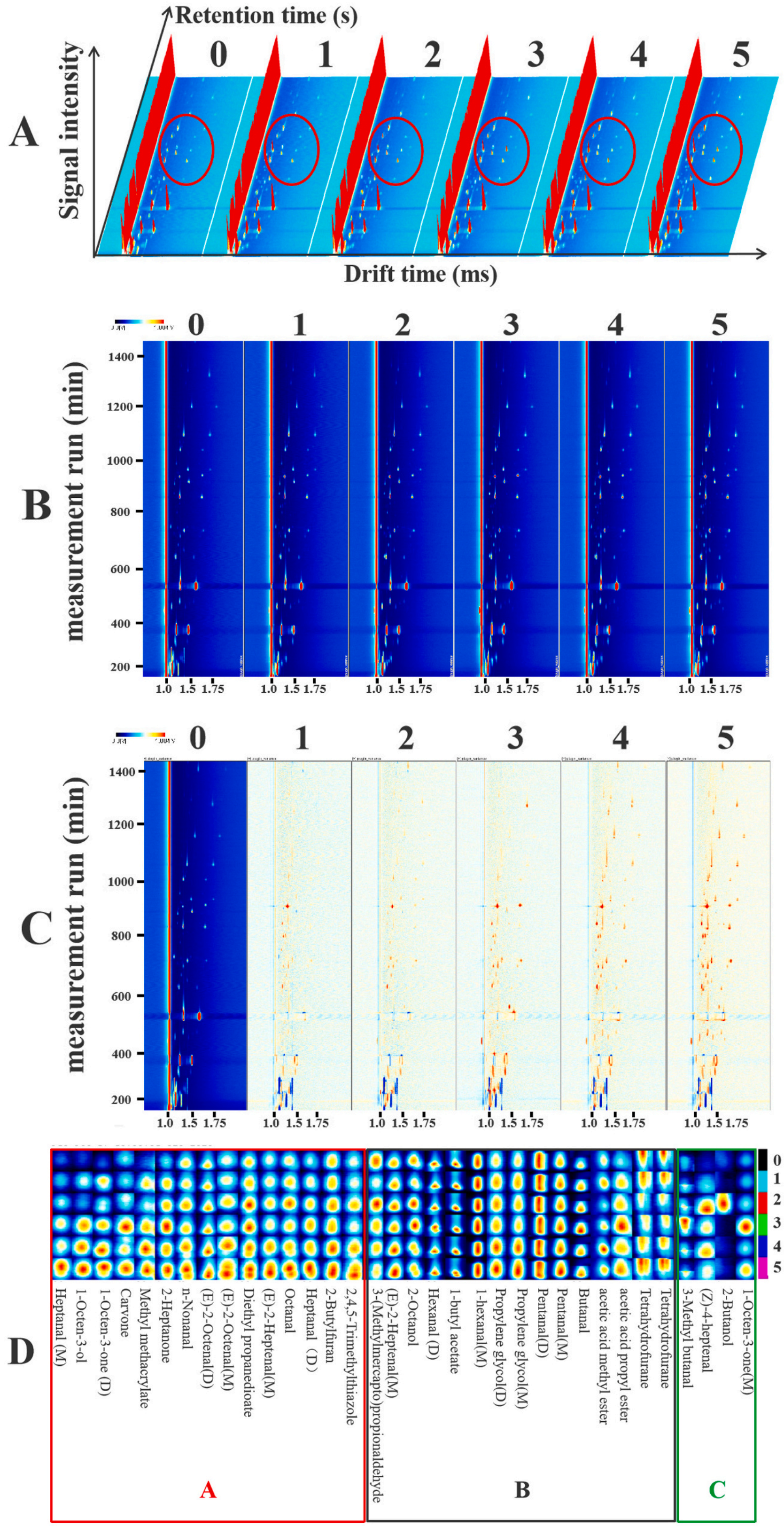

Fig. 4. Three-dimensional topography (A), 2D-topographic plots (B), 2D-topographic subtraction plots (C) and VOCs fingerprint comparisons (D) of mashed potato with different freeze-thaw cycles.

rapidly following three to five FTC, and a characteristic peak was observed at a drift time of 1.75 s, indicating that new VOCs were produced during the late stage of FTC, which accounted for a substantial proportion. The dynamic changes in volatile compounds were compared across various samples through fingerprint analysis to obtain insights (Fig. 4D). The fingerprint spectra were used to represent different number of FTC in each row, with each column expressing a volatile compound (Song et al., 2023).

