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Sensory and chemical characterization of chestnuts processed in different methods using instrumental analyses and the Check-all-that-apply method

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ABSTRACT

Chestnuts are known for their unique sensory characteristics and nutritional value. However, the changes in their sensory attributes after processing remain unclear. This study used instrumental analysis and sensory evaluation combined with multivariate statistical analysis to investigate the effect of packaging and thermal sterilization procedures on the sensory characteristics of chestnuts. The results showed that the significant variations ($p < 0.05$) between the different processing methods for chestnuts were revealed via the texture analysis, headspace solid-phase microextraction gas chromatography \times gas chromatography-time of flight-mass spectrometry (HS-SPME-GC \times GC-TOF-MS), and check-all-that-apply (CATA) sensory evaluation. The packaging had a more significant influence on the sensory quality of the chestnuts than thermal sterilization procedures. The HS-SPME-GC \times GC-TOF-MS identified 116 volatile compounds, and the partial least squares-discriminant analysis (PLS-DA) identified the 28 volatile compounds responsible for the similarities and differences among different processing methods of chestnuts. Principal component analysis (PCA) combined with texture analysis and the fingerprint of HS-SPME-GC \times GC-TOF-MS, as well as the correspondence analysis (CA) analysis of CATA, could discriminate samples from different packaging of chestnuts. This study provides chestnuts processing recommendations for satisfying consumer preferences and predicting possible related quality changes.

1. Introduction

Chestnuts (*Castanea mollissima* Blume) originated in China and have been cultivated for over 2000 years, which are widely distributed throughout Europe, Asia, and North America (Yang et al., 2022). Chestnuts are an important edible fruit and economic food resource in China and make a large contribution to the national economy (Kan et al., 2016). Chestnuts are considered an excellent energy source and are becoming increasingly popular due to their nutritional composition and potential health benefits (Özcan et al., 2023). Following consumer demand for industrialized food products, packaged steam-sterilized chestnut kernels have been industrially produced. The steam thermal sterilization process ensures the homogeneity of the product, but it also impacts the product's properties. The physicochemical properties, nutritional characteristics, and physiological activities of raw and processed chestnuts have been thoroughly studied (Li et al., 2022a). Still,

the effect of processing on the organoleptic properties of chestnuts has been minimally reported.

Sensory analysis plays an important role in the development and production of food products (Viana et al., 2021). Food quality characterization methods are divided into sensory and instrumental analytical techniques. Among the instrumental analyses, textural properties vitally affect the sensory characteristics and acceptability of food. Kan et al. demonstrated that thermal treatment altered the textural properties of chestnuts, with a significant reduction in hardness, chewiness, and gumminess due to the gradual destruction of the granular starch structure (Kan et al., 2016). In addition to food texture, flavor also has a very important influence on the sensory quality of food. Gas chromatography \times gas chromatography-time of flight-mass spectrometry (GC \times GC-TOF-MS) is a powerful analytical tool for identifying low-concentration flavors in highly complex food samples, offering superior separation, highly selective, and enhanced resolution ability (Schwanz et al., 2019).

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However, no research is available involving chestnut flavor determination using this technique.

Sensory evaluation methods based on consumer assessment are currently attracting increasing attention. Check-all-that-apply (CATA) is a popular method for consumer-based sensory characterization, which only requires to selection of appropriate attributes from a predefined list to describe each focal sample (Ares et al., 2014, Vigneau et al., 2022). CATA has been combined with instrumental analysis for the flavor and sensory evaluation of Chinese bog bilberry wines, distinct parts of Chinese blanched chicken, thermally processed sturgeon meat, and processed fava beans (Xu et al., 2020, Li et al., 2022b, Lin et al., 2022, Sharan et al., 2022), but they have not been applied to the flavor and sensory assessment of thermally processed chestnuts.

In this study, shelled chestnut kernels were packaged in different ways and then treated with thermal sterilization. This study aims to: (i) establish volatile fingerprint profiles of chestnut kernels by GC×GC-TOF-MS, (ii) establish a sensory dictionary for evaluating chestnut kernels using CATA methods, (iii) explore the relationship between sensory attributes and flavor substances of chestnut kernels by multivariate statistical analysis, and (iv) investigated the effect of packaging methods and thermal sterilization procedures on the aroma characteristics of chestnut kernels. The results contribute to clarifying the factors affecting consumer preferences for chestnuts exposed to different processing methods to provide guidance to chestnut producers. It promotes the high-quality utilization of raw chestnut materials and production development while meeting the needs of consumers.

2. Materials and methods

2.1. Sample preparation

The chestnuts (Dabanhong) were picked in Kuancheng in Hebei Province, China, in September 2022. They were stored at 4 °C after peeling. Chestnuts were packaged in different forms using a DZ-700/2S vacuum packaging machine (Yifei Intelligent Packaging Machinery Co., Ltd., Anhui, China). Fig. 1 displays the chestnut preparation process flow chart. The basic physicochemical indexes of chestnuts are shown in

Table S1.

2.2. The textural profile analysis (TPA) of the chestnuts

A TMS-Touch textural analyzer (FTC, America) was used to analyze the chestnut texture. A chestnut sample was placed in the middle of the analyzer plate, after which tests were performed using a cylindrical probe (TMS 38.1 mm Perspex) with a flat surface (dia. 38.1 mm) to determine its hardness, adhesiveness, cohesiveness, springiness, gumminess, and chewiness. The experiment was performed using a method described by Kan et al. with slight modifications (Kan et al., 2016). The analytical conditions included a 2 mm height, a 3 mm reserved height, a 1 mm/s test speed, a 2 s holding time, a 2 g trigger, and 50 % compression.

2.3. The volatile compound analysis using HS-SPME-GC×GC-TOF-MS

Volatile compounds in chestnuts were extracted using a triphasic 50/30 μm DVB/CAR/PDMS SPME fiber (Supelco Co., Bellefonte, PA, USA) with a fiber length of 2 cm. To improve the extraction efficacy, the crushed chestnut samples were vortexed with saturated NaCl at a ratio of 5:3 (Xu et al., 2022). Additionally, 1 μL of 2-methyl, 3-heptanone was added as an internal standard with a mass concentration of 0.816 mg/mL. The sample was equilibrated in a water bath for 30 min at 60 °C to acquire the extract, which was sorbed for 30 min using the SPME extraction head in the headspace.

The method delineated by Yang et al. (Yang et al., 2021) with slight modifications was employed to analyze the flavor components in the processed chestnuts using an 8890A-7000B GC-MS instrument (Agilent Technologies Inc., Santa Clara, CA, USA) equipped with a TOF-MS (He Xin Mass Spectrometry Co., Ltd., Guangzhou, China). A DB-WAX column (polar, 60 m × 0.25 mm, and 0.25 μm) and a mid-polar DB-17 ms column (non-polar, 1.85 m × 0.18 mm, and 0.18 μm) from Agilent (Palo Alto, CA, USA) were used for first and second-dimension GC analysis. An SSM1800 solid-state modulator (J&X Technology Co. Ltd, Shanghai, China) with a 4 s modulation period was inserted between the two columns to heat and cool the volatile compounds, maintaining the cold-

