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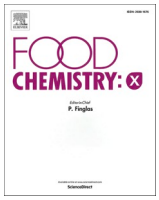

Effect of thermal treatments on volatile profiles and fatty acid composition in sweet corn (*Zea mays* L.)

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ABSTRACT

This study analyzed the effects of thermal processing on volatiles and fatty acids in sweet corn. There were 27 volatiles measured in fresh samples, and 33, 21, and 19 volatiles identified in the steaming, blanching, and roasting groups, respectively. Relative odor activity values (ROAVs) showed that characteristic aroma-active volatiles of sweet corn after thermal treatments included: (E)-2-nonenal, 1-octen-3-ol, beta-myrcene, dimethyl trisulfide, 1-(4,5-dihydro-2-thiazolyl)-ethanone, and D-limonene. Thermal treatments significantly increased the unsaturated fatty acids (oleic acid and linolenic acid) of sweet corn by 110 to 183% compared to fresh samples. Meanwhile, many characteristic volatiles were found that derived from the oxidative cleavage of fatty acids. The sweet corn aroma obtained by steaming for 5 min was considered the closest to fresh corn. Our research provided insight into aroma composition of different thermally processed sweet corn and laid the foundation for further exploring the sources of aroma compounds in thermally processed sweet corn.

1. Introduction

Corn is one of the most productive food crops in the world. The spontaneous mutation of the su ("sugary") gene in maize led to the emergence of sweetness in maize, namely the birth of sweet corn (Revilla et al., 2021). Fresh sweet corn originated in the South America and has since taken the world by storm due to its unique nutritional and flavor profile. Sweet corn has been commonly used as raw material in the food industry. Sweet corn can be eaten fresh or in a variety of processed forms, for example steaming, blanching and roasting, which can play essential roles in sterilization to prolong the storage time of sweet corn. Meanwhile, the aroma of sweet corn inevitably changes after thermal processing. For example, steaming and roasting often give sweet corn a more attractive sweet aroma and a more delicious taste (Chen, et al., 2022).

Aroma is a very popular trait of cooked sweet corn, but the cooked odor and odor intensity are the most challenging feature to describe in sweet corn taste panels (Becerra-Sanchez & Taylor, 2021). The critical volatiles that make up the aroma of heat-treated sweet corn contribute to further interpretation of the relevant characteristics. After different treatments, including blanching, steaming, frying, and freeze-drying, decylaldehyde, 1-octene-3-ol, and 2-pentylthiazole are found that can be the influential aroma compounds in corn (Zhang et al., 2022). Different drying ways also have an evident impact on the volatile compounds of sweet corn, in which thermal treatments can form more aromatic components and increase the aroma of sweet corn (Yao, et al., 2022).

Volatiles serve as essential determinants of the overall aroma properties of food and tend to impact human preferences or aversions to taste. Combinations of volatiles produce different aromas than expected from individual compounds as well as differences in the perception of volatiles in different matrices (Chambers & Koppel, 2013). Fatty acids produce some volatiles through oxidation and degradation pathways, which in turn influence changes in food aroma. Oxidation of fatty acids usually leads to the formation of off-flavors, which can be improve by heat treatment due to the inactivation of enzymes, for example lipoxygenase (Barrett et al., 2000). The unsaturated fatty acids, which produce most aldehydes, alcohols, and ketones, play a more critical role in forming volatiles than saturated fatty acids (Shahidi & Hossain, 2022). As a grain rich in unsaturated fatty acids, fresh corn may change

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the volatile composition and thus the aroma after harvest due to fatty acid oxidation. Applying heat treatment has become an important way to maintain or improve the aroma of sweet corn.

The change in sweet corn aroma determines the shift in consumer preference and even the consumer market. But now, investigations into the aroma characteristics of thermal treated sweet corn are limited. Therefore, the present investigation aims to explore the changes in volatile profiles and fatty acid composition of sweet corn under different thermal treatments. Gas chromatography-mass spectrometry (GC–MS) is employed to determine sweet corn volatiles and fatty acids. Relative odor active values (ROAVs) of volatiles are calculated to screen for aroma volatiles that make a significant contribution to sweet corn aroma. This research reveals the influence of thermal treatments on sweet corn aroma and lays a foundation for obtaining the optimal processing mode of quality sweet corn aroma.

2. Methods and materials

2.1. Chemicals

Sodium chloride (NaCl) was purchased from Sangong Biotech Co., Ltd (Shanghai, China). Analytical grade pure *n*-hexane and ethyl acetate were purchased from Tianjin Fuyu Fine Chemical Co., Ltd (Tianjin, China). The analytical reagent toluene and 10% boron trifluoride-methanol solution were obtained separately from Guangzhou Chemical Reagent Factory (Guangzhou, China) and Shanghai Macklin Biochemical Technology Co., Ltd (Shanghai, China). The S-(+)-2-octanol (98%) and heptadecanoic acid (98%) were purchased from Shanghai Yuanye Biotechnology Co., Ltd (Shanghai, China). *n*-Alkane mixed standard (C7-C30) of chromatographic grade was purchased from Sigma Aldrich (St. Louis, MO, USA).