A total of 33 volatile compounds of mashed potatoes were identified, including 14 aldehydes, 5 alcohols, 5 esters, 4 ketones, 2 furans, and 3 other compounds (Table S2). The contribution of VOCs to the overall aroma of mashed potatoes was assessed based on ROAVs, which are associated not only with their concentration but also with their odor threshold. Flavors with lower odor thresholds were easily smelled by people, whereas flavors with higher odor thresholds were difficult to detect (Xi et al., 2024; Xu et al., 2022). The 23 VOCs were identified as key volatile compounds of mashed potatoes according to ROAV >1 (Table S1). Prior to FTC, the aroma of mashed potatoes was influenced primarily by hexanal, 3-(methylmercapto) propionaldehyde, 2-octanol, and 1-butyl acetate, imparting green, fleshy, sweet, fatty, and fruity odors. However, the ROAVs of these volatile compounds gradually reduced after five FTC, indicating a decline in the original characteristic flavor of mashed potatoes with increasing number of FTC. Similar volatile compounds have been detected in cooked potatoes, and these flavors have been observed to decrease with prolonged storage duration (Nahar et al., 2020). The increase in VOC concentration in mashed potatoes with increasing number of FTC is shown in Fig. 4D (a). The ROAV exhibited a significant increase for VOCs including heptanal, n-nonanal, (E)-2-octenal, 1-octen-3-ol, 1-octen-3-one, and 2-butylfuran following five FTC. The aroma profile of mashed potatoes was characterized by fatty, mushroom, and wine odors, indicating an exacerbation of undesirable flavors in mashed potatoes with increasing number of FTC. The repeated formation of ice crystals during the FTC disrupted the microstructure of mashed potatoes, resulting in the release of proteases, lipases, and intracellular oxidase precursors that accelerate lipid oxidation. In addition, ice crystals destroyed the selective permeability of the cell membrane, leading to the loss of oxidation factors from the cell, forcing the oxidation of fat. Satisfactory flavors were produced due to moderate lipid oxidation, and the amouts of heptanal and n-nonanal increased because of increased lipid oxidation during FTC. The 1-octene-3-one was also formed primarily through lipid oxidation. The mushroom flavor of 1-octene-3-one increased with the promotion of lipid oxidation by FTC. The ROAV values of heptanal and 1-octene-3-one increased due to excessive fat oxidation, resulting in an unacceptable fatty and mushroom taste. The odor activity values of lipid degradation products, such as hexanal, nonanal, and 1-octen-3-one, in potatoes have been reported to be increased during storage (Shi et al., 2024). Furthermore, there was no significant change observed in 14 volatile compounds, such as propylene glycol, 1-butyl acetate, acetic acid methyl ester, acetic acid propyl ester, and 2-octanol with the increasing number of FTC (Fig. 4D (b)). The mashed potatoes were influenced by repeated FTC, resulting in an increase in the ROAV of 3-methyl butanal from 0 to 3.35. Moreover, the addition of (Z)-4-heptenal led to the mashed potatoes acquiring a woody and bitter taste (Fig. 4D (c)). The bitter taste and fatty flavor of potatoes have been found to develop as a result of lipid degradation during storage (Xu et al., 2022). In conclusion, the undesirable flavors (e.g., fatty and bitter tastes) of mashed potatoes were intensified, while the original characteristic flavor of mashed potatoes diminished with increasing number of FTC. A previous study had shown that fresh potatoes were rich in lipoxygenase, and potato cells were damaged due to peeled, cutted and other operations. These processes promoted the contact between lipoxygenase and its substrate, and unsaturated fatty acids were oxidized to hydroperoxide (Jiang et al., 2023). In this study, the unpleasant odors of mashed potatoes were caused by the destruction of mashed potatoes cells by ice crystals during FTC, which accelerated lipase release and lipid oxidation.

4. Conclusion

In this study, the texture characteristics, microstructure, and volatile compounds of mashed potatoes subjected to FTC were investigated. FTC decreased the hardness and stickiness of mashed potatoes. The increase in the L^* value indicated that mashed potatoes turned white owing to dispersion of water onto the surface the water spillage onto the surface. Concurrently, the content of weakly bound water in the mashed potatoes increased, leading to increased water overflow. The interactions between potato starch chains were perturbed by the formation of ice crystals during FTC, resulting in the destruction of the network structure of starch chains and the microstructure of potato starch. The ice crystals destroyed the selective permeability of the cell membrane, leading to the loss of fat oxidase from the cell, forcing the oxidation of fat with increasing number of FTC. Unpleasant flavors, such as fatty and bitter tastes, intensified in mashed potatoes, severely affecting sensory quality and consumer preference. Our findings hold great significance for analyzing the potential mechanisms underlying the interaction of mashed potatoes components under FTC conditions as well as the development and improvement of prefabricated mashed potatoes dishes. We will focus on the next step to improve the quality of the freeze-thaw mashed potatoes and strive to expand their the depth and scope of mashed potatoes applications in the food industry.

CRediT authorship contribution statement

Yingying Yu: Writing – review & editing, Writing – original draft, Investigation, Data curation. **Wuyin Weng:** Writing – review & editing, Methodology, Data curation. **Zhongyang Ren:** Writing – review & editing, Funding acquisition. **Yucang Zhang:** Supervision, Funding acquisition. **Ping Li:** Writing – review & editing. **Linfan Shi:** Writing – review & editing, Methodology, Funding acquisition, Data curation, Conceptualization.

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

The authors do not have permission to share data.

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Appendix A. Supplementary data

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