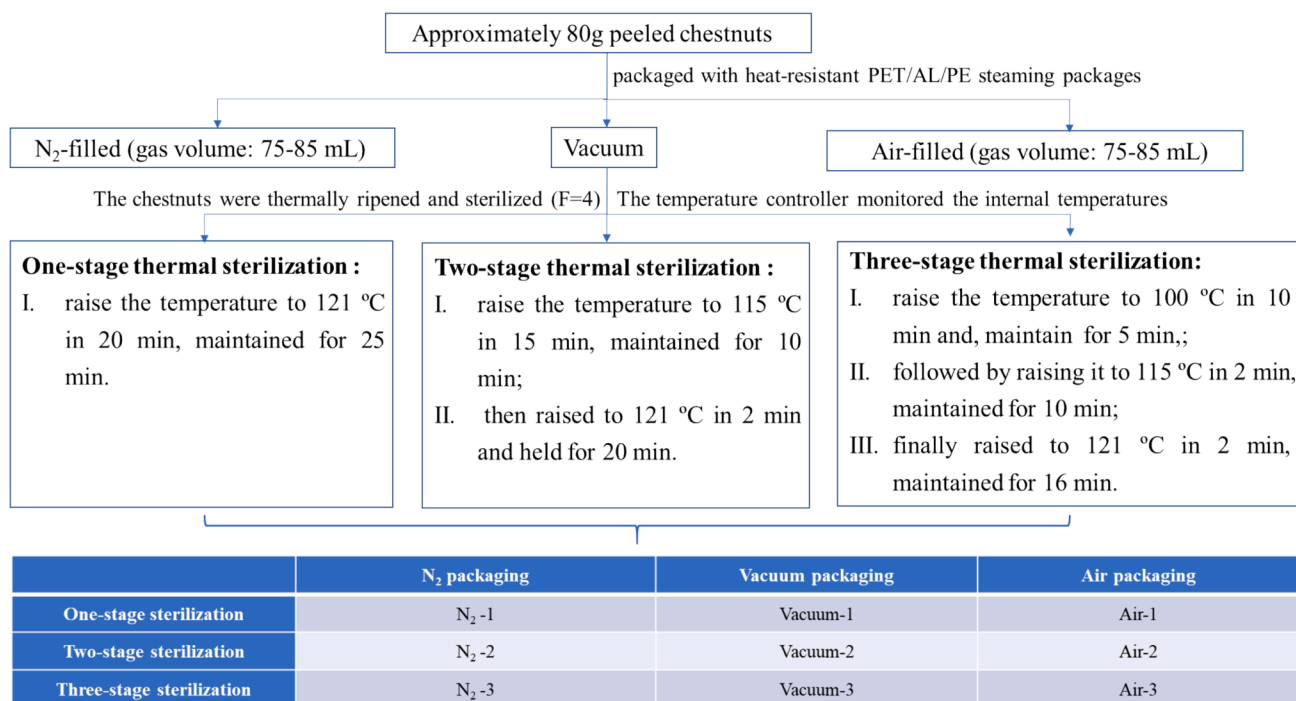


Fig. 1. Chestnut preparation process flow chart.

zone temperature at $-51\text{ }^{\circ}\text{C}$. The column was initially heated to $40\text{ }^{\circ}\text{C}$ and maintained for 3 min, followed by an increase to $230\text{ }^{\circ}\text{C}$ at $6\text{ }^{\circ}\text{C}/\text{min}$, where it was held for 5 min. The carrier gas consisted of 99.999 % ultrapure helium (Beijing AP BAIF Gases Industry Co., Ltd., Beijing, China), while the electron-impact mass spectra were acquired at an ionization energy of 70 eV in a 50–350 amu m/z scanning range. The quadrupole temperature was set at $150\text{ }^{\circ}\text{C}$, while that of the MS source was $230\text{ }^{\circ}\text{C}$.

The Canvas software was employed to process the 2D and draw complete 2D TIC contour maps. The peaks displaying signal-to-noise ratios over 10 were identified, where the individual peaks corresponded to a specific compound, each of which was determined via the dimensional retention time (min) in the x-axis direction and the retention time (s) in the y-axis direction. The identification of volatile components was performed by comparing their LRI values, one- and two-dimensional GC \times GC-TOF-MS retention times, and mass spectra with the NIST 17 database. The retention index (RI) of compounds was calculated relative to serial alkanes (C7 ~ C30), and compared with the RI values reported in the literature. The peak area ratio of the compound to the internal standard was employed to determine the individual volatile component levels via the internal standard semi-quantitative approach (Yang et al., 2021).

2.4. The sensory evaluation using the CATA test

The internal sensory panel of the Sensory Evaluation Centre (SEC) comprised 12 trained panelists (six females and six males, aged 22 to 28) and was generated from the School of Food and Health, Beijing Technology and Business University (BTBU). The free choice profiling (FCP) method was first used to describe their sensory descriptors freely. Then a series of sensory evaluation descriptors were obtained from relevant literature (Warmund et al., 2011, Corona et al., 2021). Combining sensory descriptors from FCP and the literature to define the sensory chestnut sample descriptors for the CATA questionnaire (Table S2). A sensory analysis sheet was presented to survey the visual, olfactory (flavor), and gustatory (texture and taste), attributes of the different chestnuts in the various processing systems. The trained panelists were excluded from the consumer study.

The consumer evaluation included 102 untrained participants (58 females and 44 males, aged 20–32) consisting primarily of BTBU students. Only voluntary, healthy participants, who were not pregnant, and not intolerant to alcohol were included. The sensory evaluation was performed in the sensory evaluation lab at the School of Food and Health, BTBU (ISO, 8589-2007). The participants were briefly trained in the experimental procedure and using the CATA questionnaire. Ten chestnut samples were randomly coded with three digits and presented in a monadic sequence at $25\text{ }^{\circ}\text{C}$. Water ($25\text{ }^{\circ}\text{C}$) was used as a palate cleanser between samples to avoid the cross-linking effect. The participants were required to observe and smell each sample and then taste it. Re-testing was not allowed to prevent sensory fatigue. The participants were asked to complete a CATA questionnaire consisting of 27 sensory terms by selecting those they perceived from the sample while rating the acceptability of each specimen on a 9-point hedonic scale (“1” = “dislike extremely”, “5” = “neither like nor dislike”, “9” = “like extremely”). All sensory tests were conducted in an air-conditioned room at $25\text{ }^{\circ}\text{C}$ to meet the ISO 8589-2007 standard. The participants were asked to sit quietly in individual booths during each sensory evaluation session. The sensory data were collected using Questionnaire Star (<https://www.wjx.cn/>).

2.5. Statistical analysis

Statistical analysis was conducted to evaluate the variance and significant difference tests using one-way analysis of variance (ANOVA) and Duncan's multiple range test ($p < 0.05$). The data were merged and subjected to multivariate statistical analysis. To perform heat map

analysis, the OmicStudio Cloud Platform (<https://www.omicstudio.cn>) was utilized, while principal component analysis (PCA) and partial least squares-discriminant analysis (PLS-DA) of the volatile compounds were carried out using SIMCA-P version 13.0 software (Umetrics, Malmö, Sweden). MetaboAnalyst (<https://www.metaboanalyst.ca/>) was utilized to conduct the textural hierarchical cluster analysis (HCA). XLSTAT 2019 (New York, USA) was used for partial least squares regression (PLSR) and CATA analysis. Cochran's Q test was employed in the XLSTAT software to determine the significant differences between the samples for each sensory term. To perform pairwise comparisons, sign tests were utilized. Correspondence analysis (CA) was carried out, utilizing chi-square distances to obtain a bi-dimensional representation of the samples based on the frequency of emotional and sensory descriptors. The remained images were then plotted using Origin (version 2018, Origin Lab, Hampton, Massachusetts, USA).

3. Results and discussion

3.1. The textural evaluation of the chestnuts exposed to different packing methods during thermal sterilization

Texture is important to assess the palatability of food products, which is closely related to rheological and structural changes (Min et al., 2023). The texture is significantly influenced by changes in moisture, fats, structural carbohydrate levels, hydrocolloids, and proteins in food products (Kutlu et al., 2022). This study did not employ TPA to test raw chestnuts since they were too hard and beyond the limits of the force arm of the mass spectrometer ($F_{\text{max}} = 250\text{ N}$). While, the physical, chemical, and structural changes that occur during thermal sterilization via moisture evaporation, starch gelatinization, protein structure denaturation, and pectin cell solubilization significantly affect the textural properties of chestnuts (Patra et al., 2022). These changes modified the textural properties of the processed chestnuts, improving consumer acceptance. Therefore, to analyze the impact of diverse handling methods on chestnut texture, an unconstrained dimensionality-reduced PCA approach was executed on the TPA output dataset using the TPA test indexes as output variables. The PCA findings (Fig. 2A) revealed that two principal components were responsible for 85.5 % of the overall variance. PC1 accounted for 69.3 %, while PC2 attributed to 16.2 %, signifying that they adequately represented the majority of taste information in the samples. In conjunction with Fig. 2B, it was observed that the sterilization procedure employed in this investigation did not significantly influence the textural properties of the chestnut samples. On the contrary, the packing methods exhibited a substantial impact. Among the three packaging techniques, the vacuum-packaged samples displayed distinct textural properties compared to the other two methods, whereas the N_2 and air packaging samples exhibited comparable textures.