2.2. Thermal processes of sweet corn

The fresh kernels of sweet corn (variety YT28) were provided by the Crop Research Institute of Guangdong Academy of Agricultural Sciences (Guangzhou, China). A total of 60 cobs of sweet corn were harvested, and 1800 g of kernels were obtained after harvesting and divided evenly into nine portions of 200 g of kernels each. And then the kernels were treated by three thermal treatments including steaming, blanching, and roasting. Steaming treatments were divided into three groups, including 100 °C for 5 min (S-5), 10 min (S-10), and 15 min (S-15). Blanching treatments were divided into three groups, including 100 °C for 1 min (B-1), 3 min (B-3), and 5 min (B-5). Roasting treatments were also divided into three groups, including 180 °C for 5 min (R-5), 8 min (R-8), and 10 min (R-10). Fresh sweet corn kernels (Fresh) without thermal treatment were set as the control in this study. All the samples were immediately frozen with liquid nitrogen after heat treatments and stored at −20 °C until analysis.

2.3. The determination of volatiles

The determination of volatiles was carried out by headspace solid phase microextraction combined with gas chromatography-mass spectrometry (HS-SPME-GC–MS) according to the previous report with modification (Xiang et al., 2022). Three grams of freshly ground sample was weighed in a 20 mL headspace bottle and equilibrated at 60 °C for 15 min. Then, volatiles were adsorbed by a 50/30 μm of ACAR/PDMS/DVB extraction fiber (Qingdao Zhenzheng Analytical Instrument Co., Ltd, China) for 45 min and desorbed for 4 min. An HP-INNOWAX column (30 m × 250 μm × 0.25 μm, Agilent Technologies, Palo Alto, CA, USA) was used to separate volatiles, which were analyzed by a gas chromatography system (GC, Agilent Technologies 7890B, Palo Alto, CA, USA) equipped with a triple quadrupole-MS (TQ-MS, 7000C GC/MS Triple Quad, Agilent Technologies, Palo Alto, CA, USA). The temperature program was as below: 50 °C for 3 min, increased to 100 °C at 5 °C·min^{-1}, kept at 100 °C for 5 min, and then raised to 150 °C at 5 °C·min^{-1} and held for 3 min, finally increased to 250 °C at 20 °C·min^{-1} and keep at 250 °C for 4 min. As for the mass spectrometry conditions, the electronic energy of the EI mode was 70 eV with 33–400 *m/z* of the mass scan range, 230 °C of the ion source temperature, and 150 °C of the quadrupole temperature. S-(+)-2-Octanol was selected as internal standard. Each compound was qualitatively matched against the NIST 20 standard mass spectrometry library (Agilent Technologies G1033A, Palo Alto, CA, USA).

Furthermore, the retention index (RI) value was compared with those reported in the literature and was used to confirm selected compounds. The RI value was calculated using Eq. (1):

$$RI = 100^{*}N + 100^{*}\frac{R_t(X) - R_t(N)}{R_t(N+1) - R_t(N)} \quad (1)$$

where R_t (X) is the retention time of the chromatographic peak of the analyzed compound, R_t (N) and R_t (N + 1) are the retention time of the chromatographic peaks of *n*-alkanes, with carbon number N and N + 1, before and after the outflow of the target compound (X).

According to the method of Rotsatchakul et al. (2007) (Rotsatchakul et al., 2008), the concentrations of the volatile compounds were determined using Eq. (2):

$$C_i = C_{is} \times \frac{A_i}{A_{is}} \times f_i \quad (2)$$

where C_i and A_i are the concentration and peak area of compound i, respectively, with the concentration and peak area of the internal standard being C_{is} and A_{is}. As the correction factor of compound i, f_i was calculated using quantitative ion peak area of internal standard and compound i standard. The data were reported as ng·g^{-1} FW mean ± SD (n = 3).

2.4. Relative odor activity value (ROAV)

The relative odor activity value (ROAV) of all the volatile compounds obtained from sweet corn were calculated to evaluate the contribution of individual compounds to the overall aroma using Eq. (3)(Zhang et al., 2022):

$$ROVA_i = \frac{C_i}{T_i} \times \frac{T_{max}}{C_{max}} \times 100 \quad (3)$$

where C_i is the relative concentration of the volatile compound, T_i is the odor threshold of the volatile compound to be measured in water (mg/kg), and T_{max} and C_{max} are the maximum of C_i/T_i among all the compounds in the sample. The odor threshold of each volatile compound was accessed from literature data reported by Gemert (Gemert, 2011).

The range of ROAV is from 0 to 100. Volatile compounds with ROAV $\geq$ 1 are considered key odor compounds, with volatile compounds greater than 0.1 and $<$ 1 have a modifying effect on aroma, and volatile compounds $<$ 0.1 are potential aroma compounds.

2.5. The extraction and determination of fatty acids

The extraction and determination methods of fatty acids were performed refer to the reported study with modification (Li et al., 2022). Sweet corn kernels were powdered with liquid nitrogen, weighed four grams and extracted three times with 10 mL of *n*-hexane/ethyl acetate (9:1, v/v). The 1 mL supernatant was dried in a vacuum concentrator and then mixed with 250 μL of toluene and 1250 μL of boron trifluoride (BF_3)/methyl alcohol solution (10 g BF_3 dissolved in 100 mL methyl alcohol). Then, after reacted at 90 °C for 60 min, the mixtures were added with 800 μL of distilled water and 1 mL of *n*-hexane for analysis. The extracts were stored at −20 °C until determination.

A 0.4 μL sample was injected and analyzed by GC–MS using a gas chromatography system (GC, Agilent Technologies 7890B, Palo Alto, CA, USA) equipped with a triple quadrupole-MS (TQ-MS, 7000C GC/MS

Triple Quad, Agilent Technologies, Palo Alto, CA, USA). The temperature program of capillary column (HP-INNOWAX, 30 m × 250 μm × 0.25 μm, Agilent Technologies, Palo Alto, CA, USA) was 100 °C for 5 min, increased to 250 °C at 5 °C·min^{-1} and kept at 250 °C for 10 min. The mass spectrometry conditions were as follows: the ion source temperature was 230 °C, the quadrupole temperature was 150 °C, and the electronic energy of the EI mode was 70 eV with 33–500 *m/z* of the mass scan range. Similarly, to the volatiles, the concentrations of the fatty acid compounds were also determined by Eq. (2). The results were expressed as μg·g^{-1} FW mean ± SD (n = 3).