In the process of thermal sterilization, the escape of vapor via the food microstructure generated weaknesses, cracks, defects, capillaries, and channels inside the cellular structure, reducing the hardness. Hardness is an important parameter that directly reflects the mouthfeel of food and significantly affects the gumminess and cohesiveness of the textural attributes (Min et al., 2023). The N_2 -packaged samples were slightly harder and chewier than those packaged using air, while the adhesiveness was similar to the air-packaged samples but significantly higher than the vacuum-packaged samples (Table S3). This phenomenon was partly due to that, the saturated steam internal molecular energy and relative motion between the molecules increased in the sustained high-temperature thermal sterilization environment, expanding steam volume. Since vacuum packaging restricted the movement space, the expanding water vapor destroyed and damaged the internal chestnut structure, while the high temperature accelerated the degradation of sugars, proteins, and lipids. Therefore, the hardness, gumminess, and cohesiveness are significantly lower than in the samples of the other two packaging methods (Woo Choi et al., 2023). In the N_2 - and air-

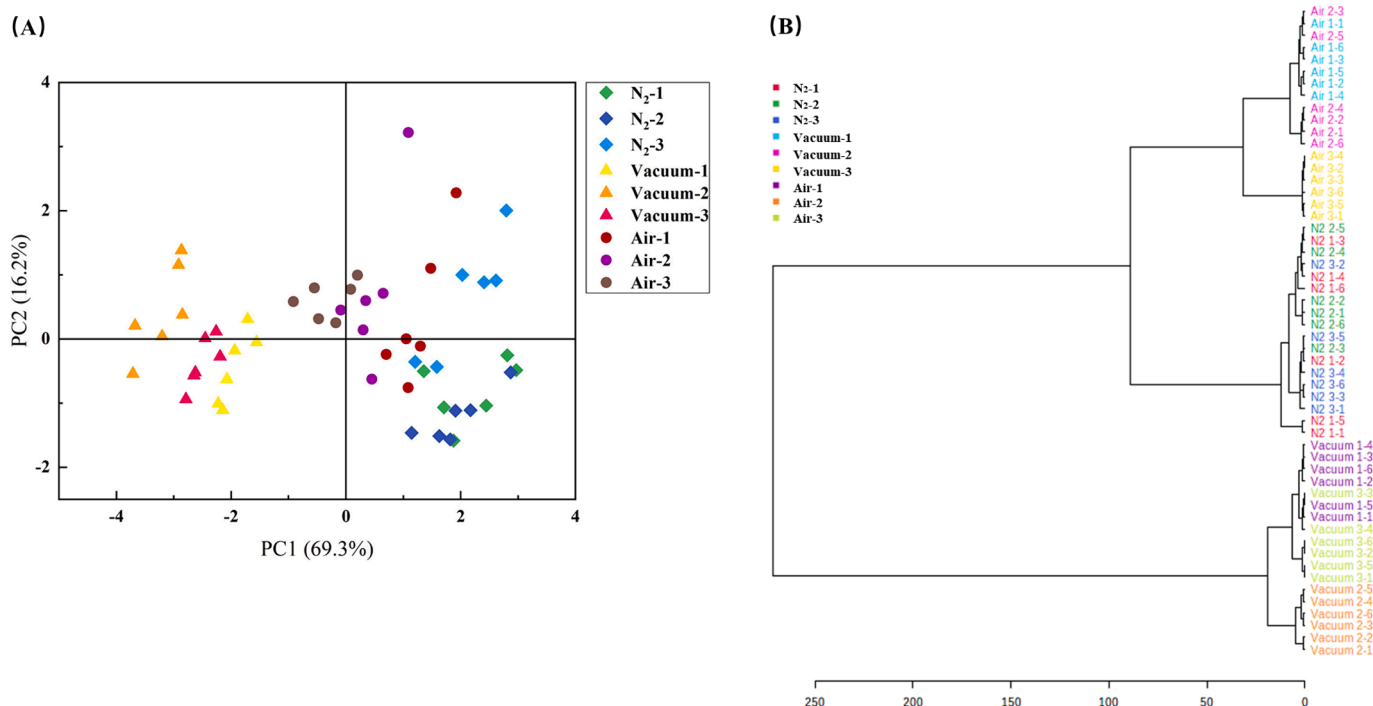


Fig. 2. The textural analysis of the chestnuts exposed to different processing methods. (A) PCA analysis of textural attributes. (B) HCA analysis of textural attributes.

packaged products, the expanding water vapor had enough release space during the thermal sterilization process, limiting the destruction of the chestnut structure by water evaporation compared to vacuum packaging, while a series of physicochemical changes occurred, such as starch pasting and protein deformation. Various reactions are more likely to occur in the air-packaged samples during thermal processing due to oxygen catalysis. Since the N₂-packaged samples were not exposed to the same interfering factors, the hardness, gumminess, and cohesiveness of the chestnuts were slightly stronger than the air-packaged samples (Ge et al., 2021).

3.2. Analysis of volatile compounds in chestnuts exposed to different processing methods by GC×GC-TOF-MS

Food flavor is a vital factor influencing consumer choice. Thermal sterilization of chestnuts involves complex processes such as starch pasting, protein denaturation, and fat oxidation, altering the flavor of the food (Fu et al., 2021). To explore the effect of processing on the flavor of chestnut, HS-SPME-GC×GC-TOF-MS was utilized to assess the volatile substances. A total of 116 volatile compounds were detected in the raw and processed chestnuts (Fig. 3, Table S4), including 32 alcohols, 25 aldehydes, 3 acids, 9 esters, 25 ketones, 6 alkenes, 14 heterocycles, and 2 other compounds. A total of 38 volatile compounds were detected in raw chestnuts, including mainly alcohols, ketones, and minor aldehydes, while 95 volatile compounds were detected in thermally processed chestnuts. This meant that new volatile flavor compounds were generated after thermal processing, positively impacted their flavor, and improved their sensory qualities. Predominantly, Maillard reactions, lipid oxidation processes, non-enzymatic oxidation, thermal cracking, and other reactions of glucosides and glycosides generate alcohols, ketones, aldehydes, alkenes, and aromatic hydrocarbons play pivotal roles during thermal sterilization (Krist et al., 2004, Li et al., 2016).

Alcohols commonly originate from the degradation of fatty acid oxides or the reduction of carbonyl compounds, typically possessing a distinctive aromatic odor (Zhou et al., 2023). Chestnuts were rich in unsaturated fatty acids, of which oxidation of linoleic acid generated

short-chain alcohols with a low threshold for flavor activity in chestnuts (Frankel, 1983). Among the alcohols, 3-hexanol, characterized by its wine-like, miscellaneous alcohol flavor, dominated in Raw chestnuts, followed by ethanol with an herbal flavor. After thermal processing treatment, alcohols in raw chestnuts were converted to other compounds such as aldehydes and esters, and their content experienced reduction or even complete elimination, which was consistent with the results reported in previous research (Xu et al., 2023). In addition, a high level of 2-furanmethanol was found in chestnuts after thermal processing, and previous research has also indicated that high-temperature treatment increases 2-furanmethanol content in red ginseng (Lee et al., 2010). There was no significant difference in the content of 2-furanmethanol between nitrogen-packed and air-packed chestnuts, while vacuum-packed chestnuts had the highest content of 2-furanmethanol, which could be attributed to the fact that the vacuum packaging caused some damage to the microstructure of chestnuts, and the Maillard reaction of chestnuts was more intense during the thermal processing treatment (Geng et al., 2024).

Ketones may arise from the enzymatic breakdown of polyunsaturated fatty acids, degradation of amino acids, Maillard reactions, or microbial oxidation, and they can contribute to the floral and fruity sweetness of the flavor profile. Ketones can alter the odor of a product due to their high threshold (Xu et al., 2014). Raw chestnuts contain high concentrations of 3-hexanone and 2-hexanone, where they synergize with other ketones to produce the sweet and fruity flavor of raw chestnuts (Pino et al., 2017). The processing temperature of the product tends to affect its concentration, so the ketone concentration in the chestnuts decreased significantly after thermal sterilization (Gonzalez and Barrett, 2010, Yang et al., 2016).

Aldehydes are primarily produced via lipid oxidation and amino acid degradation and include both straight- and branched-chain aldehydes (Wu and Wang, 2019, Chang et al., 2020). Lower threshold aldehydes with distinct aromas substantially influenced the product's odorous profile, which generally exhibits "fatty", "green", "fruity", and "leguminous" flavors (Moretti et al., 2017). The raw chestnuts displayed a low aldehyde level, with nonanal representing the primary aldehyde (Li et al., 2016). The aldehyde content increased significantly in chestnuts

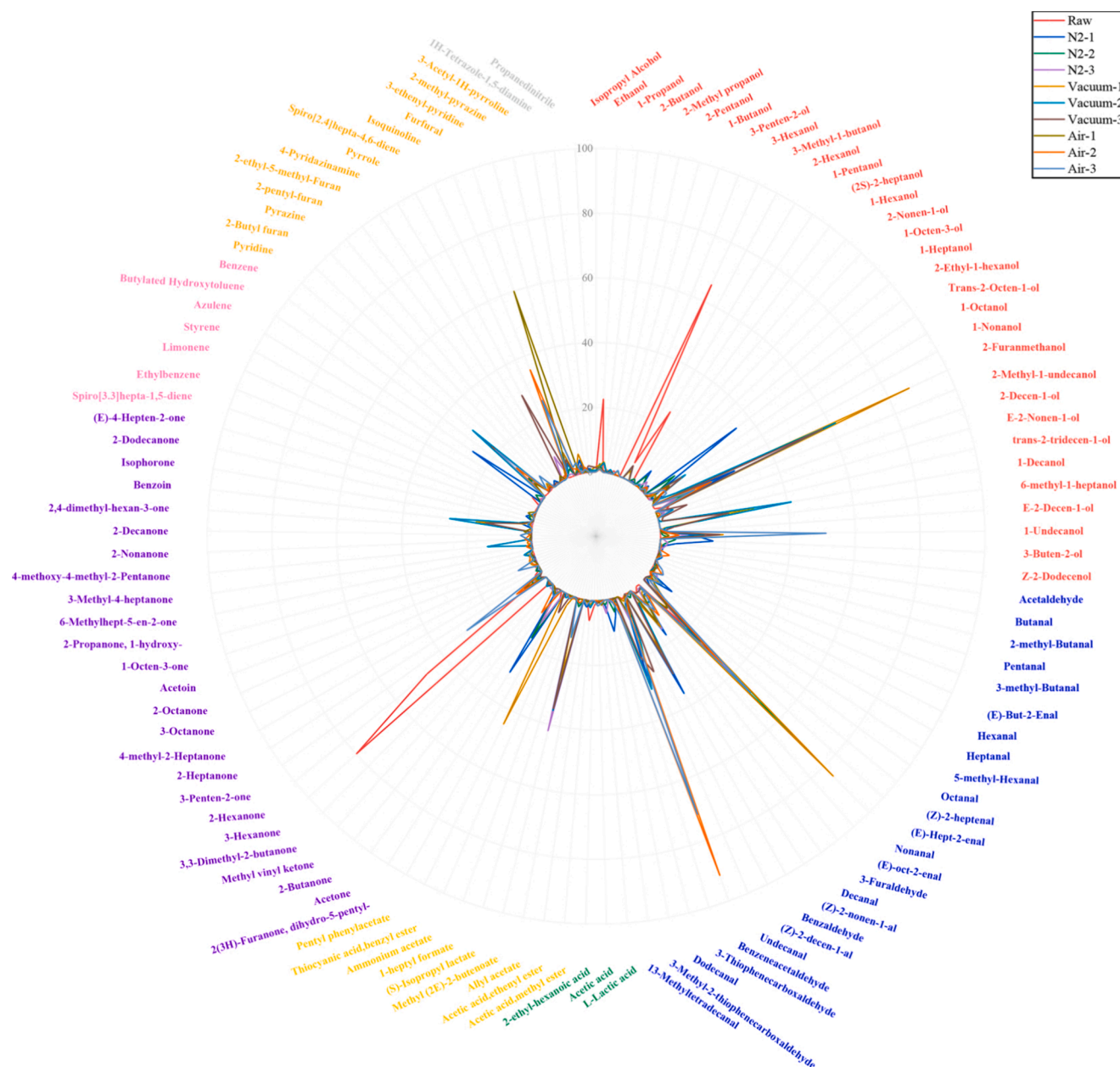


Fig. 3. The characteristic volatile profiles of the volatile compounds in chestnuts by different processing methods were identified by GC×GC-TOF-MS.

after thermal sterilization, yielding straight-chain aldehydes such as hexanal, heptanal, octanal, nonanal, decanal, undecanal, and dodecanal. Straight-chain aldehydes are mainly derived from the oxidative degradation of unsaturated fats, where lipids are oxidized to hydroperoxides, producing different types of hydroperoxides at different oxidation sites, which are then degraded to small molecule volatiles (Zeng et al., 2017). In addition, branched-chain aldehydes such as benzaldehyde, benzeneacetaldehyde, 2-methyl-butanal, 3-methyl-butanal, 5-methyl-hexanal, and 3-furaldehyde were detected, which were mainly derived from the Strecker degradation pathway, involving amino acid deamination and decarboxylation (Shakoor et al., 2022). For instance, benzaldehyde stems from the Strecker degradation of phenylalanine, which has a low odor threshold and can impart a nutty flavor to meat products.

The ester and heterocyclic compound content in the chestnuts increased after thermal processing. The esters mainly resulted from esterification reactions between the free fatty acids and alcohols produced by fat oxidation. Due to their low concentration and high odor

threshold, esters typically offer fruity and floral notes, which have a minimal impact on the chestnut's odor (Zhang et al., 2022). Thiocyanic acid, benzyl ester with almond flavor detected in processed chestnuts. Various heterocyclic compounds were detected among processed chestnuts after thermal sterilization. These are mainly derived from lipid degradation and Maillard reactions of reduced sugars and amino acids during the thermal sterilization process (Morini and Maga, 1995). 2-pentyl-furan was detected in processed chestnuts, air-packed chestnuts had a higher content of 2-pentyl-furan than vacuum-packaged chestnuts, and both were higher than N₂-packaged chestnuts, and chestnuts had a typical nutty aroma at low concentrations and a soybean aroma at high concentrations (Zeng et al., 2007). 2-Pentyl-furan was a secondary oxidation product of lipid hydroperoxides. When single linear oxygen attacked the 9-hydroperoxide of methyl linoleate to produce a cyclic peroxide, the substance was degraded to pentyl furfural, which was then further decomposed to pentyl furan. Therefore, oxygen in air-packaged chestnuts promoted the conversion of lipid hydroperoxides to 2-Pentyl-furan in the heating environment, and due to the slight alteration of the

internal microstructure of vacuum-packaged chestnuts, their internal lipid hydroperoxides were more readily converted to 2-Pentyl-furan than those of N₂-packed chestnuts (Frankel, 1983).