2.6. Statistical analysis

Each sample was analyzed in triplicate and performed as mean ± SD/SE. Agilent NIST 20 was used to identify fatty acids and volatile compounds. The partial least-squares discriminant analysis (PLS-DA) and heatmap were plotted via MetaboAnalyst 5.0 and the online website (http://www.bioinformatics.com.cn) with modification, respectively. The line charts, bar charts and stacking area maps were plotted by Origin 2018 (Origin Lab Corporation, Northampton, MA, USA). Significance tests were performed using one-way analysis of variance (ANOVA) followed by Duncan's test via SPSS 25 software (IBM Corp., Armonk, NY, USA) and evaluated at $p < 0.05$.

3. Results

3.1. Change of volatile profiles on sweet corn after thermal processing

A total of 43 volatiles in sweet corn YT28 were detected by HS-SPME-GC–MS (Table S1), and they were broadly classified into 10 categories (Fig. 1). Meanwhile, the ROAVs of characteristic volatile compounds were calculated, as presented in Table 1, to evaluate the aroma potency of odorants in raw and cooked sweet corn.

27 volatile components were measured in fresh samples, which could be divided into 9 types, including 7 aldehydes, 6 alcohols, 3 esters, 2 acids, 3 ketones, 2 terpenes, 2 furans, 1 hydrocarbon, and 1 anhydride. In the first place of volatiles were aldehydes (42.91%), dominated by hexanal (338.3 ± 15.8 ng·g^{-1} FW) and 5-ethylcyclopent-1-enecarboxaldehyde (518.7 ± 18.8 ng·g^{-1} FW). Alcohols (18.99%) and acids (12.38%) ranked second and third, where the main components were 1-pentanol (143.4 ± 1.1 ng·g^{-1} FW), 1-hexanol (142.3 ± 11.0 ng·g^{-1} FW), 1-octen-3-ol (146.7 ± 8.5 ng·g^{-1} FW), and hexanoic acid (295.2 ± 34.9 ng·g^{-1} FW), respectively. Additionally, (E)-2-nonenal, 1-octen-3-ol, and hexanal were the top three highest ROAVs in F with 100, 17.22 ± 0.87,

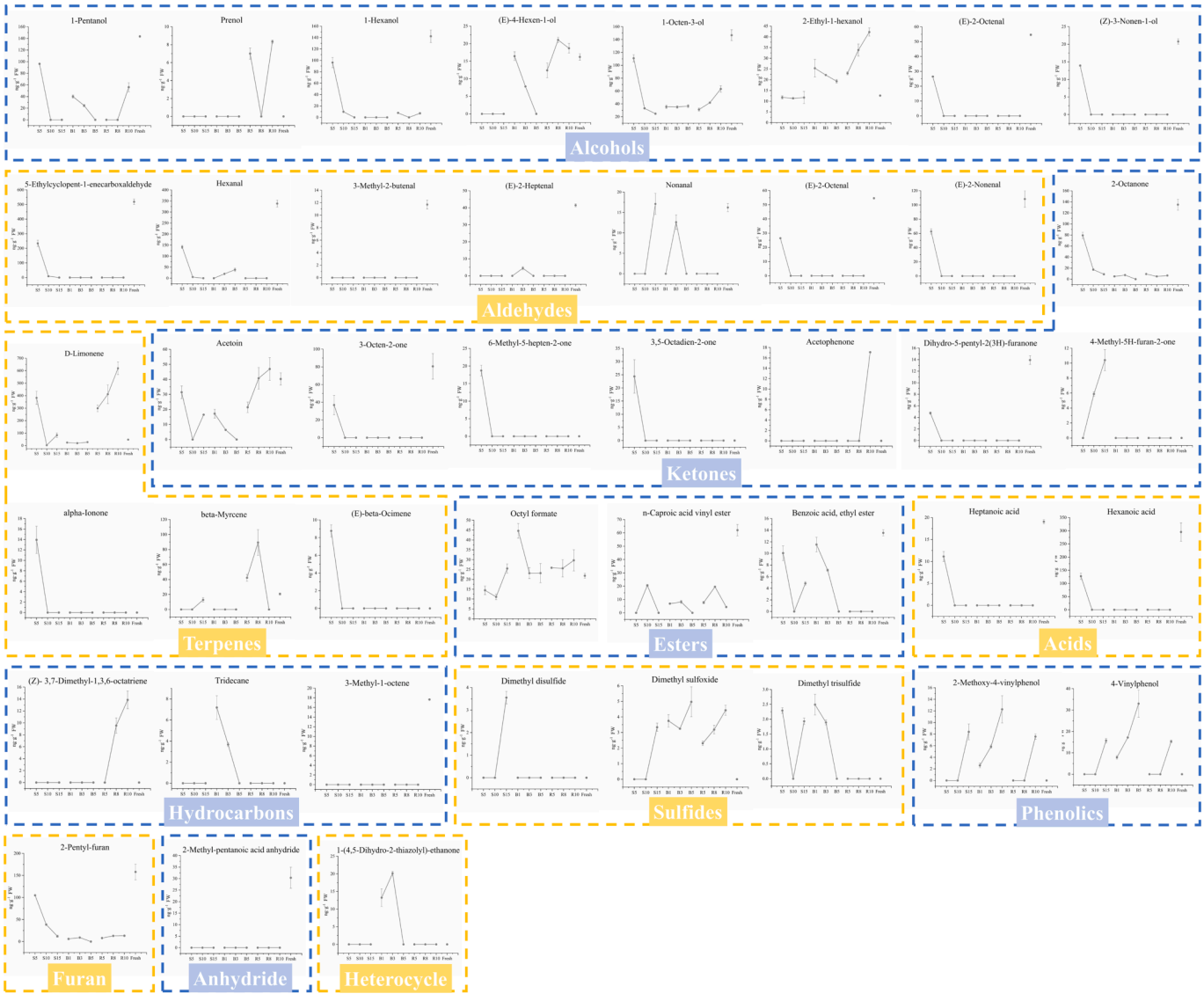

Fig. 1. Varied contents of volatiles in sweet corn after different thermal treatments (mean ± SD, n = 3).

Table 1
Aroma-active compounds identified by rOAVs in sweet corn after different thermal treatments.

NO.	Volatile composition	T_i(mg/kg)	Odor description	Fresh	S5	S10	S15	B1	B3	B5	R5	R8	R10
C1	Dimethyl disulfide	0.003	Cooked, cabbage-like	ND	ND	ND	6.13 ± 0.25	ND	ND	ND	ND	ND	ND
C2	Hexanal	0.005	Grassy, green, fresh	11.67 ± 1.12	8.80 ± 0.48	5.55 ± 0.38	ND	ND	16.83 ± 0.27	28.26 ± 2.19	ND	ND	ND
C3	beta-Myrcene	0.0012	Pine-like, green, grassy	2.90 ± 0.30	ND	ND	57.13 ± 2.54	ND	ND	ND	100	100	ND
C4	D-Limonene	0.034	Citrus-like, fruity	0.246 ± 0.026	3.12 ± 0.30	0.407 ± 0.022	11.26 ± 0.94	3.14 ± 0.23	2.40 ± 0.26	3.32 ± 0.34	22.59 ± 2.00	21.59 ± 0.71	43.21 ± 0.13
C5	3-Methyl-2-butenal	0.5	Fruity, doughy	< 0.1	ND	ND	ND	ND	ND	ND	ND	ND	ND
C6	2-Pentyl-furan	0.0058	Earthy, beany	4.60 ± 0.436	5.50 ± 0.35	30.18 ± 0.24	11.91 ± 1.16	4.24 ± 0.38	6.78 ± 0.75	ND	3.63 ± 0.28	3.34 ± 0.33	5.45 ± 0.09
C7	(Z)- 3,7-Dimethyl-1,3,6-octatriene	0.034	sweet	ND	ND	ND	ND	ND	ND	ND	ND	0.415 ± 0.046	0.965 ± 0.028
C8	(E)-beta-Ocimene	0.034	Herb, floral	ND	< 0.1	ND	ND	ND	ND	ND	ND	ND	ND
C9	1-Pentanol	0.1502	Fermented, solvent	0.169 ± 0.020	0.195 ± 0.014	ND	ND	1.08 ± 0.07	0.698 ± 0.016	ND	ND	ND	0.791 ± 0.076
C10	2-Octanone	0.0502	Fatty, green, floral, fruity	0.473 ± 0.017	0.459 ± 0.028	1.57 ± 0.19	0.768 ± 0.027	0.456 ± 0.091	0.629 ± 0.004	ND	0.489 ± 0.032	0.132 ± 0.016	0.312 ± 0.003
C12	Acetoin	0.014	Buttery-like	0.512 ± 0.107	0.754 ± 0.074	ND	6.12 ± 0.421	4.52 ± 0.55	1.95 ± 0.07	ND	4.37 ± 0.10	3.67 ± 0.09	7.87 ± 0.10
C13	(E)-2-Heptenal	0.0028	Fatty, fruity	2.61 ± 0.227	ND	ND	ND	ND	6.66 ± 0.18	ND	ND	ND	ND
C16	6-Methyl-5-hepten-2-one	2	Fruity, floral	ND	< 0.1	ND	ND	ND	ND	ND	ND	ND	ND
C17	1-Hexanol	0.0056	Green, pungent	4.35 ± 0.58	5.41 ± 0.58	8.04 ± 0.33	ND	ND	ND	ND	4.04 ± 0.45	ND	3.32 ± 0.27
C18	Dimethyl trisulfide	0.0001	Savory, green onion	ND	6.98 ± 0.71	ND	100	100	80.84 ± 6.21	ND	ND	ND	ND
C20	Nonanal	0.0011	Waxy, citrus	2.39 ± 0.19	ND	ND	80.31 ± 8.29	ND	48.95 ± 4.23	ND	ND	ND	ND
C23	(E)-2-Octenal	0.003	Fatty, green, cucumber	3.22 ± 0.38	2.68 ± 0.20	ND	ND	ND	ND	ND	ND	ND	ND
C24	1-Octen-3-ol	0.0015	Mushroom-like, earthy	17.22 ± 0.87	22.42 ± 0.23	100	85.61 ± 2.02	95.37 ± 6.46	100	100	53.89 ± 4.47	41.62 ± 4.55	100
C26	2-Ethyl-1-hexanol	0.3	Floral, sweet	< 0.1	< 0.1	0.171 ± 0.0001	0.177 ± 0.017	0.338 ± 0.009	0.317 ± 0.019	0.265 ± 0.028	0.220 ± 0.031	0.167 ± 0.016	0.336 ± 0.042
C27	(E)-2-Nonenal	0.00019	Green, tallow	100	100	ND	ND	ND	ND	ND	ND	ND	ND
C30	3,5-Octadien-2-one	0.15	Pungent, unpleasant	ND	< 0.1	ND	ND	ND	ND	ND	ND	ND	ND
C31	(Z)-2-Octen-1-ol	0.02	Fresh, fatty, fruity, melon, earthy	ND	0.118 ± 0.011	ND	ND	1.45 ± 0.17	1.69 ± 0.03	ND	ND	ND	1.59 ± 0.17
C32	Acetophenone	0.065	Almonds, flower, meat	ND	ND	ND	ND	ND	ND	ND	ND	ND	0.627 ± 0.054
C34	Benzoic acid, ethyl ester	0.05556	Grainy	< 0.1	< 0.1	ND	0.452 ± 0.010	0.834 ± 0.024	0.549 ± 0.016	ND	ND	ND	ND
C36	1-(4,5-Dihydro-2-thiazolyl)-ethanone	0.001	Popcorn-like, roasty	ND	ND	ND	ND	48.28 ± 5.98	86.45 ± 6.96	ND	ND	ND	ND
C37	alpha-Ionone	0.00378	Raspberry-like	ND	1.11 ± 0.14	ND	ND	ND	ND	ND	ND	ND	ND
C38	Hexanoic acid	0.89	Cheesy, fatty	< 0.1	< 0.1	ND	ND	ND	ND	ND	ND	ND	ND
C40	Heptanoic acid	0.775	Sweaty, cheesy	< 0.1	< 0.1	ND	ND	ND	ND	ND	ND	ND	ND