3.3. Multivariate statistical analysis

3.3.1. PCA and clustering heatmap analysis

The processing techniques affected the volatile compounds in the chestnuts differently. To investigate the effect of processing on the volatile flavor of chestnuts, based on the quantitative analysis of detected volatile compounds, the PCA and HCA models were established to analyze the detected compounds, and employed to investigate the similarities and differences among volatile compounds present in the different processing methods of chestnuts. As shown in Fig. 4A, the three principal components accounted for 65.8 % of the total variance via PCA, of which PC1 explained 25.9 %, PC2 denoted 20.7 %, and PC3 represented 19.2 %. This indicated that the PCA model was reliable and provided adequate characterization of flavor information for most of the samples. PCA analysis showed that chestnuts with different processing methods were classified into four categories based on packaging methods. Furthermore, changes in the concentration of different classes of volatile compounds were observed by HCA (Fig. 4B). The hierarchical clustering was executed using Euclidean distance and the Ward algorithm. As demonstrated in the cluster heatmap, the 116 volatile components were categorized into four primary categories based on the packaging method, of which 38, 61, 56, and 71 were detected in the raw, N₂-, vacuum-, and air-packaged chestnuts, respectively (Fig. 4C).

3.3.2. Analysis of potentially characteristic volatile compounds by PLS-DA

Both PCA and clustering heatmap analysis indicated that the packaging method exerted the most significant effect on the chestnut flavor. PLS-DA analysis was conducted to further determine the volatile components responsible for the aroma differences of the chestnuts subjected to different processing treatments. PLS-DA has been widely used in food research for its supervised function as a discriminant analysis method (Botelho et al., 2015). A clear separation was evident between the samples exposed to different packaging methods, which were clustered into four categories (Fig. 5A). A substitution test was conducted to assess the suitability of the PLS-DA model for screening volatile compounds. The results of this test are illustrated in Fig. 5B. 200 permutation test replications were performed to improve PLS-DA model robustness. As seen in Fig. 5B, the intercept between the Q2 regression line was less than 0 with no overfitting (R² = 0.187, Q² = -0.801), confirming model reliability and could be directly used for subsequent discriminant analysis of volatile compounds. The variable importance in the projection (VIP) was calculated, in which volatile compounds with VIP greater than 1 could be used as potential characteristic markers of the samples. A total of 28 volatile compounds (6 aldehydes, 10 alcohols, 5 ketones, 2 esters, 1 alkene, and 4 heterocycles) were identified as potential signature compounds for chestnuts with different processing methods, among which 2-furanmethanol, 3-hexanone, 3-hexanol, benzeneacetaldehyde, 1-decanol, 2-pentyl-furan, 2(3H)-furanone, dihydro-5-pentyl-, and 2-hexanone are the most contributing compounds with VIP values greater than 2.0 (Table S5).

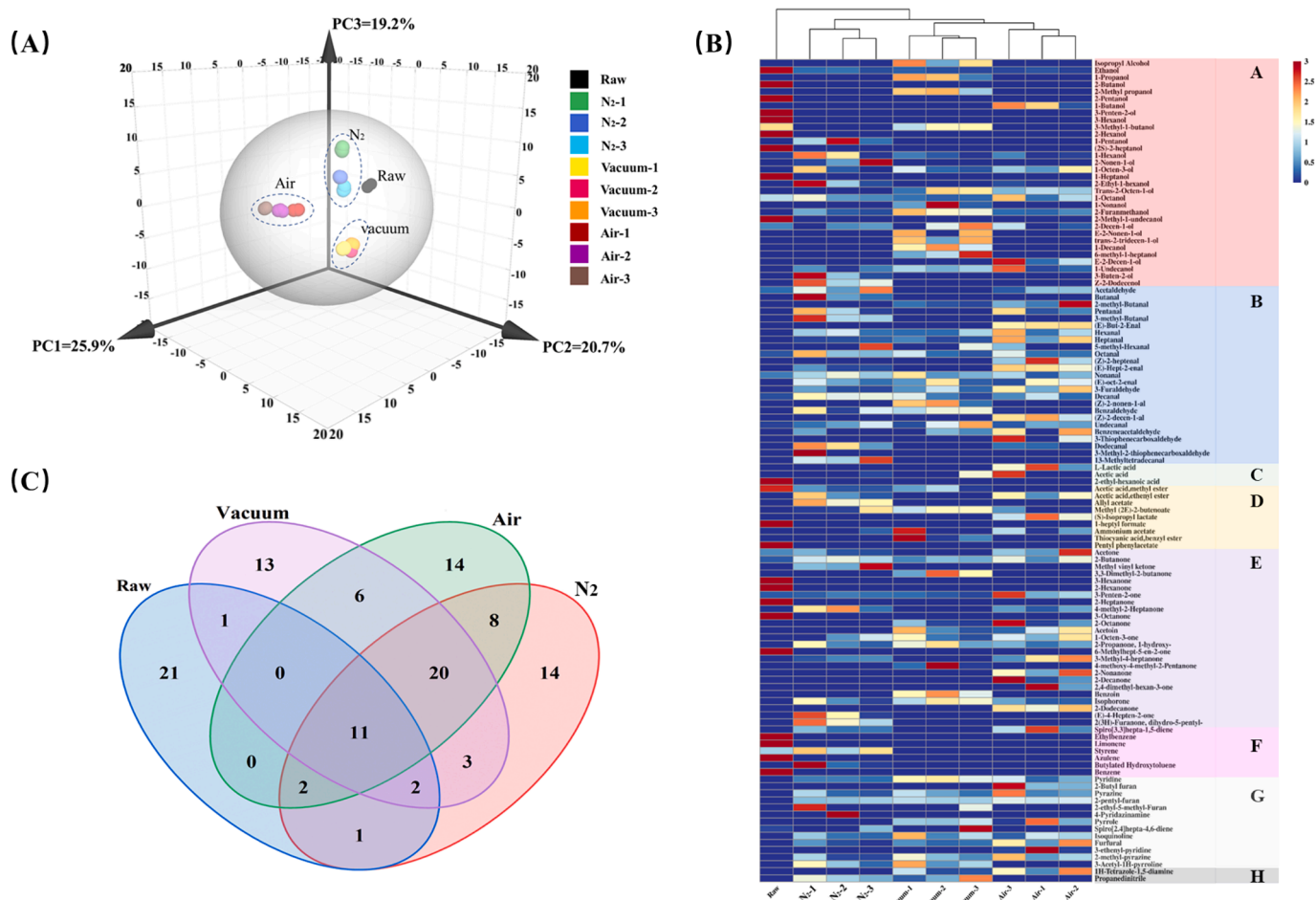


Fig. 4. The volatile compounds identified via GC×GC-TOF-MS in the chestnuts subjected to different processing methods. (A) The PCA analysis of the number of volatile compounds. (B) The heatmap of the volatile compound concentration in the chestnuts exposed to different processing methods. (C) The Venn diagram of the number of volatile compounds.

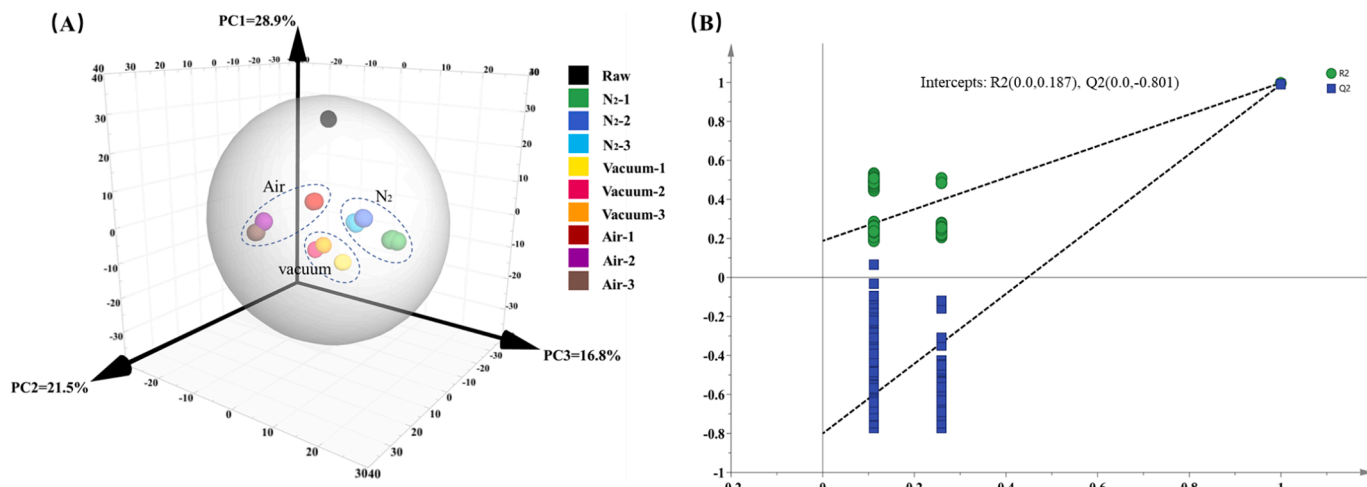


Fig. 5. Effect of different processing methods on flavor components of chestnut. (A) The PLS-DA analysis. (B) The permutation test plot of the PLS-DA model.