ND: Not detected.

and 11.67 ± 1.12, respectively, which reflected the mainly green, grassy, and beany of odors from fresh sweet corn.

After the treatment of steaming, a total of 33 volatiles were detected in the sweet corn. Compared to fresh samples, the volatiles contained broadly significant decreases in aldehydes, alcohols, ketones, acids, furans, and anhydrides, with the absence of 3-methyl-2-butenal, (E)-2-heptenal, (E)-4-hexen-1-ol, 3-methyl-1-octene, 2-methyl- pentanoic acid anhydride. At the same time, the application of steaming led to the production of terpenoids, phenols, sulfides, and a few ketones and alcohols, resulting in the appearance of (E)-beta-ocimene (S5), 6-methyl-5-hepten-2-one (S5), 3,5-octadien-2-one (S5), (Z)-2-octen-1-ol (S5), alpha-ionone (S5), dimethyl trisulfide (S5, S15), 4-methyl-5H-furan-2-one (S10, S15), dimethyl sulfoxide (S15), dimethyl disulfide (S15), 2-methoxy-4-vinylphenol (S15), and 4-vinylphenol (S15) as new volatiles. Differences in steaming time further led to variations in sweet corn volatiles. With longer steaming time, there was some reduction in the category of sweet corn volatiles along with a 34.58–93.49% decrease in total content. To further distinguish S5, S10, and S15, PLS-DA analysis was performed on the steaming groups (Fig. 2A). The results indicated that the VIP values of seven volatiles, 4 (D-limonene), 22 (5-ethyl-cyclopent-1-enecarboxaldehyde), 2 (hexanal), 38 (hexanoic acid), and 17 (1-hexanol), were greater than 1, indicating that they were closely related to the time of steaming. In the steaming groups, hexanal (141.3 ± 7.1 ng·g^{-1} FW), D-limonene (382.4 ± 52.1 ng·g^{-1} FW), 1-hexanol (96.15 ± 9.02 ng·g^{-1} FW), 5-ethylcyclopent-1-enecarboxaldehyde (233.8 ± 21.1 ng·g^{-1} FW), and hexanoic acid (127.4 ± 12.0 ng·g^{-1} FW) all achieved a maximum at S5. And (E)-2-nonenal topped the ROAV for S5 at 100, closely followed by 1-octen-3-ol and hexanal with ROAVs