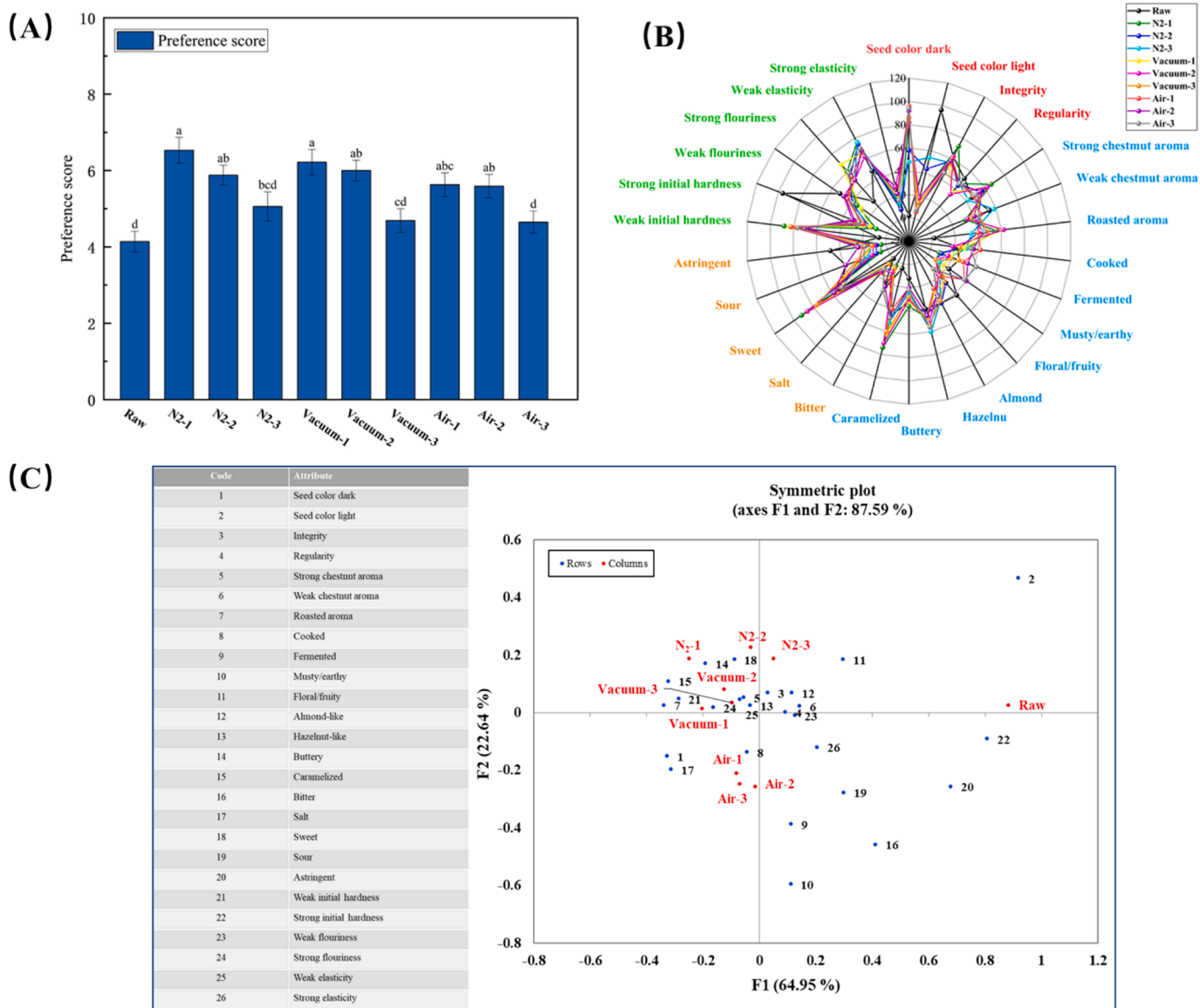


Fig. 6. The CATA analyses of the overall sensory evaluation of the chestnuts exposed to different processing methods. (A) Overall consumer preference score bar chart; (B) Radar chart of CATA method descriptors (citation frequency > 10%); (C) The CA analysis.

3.4. The sensory evaluation using CATA

3.4.1. Overall preference scores

The overall participant preferences for the chestnuts with different processing methods were analyzed via one-way ANOVA by considering the sample a fixed source of variation and consumers as random effects (Fig. 6A). When the mean was found to be significant, the difference was calculated using the Tukey test. The results showed significant variations in the preferences for the chestnuts subjected to different processing methods, indicating that participants could clearly perceive the differences between the samples. Generally, the overall mean preference scores for the N₂- and vacuum-packaged chestnuts were higher than those for the air-packaged chestnuts. This may be because air-packed chestnuts have a darker brown color and a more pronounced bitter taste due to a more intense oxygen-catalyzed Maillard reaction in the surface layer of chestnuts at the same central heat sterilization temperature (Li et al., 2022c). The highest overall mean preference score was obtained from sample N₂-1 (6.53), while the lowest was produced by sample raw (4.14). And a gradual decrease was evident in the preference for the three different chestnut packaging methods as the thermal sterilization procedure increased.

3.4.2. CATA questionnaire

All CATA results were analyzed based on the response frequency to the CATA descriptors. The CATA questionnaire consisted of 27 descriptors. Table 1 shows the usage frequency of each descriptor. The usage frequency of “mustard” flavor was below 10 %, indicating that consumers had difficulty perceiving this flavor in the samples. Therefore, this descriptor was removed from subsequent analyses. The radar chart showed the 26 sensory descriptors of the raw and processed chestnuts and the frequency the assessors selected (Fig. 6B). The

processed chestnuts were visually noticeable due to the “dark seed color”, and in terms of flavor, the “roasted aroma” and “caramelized” attributes were selected significantly frequently compared to the raw chestnuts, mainly due to the Maillard reaction that the chestnuts underwent during the thermal sterilization process. In addition, the frequency of selection for the “fermented” flavor, “musty/earthy”, and “floral/fruity” attributes of processed chestnuts were significantly less than that of raw chestnuts in terms of visual appearance. No consumers perceived the salty flavor in raw chestnuts in terms of taste, and in terms of texture, raw chestnuts were noted for their “strong initial hardness” attribute.

The Cochran Q-test was used to assess whether the descriptors selected by the participants differed significantly between the samples (Meyners et al., 2013). The results (Table 1) showed considerable differences in the frequency of 21 of the 26 terms ($p < 0.05$), indicating that the participants could differentiate between these descriptors in the ten samples. Notably, the selection frequency of descriptors such as “seed color dark”, “roasted aroma”, “caramelized”, and “weak initial hardness” increased significantly for the thermally treated samples compared to the raw chestnuts, representing the primary sensory characteristics identified by the evaluators. This was attributed to the high starch content in the chestnuts and the decomposition of the starch chains after processing, altering the textural properties of the chestnut starch paste and retrogradation (Panda et al., 2015, He et al., 2024). The chestnuts turned brown after thermal processing, mainly due to non-enzymatic reactions, such as the Maillard reaction or caramelization, typically improving the flavor and color (Liu et al., 2023). Moreover, the textural properties of chestnuts were correlated with their moisture content, while the springiness, chewiness, and hardness increased significantly as the moisture content decreased (Yi et al., 2024).