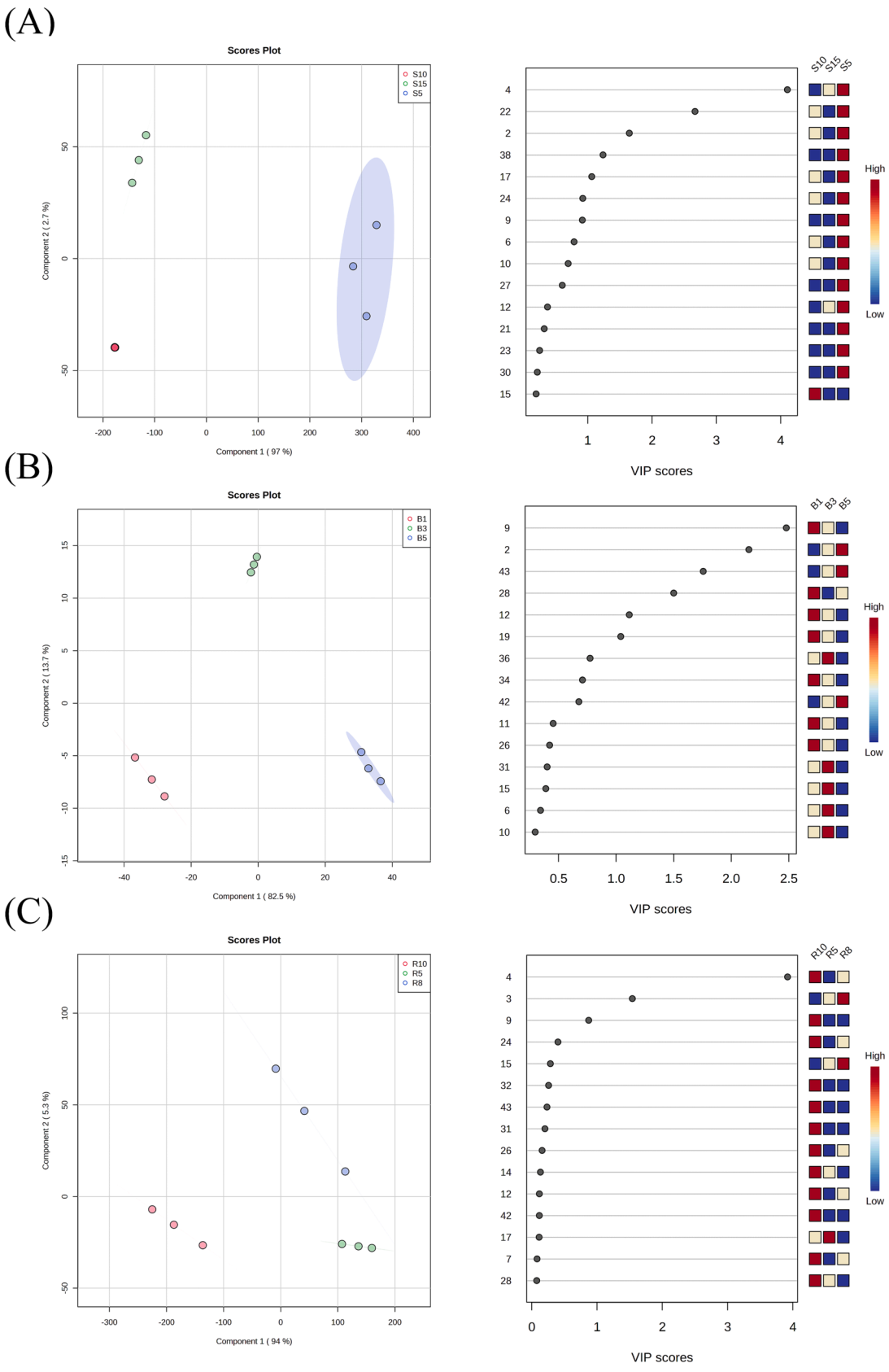

Fig. 2. PLS-DA of volatiles. (A) Steaming groups; (B) Blanching groups; (C) Roasted groups. The full name of compounds could be referred to in Table S1 according to No.

of 22.42 ± 0.23 and 8.80 ± 0.48 respectively. The key aroma-active components of S5 were closer to those of fresh samples, suggesting that the aroma of S5 might be closer to that of fresh samples. As for S10, the hexanal content of it decreased by 98.32% compared to fresh samples, dominated by 2-pentyl-furan (38.61 ± 1.49 ng·g^{-1} FW), 1-octen-3-ol (33.08 ± 1.02 ng·g^{-1} FW), and *n*-caproic acid vinyl ester (20.55 ± 0.74 ng·g^{-1} FW). Also, 1-octen-3-ol (1 0 0) was the substance with the highest ROAV in S10 and had a crucial influence on the aroma composition of S10. S15 no longer contained hexanal but was dominated by D-limonene (83.31 ± 18.41 ng·g^{-1} FW), 1.75-fold more than fresh samples. As the aroma-active volatiles in S15 with ROAVs greater than 50, dimethyl trisulfide, 1-octen-3-ol, nonanal, and beta-myrcene apparently effected the presentation of S15 aroma.

Only 21 volatiles were measured in sweet corn after blanching. It was clear that large amounts of volatiles were lost during blanching process, with alcohols, aldehydes, terpenes, acids, furans, and anhydrides being the most pronounced. Also, small amounts of volatiles were generated during the thermal process of blanching, such as tridecane, dimethyl trisulfide, (Z)-2-octen-1-ol, and 1-(4,5-dihydro-2-thiazolyl)-ethanone, common to B1 and B3, and 2-methoxy-4-vinylphenol, dimethyl sulfoxide, and 4-vinylphenol, common to B1, B3, and B5. However, different treatment times still resulted in slight discrepancies. The loss of sweet corn volatiles intensified as the blanching time increased, most notably in B5, with only eight volatiles remaining. The PLS-DA analysis was presented in Fig. 2B. There were 6 volatile components with VIPs larger than 1, which could be used as potential differentiating volatiles for different time of blanching treatment, including 9 (1-pentanol), 2 (hexanal), 43 (4-vinylphenol), 28 (octyl formate), 12 (acetoin), and 19 ((E)-4-hexen-1-ol). Among the blanched samples, octyl formate (44.63 ± 3.70 ng·g^{-1} FW), 1-pentanol (39.98 ± 2.74 ng·g^{-1} FW), acetoin (17.19 ± 2.99 ng·g^{-1} FW), and (E)-4-hexen-1-ol (16.44 ± 1.23 ng·g^{-1} FW) attended the peak values at B1. In addition, the top three substances with the largest ROAVs for B1 were dimethyl trisulfide (1 0 0), 1-octen-3-ol (95.37 ± 6.46) and 1-(4,5-dihydro-2-thiazolyl)-ethanone (48.28 ± 5.98), which would give B1 a distinctive aroma. The main composition of B3 was similar to that of B1, but the content of 1-pentanol was 38.62% lower compared to B1 and the amount of octyl formate was 51.87% of that of B1. But the content of acetoin and (E)-4-hexen-1-ol for B3 were lower than half of those of B1. In the same way, the largest dominant aromatic active substances of ROAVs in B3 were close to B1, with the only difference being the addition of nonanal (48.95 ± 4.23). B3 was probably closer to B1 in aroma generally. After 5 min of blanching, sweet corn volatiles were dominated by hexanal (38.50 ± 7.45 ng·g^{-1} FW), 1-octen-3-ol (36.64 ± 2.36 ng·g^{-1} FW), and 4-vinylphenol (33.02 ± 6.38 ng·g^{-1} FW). Among these, 1-octen-3-ol had a ROAV of 100, the largest of the B5, and probably dominated the aroma presentation of the B5.

Similarly, roasting treatment resulted in significant changes in sweet corn volatiles, mainly in the loss of alcohols, aldehydes, acids, furans, anhydrides, and some esters and ketones. Only 19 volatiles were detected in roasted sweet corn. The roasting conditions catalyzed the production of various new substances, such as prenol, (Z)-3,7-dimethyl-1,3,6-octatriene, and dimethyl sulfoxide, common to R5, R8, and R10, and (Z)-2-octen-1-ol, acetophenone, 2-methoxy-4-vinylphenol, and 4-vinylphenol, unique to R10 in the roasting groups. Meanwhile, the total volatile content of roasting groups increased with longer treatment time and was 19.56 ~ 38.89% of fresh samples (2539 ± 120 ng·g^{-1} FW). The roasting treatment resulted in the most significant proportion of terpenoids in sweet corn, dominated by D-limonene. In order to better distinguish between R5, R8, and R10, the VIPs of 4 (D-limonene), 3 (beta-myrcene), and 9 (1-pentanol) were found to be greater than 1 with the help of PLS-DA analysis (Fig. 2C), and they could be used as characteristic volatiles to help distinguish between samples treated with different roasting time. In roasting groups, D-limonene was the richest, with content of 298.3 ± 27.1 to 619.4 ± 50.8 ng·g^{-1} FW, accounting for 57.86% to 63.00% of the total, and 6.27 to 13.02 folds more than fresh samples. beta-Myrcene ranked second in the volatiles of R5 and R8, and was more than twice as high as in fresh samples, reaching 42.28 ± 4.53 and 89.35 ± 17.11 ng·g^{-1} FW. D-limonene attained the maximum value for all sample at R10. Also, beta-myrcene (R5 100; R8 100), 1-octen-3-ol (R5 53.89 ± 4.47; R8 41.62 ± 4.55), and D-limonene (R5 22.59 ± 2.00; R8 21.59 ± 0.71), which ROAVs ranked top three, were the most significant aroma-active substances in both R5 and R8, characterizing that R5 and R8 might share a similar aroma profile. Different from R5 and R8, R10, without beta-myrcene, attained the peak values of 1-pentanol (56.05 ± 7.79 ng·g^{-1} FW), which did not appear in R5 and R8. The most predominant aroma-active substances in R10 overlapped with R5 and R8, namely 1-octen-3-ol (1 0 0) and D-limonene (43.21 ± 0.13), which ROAVs ranked top two, but probably had a less identical aroma profile to R5 and R8.

3.2. Change of fatty acid composition on sweet corn after thermal processing

Two saturated fatty acids (SFAs) and two unsaturated fatty acids (UFAs) were measured in sweet corn subjected to different thermal treatments (Table S2). The main components were palmitic acid (SFA), stearic acid (SFA), oleic acid (UFA), and linoleic acid (UFA), and the results are shown in Fig. 3. The data indicated that, in contrast to fresh samples (1.56), thermal treatments resulted in a 16.67–38.46% increase of UFA/SFA ratio in sweet corn, reaching a maximum at R8 (2.16) and R10 (2.11). The largest proportion of fatty acids was linoleic acid, which accounted for about 35.72% to 37.95% of the total fatty acid content. The linoleic acid content of sweet corn was significantly increased by heat treatment, with the largest percentage increase in linoleic acid content in B1 (7522 ± 80 μg·g^{-1} FW) and B3 (7572 ± 609 μg·g^{-1} FW), with 83.60% and 84.82% increases compared to fresh samples (4097 ± 86 μg·g^{-1} FW). B5 (6667 ± 204 μg·g^{-1} FW) was a smaller improvement, with only a 62.73% increase compared to fresh samples. The roasting group exhibited a remarkable enhancement in linoleic acid content as the roasting time increased, with a significant increase from 4876 ± 489 (R5) to 7013 ± 274 (R10) μg·g^{-1} FW, being 1.10–1.39-fold of fresh samples. The overall increase in fatty acid content was lower in steaming groups, with the lowest elevation of linoleic acid of all treatments at S10 (4516 ± 308 μg·g^{-1} FW), being 10.23% higher than fresh samples. In contrast, the linoleic acid content was elevated by 28.09% and 39.10% for S5 (5248 ± 482 μg·g^{-1} FW) and S15 (5699 ± 51 μg·g^{-1} FW), respectively. The content of oleic acid ranked second in the total fatty acids of sweet corn, which was dramatically enhanced by thermal treatment. The oleic acid content was boosted the most in blanching groups, ranging from 1.61 to 1.81-fold that of fresh samples (3293 ± 87 μg·g^{-1} FW), reaching a maximum at B1 and B3 with 5938 ± 161 and 5959 ± 527 μg·g^{-1} FW, correspondingly. B5 (6667 ± 204 μg·g^{-1} FW) was the lowest value in blanching groups, but still 1.63-fold higher than fresh samples. The roasted samples ranked second in the total, which also effectively increased the oleic acid content. As the roasting time increased, the oleic acid in the samples gradually increased by 21.74 to 71.97% compared to fresh samples, reaching the maximum at R5 (4009 ± 348 μg·g^{-1} FW) and the minimum at R10 (5663 ± 257 μg·g^{-1} FW). Lastly, the steaming treatment was the least effective in raising oleic acid, being only 1.10 to 1.36-fold more than fresh samples, with both S5 (4035 ± 342 μg·g^{-1} FW) and S15 (4487 ± 83 μg·g^{-1} FW) having a higher oleic acid content than S10 (3619 ± 207 μg·g^{-1} FW). As for palmitic acid, although its content increased to some extent after processing, its share of the total fatty acids tended to decrease. Blanching raised the palmitic acid content of samples by 29.20 to 65.43% over fresh samples (3243 ± 98 μg·g^{-1} FW). However, palmitic acid content continued to decline as blanching treatment longer, with B1 having the highest palmitic acid content of 5365 ± 526 μg·g^{-1} FW. The minimum value of palmitic acid after blanching treatment was reached at B5 (4190 ± 49 μg·g^{-1} FW). The palmitic acid content of both R8 (4054 ± 276 μg·g^{-1} FW) and R10 (4448 ± 55 μg·g^{-1} FW) was slightly increased by