CA is a multivariate graphical technique designed specifically for

Table 1

The frequency of attributes selected (CATA) by the panel (n = 102) of the chestnuts exposed to different processing methods.

| Attributes | Raw | N ₂ -1 | N ₂ -2 | N ₂ -3 | Vacuum-1 | Vacuum-2 | Vacuum-3 | Air-1 | Air-2 | Air-3 |
|--|-----|-------------------|-------------------|-------------------|----------|----------|----------|-------|-------|-------|
| Appearance | | | | | | | | | | |
| Seed color dark ^{***} | 2 | 82 | 58 | 48 | 92 | 82 | 86 | 96 | 92 | 94 |
| Seed color light ^{***} | 96 | 18 | 44 | 54 | 10 | 18 | 14 | 6 | 8 | 8 |
| Integrity ^{***} | 62 | 72 | 58 | 56 | 54 | 62 | 56 | 60 | 58 | 46 |
| Regularity ^{***} | 52 | 46 | 44 | 46 | 36 | 34 | 46 | 46 | 46 | 46 |
| Aroma | | | | | | | | | | |
| Strong chestnut aroma ^{ns} | 46 | 66 | 56 | 42 | 62 | 62 | 54 | 48 | 54 | 50 |
| Weak chestnut aroma ^{ns} | 54 | 32 | 44 | 58 | 36 | 34 | 44 | 46 | 40 | 44 |
| Roasted aroma ^{***} | 2 | 56 | 40 | 34 | 60 | 62 | 48 | 40 | 44 | 46 |
| Cooked ^{ns} | 22 | 30 | 24 | 30 | 28 | 18 | 24 | 42 | 36 | 34 |
| Fermented ^{***} | 26 | 8 | 10 | 18 | 18 | 16 | 26 | 30 | 34 | 42 |
| Musty/earthy ^{***} | 22 | 8 | 8 | 12 | 16 | 8 | 8 | 38 | 40 | 38 |
| Floral/fruity ^{**} | 42 | 20 | 28 | 20 | 26 | 18 | 26 | 20 | 12 | 12 |
| Almond-like ^{ns} | 40 | 26 | 40 | 38 | 28 | 32 | 38 | 26 | 32 | 34 |
| Hazelnut-like | 40 | 48 | 44 | 60 | 48 | 52 | 54 | 46 | 46 | 56 |
| Buttery ^{ns} | 12 | 36 | 32 | 24 | 32 | 30 | 28 | 20 | 20 | 22 |
| Caramelized* | 4 | 74 | 46 | 52 | 62 | 70 | 60 | 40 | 48 | 48 |
| Taste | | | | | | | | | | |
| Bitter ^{***} | 28 | 4 | 8 | 6 | 8 | 10 | 6 | 16 | 24 | 28 |
| Salt* | 0 | 6 | 12 | 6 | 16 | 14 | 6 | 16 | 14 | 12 |
| Sweet ^{***} | 52 | 92 | 86 | 76 | 78 | 86 | 76 | 58 | 58 | 50 |
| Sour ^{***} | 38 | 6 | 10 | 18 | 22 | 14 | 28 | 26 | 38 | 22 |
| Astringent ^{***} | 48 | 4 | 8 | 12 | 12 | 10 | 12 | 20 | 24 | 18 |
| Texture | | | | | | | | | | |
| Weak initial hardness ^{***} | 6 | 88 | 84 | 84 | 84 | 70 | 78 | 82 | 70 | 76 |
| Strong initial hardness ^{***} | 96 | 10 | 18 | 16 | 16 | 30 | 24 | 22 | 32 | 26 |
| Weak flouriness ^{ns} | 52 | 34 | 40 | 44 | 30 | 46 | 44 | 44 | 48 | 38 |
| Strong flouriness ^{**} | 26 | 60 | 56 | 50 | 68 | 50 | 50 | 54 | 50 | 58 |
| Weak elasticity* | 48 | 76 | 74 | 76 | 66 | 62 | 68 | 66 | 68 | 70 |
| Strong elasticity* | 30 | 10 | 8 | 14 | 20 | 28 | 22 | 22 | 22 | 16 |

Note: Significant difference for * $p < 0.05$, ** $p < 0.01$, *** $p < 0.001$.

analyzing categorical variables, and its value is most evident when applied to nominal variables (Sourial et al., 2010). Fig. 6C shows the results of the 10 samples assessed using the CA, with one- and two-dimensions accounting for 64.95 % and 22.64 % of the total variance, respectively, and a cumulative variance contribution of 87.59 %. The raw chestnuts were distinctly differentiated from the processed samples. The raw chestnuts were positively correlated with “seed color light”, “astringent”, and “strong initial hardness”, mainly due to the that the presence of phenolic acids, flavonoids, and tannins provided the chestnuts with a distinctly bitter, astringent flavor and inhibited the perception of sweetness (Santos et al., 2022). The various flavors were related to the free fatty acid, reducing sugar, and nucleotide content (Tseng et al., 2005).

Different thermal processing procedures changed the levels of these substances in the chestnuts, modifying the senses of each sample (Sashikala et al., 2015). In the processed chestnut, the Maillard reaction between reducing sugars and amino acids is known to be a major factor in the formation of volatile compounds during thermal processing (Morini and Maga, 1995). Furthermore, non-enzymatic browning caramelization, which generates an enolic intermediate and a final dehydration product when sugar is heated above its fusion temperature, also produces rich colors and flavors in high-sugar foods during processing (Croguennec, 2016). The N₂- and vacuum-packaged chestnuts were mainly located in the second quadrant, where “strong chestnut aroma”, “roasted aroma”, “buttery”, “almond”, “hazelnut”, “caramelized”, and “sweet” sensory attributes were evident in both groups, which probably due to phenolic acid degradation via continuous thermal treatment at high temperatures that decreased the content levels, increased the perception of sweetness, and enriched the flavor of the chestnuts. The air-packaged chestnuts were located in the third quadrant due to their positive correlation with sensory attributes such as “seed color dark”, “cooked”, “salty”, and “fermented”, which was probably due to the presence of oxygen increasing the rate of the Maillard reaction in air-packaged chestnuts. In addition, the fermented flavor of air-packed chestnuts began to appear, suggesting that air-packing was not conducive to the shelf-life of chestnuts (Yang et al., 2019).

The preference ratings revealed that consumers preferred processed chestnuts, and the CATA results showed that the chestnut sensory changed with the packaging method. Thermal sterilization can enrich

the sensory quality of processed chestnuts. The preference ratings and CATA results indicated that the sensory attribute change caused by the Maillard volatility of chestnuts after thermal processing is widely preferred by consumers.

3.5. The sensory and chemical data correlation analysis

To further analyze the differences in the sensory quality of chestnuts in different processed methods, Pearson’s correlation analysis between sensory evaluation (CATA attributes) and instrumental analyses (textural indices and volatile compounds) was established. From the results of this correlation analysis. The correlation network diagram (Fig. 7A) displayed the correlations between all sensory-related indicators of chestnuts. As seen in Fig. 7A, the overall preference of chestnuts in different processing methods was positively correlated with “caramelized”, “buttery”, “roasted aroma”, “sweet”, “strong chestnut aroma”, “weak initial hardness”, and “strong flouriness”, which indicated that a series of physicochemical changes occurring in chestnuts during the thermal processing process enriched the organoleptic attributes of the chestnuts and increased the preference of consumers. Visually, chestnut color intensity was positively correlated with 2-pentyl-furan, “roasted aroma”, and “weak initial hardness”. In terms of texture, “weak initial hardness” was positively correlated with adhesiveness, cohesiveness, gumminess, springiness, the strength of “elasticity” and “flouriness” was negatively correlated with the strength of initial hardness, and “regularity” attributes were significantly positively correlated with chewiness. The results indicated that the rich physicochemical reactions during the thermal processing of chestnuts enhanced the glutinous taste quality of chestnuts (He et al., 2024), and the appropriate amount of melanin-like substances formed by the Maillard and caramelization reactions were effective in improving the organoleptic properties of the foodstuffs (Nooshkam et al., 2019).

The relationship between the sensory attributes (frequencies) and volatile compositions was explored via PLSR-based modeling. The correlation between the CATA flavor attributes (Y-variables) and different volatile components with VIP > 1 (X-variables) was analyzed based on correlation network analysis in combination with PLSR (Fig. 7B). In Fig. 7B, PC1 and PC2 well explained 74.7 % of cross-validation variance in the X-variables and 66.3 % in the Y-variables. As shown in the PLSR

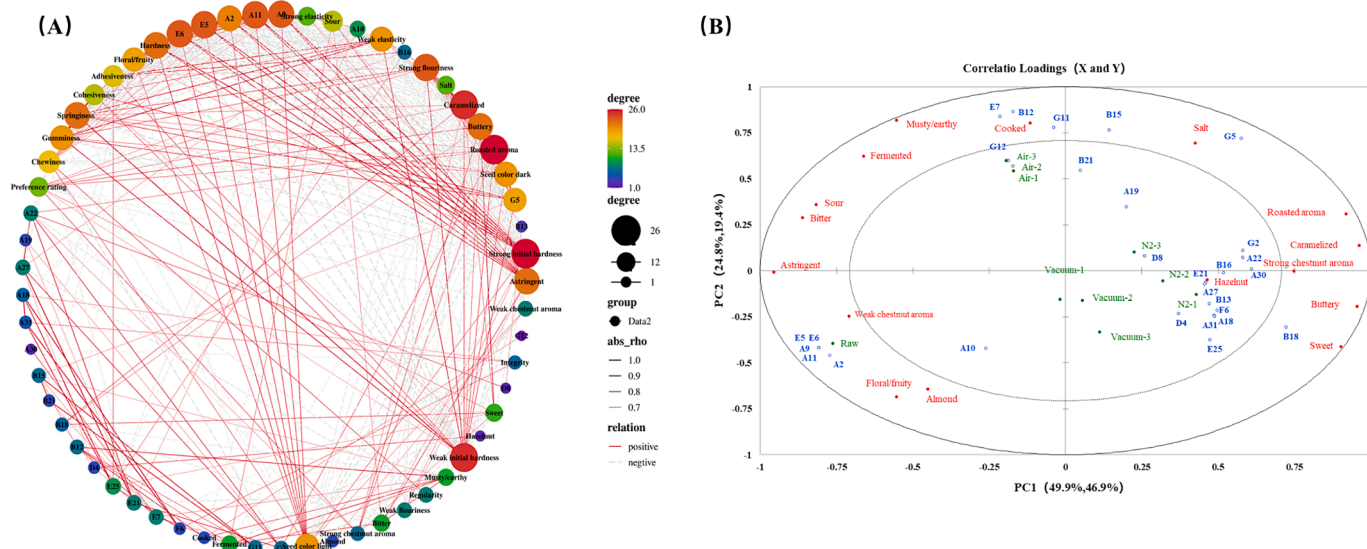


Fig. 7. (A) The visualization network between textural indicators, volatile compounds and sensory descriptors (the red and grey lines indicated positive and negative correlations, respectively, the thickness of the lines signified the correlation strength, and the dot sizes and color shades indicated the number of relevant targets). (B) The PLSR correlation loading plot. The inner and outer ellipses in the plot indicate 50 % and 100 % explained variances. (X represents the key compound differences in the chestnuts with VIP > 1 values after exposed to the different processing methods as detected via GC×GC-TOF-MS. Y denotes the sensory descriptions of the CATA aroma and taste in the chestnuts.) (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

analysis, the CATA sensory attributes of “weak chestnut aroma”, “floral/fruity” and “almond” were characteristic flavors of raw chestnut, and were highly correlated with ethanol (A2), 3-hexanol (A9), 2-hexanol (A11), 3-hexanone (E5) and 2-hexanone (E6). In addition, raw chestnuts exhibited an astringent flavor, consistent with the results of the CA analysis. The flavor profiles of N₂- and vacuum-packaged chestnuts were similar. Vast volatile compounds such as thiocyanic acid, benzyl ester (D8), methyl(2E)-2-butenate (D4, green, fruity), 1-decanol(A27, sweet, floral), nonanal (B13, rose, orange peel), butylated hydroxytoluene (F6), 2-Ethyl-1-hexanol (A18), 3-Buten-2-ol (A31), 2(3H)-Furanone, dihydro-5-pentyl- (E25), pyridine (G2, spicy), 2-furanmethanol (A22, sweet, caramel), benzoin (E21), 1-undecanol (A30, floral, sweet, fruity) were covaried with the CATA attributes of “hazelnut”, “roasted aroma”, “caramelized”, “strong chestnut aroma”, “buttery”, and “sweet”, indicating their high correlation. While the “floral/fruity”, and “cooked” CATA sensory attributes in the air packaged chestnuts were highly correlated with 3-ethenyl-pyridine (G12), 3-Penten-2-one (E7, honey), (E)-Hept-2-enal (B12), furfural (G11, sweet, woody), 3-furaldehyde (B15), benzeneacetaldehyde (B21, green, sweet, floral). Thermal processing stimulated the organoleptic attributes of the chestnuts. The heating process catalyzed the Maillard reaction between amino acids and sugars, which developed the product color and volatile heterocyclic compounds (Zhang et al., 2018). N₂ and vacuum packaging decreased the catalytic effect of air on the physical and chemical reactions (Maillard reaction, lipid oxidation and degradation, oxidized lipid, and amino acid interaction, and long-chain compound degradation) during thermal processing, while adverse reactions were reduced to a certain extent, and the organoleptic quality of chestnuts displayed a good protective effect.

4. Conclusions

This study aims to explain the chemical basis of the sensory properties of chestnuts after different processing treatments. Instrumental analysis is combined with sensory evaluation to comprehensively evaluate the sensory chestnut quality. The sensory qualities of chestnuts in different packaging methods were significantly different after thermal sterilization, but the differences in sensory qualities of chestnuts in the same packaging methods with different thermal sterilization procedures were not significant. The combination of texture analysis, HS-SPME-GC×GC-TOF-MS volatile fingerprinting, and CATA analysis were effective in distinguishing chestnuts with different packaging methods. HS-SPME-GC×GC-TOF-MS identified 116 volatile compounds from chestnuts with different processing methods, and PLS-DA analysis highlighted 28 compounds associated with the characteristics of differently processed chestnuts. Significant differences are evident in the frequency of 21 CATA questionnaire terms ($p < 0.05$), the participants can distinguish between these descriptors in the 10 samples. The overall consumer preference scores of chestnuts were significantly enhanced after processing, and the overall sensory quality of chestnuts from N₂-1 and Vacuum-1 was better than that of chestnuts from other processing treatments. The results of this study provide vital information for improving the sensory quality and acceptance of chestnuts subjected to different processing methods and promoting the development of the chestnut industry.

CRediT authorship contribution statement

Kunli Xu: Conceptualization, Data curation, Investigation, Methodology, Software, Writing – original draft, Writing – review & editing. **Kexin Jiang:** Conceptualization, Data curation, Writing – review & editing. **Aolin Yang:** Conceptualization, Data curation, Writing – review & editing. **Zheting Zhang:** Data curation, Methodology. **Zhengyu Lin:** Data curation, Methodology. **Tielong Wang:** Formal analysis, Writing – review & editing. **Lingyun Xu:** Formal analysis, Writing – review & editing. **Fanyu Meng:** Methodology, Writing – review & editing. **Bei**

Wang: Conceptualization, Funding acquisition, Supervision, 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.

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Appendix A. Supplementary data

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