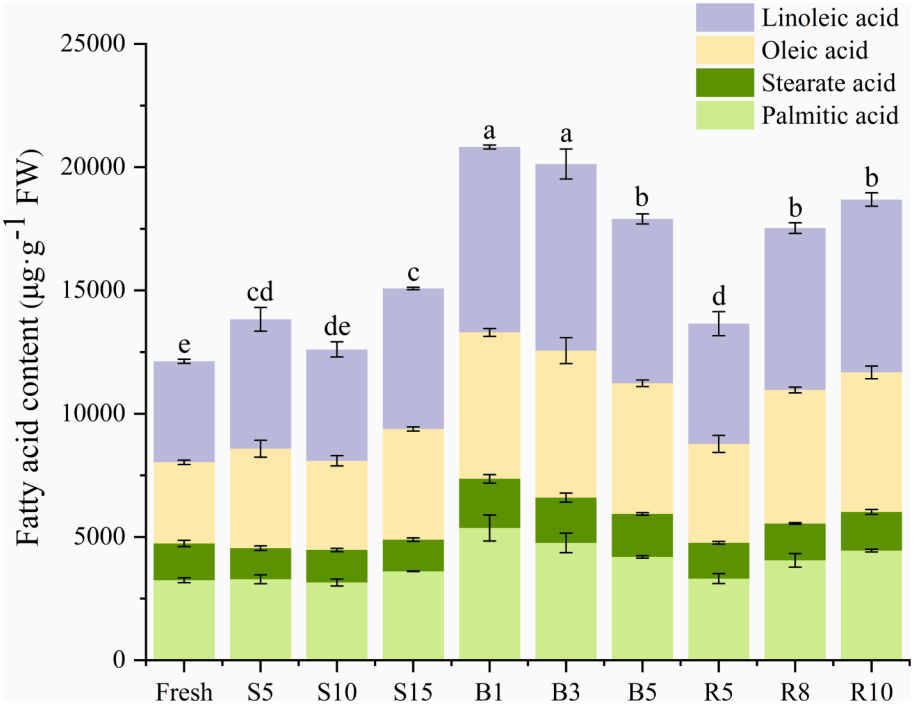

Fig. 3. The content of fatty acids in sweet corn after different thermal treatments (mean ± SD, n = 3). Bars with different letters differ significantly at $p < 0.05$.

25.01 and 37.16% in the roasting group compared to fresh samples, except for R5 was not significantly different from the control group. Also, the palmitic acid content obtained from the steaming treatment was not significantly different from that in fresh samples. As the least abundant fatty acid, stearic acid, its content was enhanced by blanching treatment. The stearic acid content reduced with increasing blanching time, reaching maximum values at B1 and B3, being 1994 ± 174 and 1841 ± 185 μg·g^{-1} FW, respectively, 1.23–1.34-fold of F (1492 ± 126 μg·g^{-1} FW). B5 (1748 ± 52 μg·g-1 FW) had the lowest stearic acid content among blanching groups. The roasting groups had the second highest stearic acid content after blanching groups. The stearic acid content of R5 and R8 had no significant difference with fresh samples but presented an increased trend as roasting time was extended, achieving a maximum at R10 (1566 ± 99 μg·g^{-1} FW), being 1.05-fold of fresh samples. Yet, compared to fresh samples, the stearic acid in sweet corn dropped by 11.53–15.48% after steaming treatment, reaching the maximum at S10 (1320 ± 69 μg·g^{-1} FW) and the minimum at S5 (1261 ± 92 μg·g^{-1} FW).

4. Discussion

4.1. Effect of steaming on volatiles and fatty acids of sweet corn

Steaming significantly altered the volatile properties of sweet corn. The aldehydes in sweet corn were dramatically reduced after steaming. As the most abundant aldehyde, hexanal had a smell of grassy and green and possibly reduced sharply because of autoxidative linoleic acid degradation (Cremer & Eichner, 2000). Similar to hexanal, nonanal and (E)-2-nonenal were also produced by oxidative cleavage of lipids. The decline or even disappearance of nonanal and (E)-2-nonenal could be due to the destruction of lipoxygenase in heat-processed corn (Cho & Peterson, 2010). And (E)-2-nonenal only occurred in S5 with a ROAV of 100, indicating that (E)-2-nonenal could be considered to be a potential major odorant of S5, and probably dominated the overall odor of S5 as green and tallow. Interestingly, nonanal was only detected in S15 in steaming groups, which might be the result of the non-enzymatic oxidation of lipids. Nonanal added a citrus-like aroma to S15. Simultaneously, as the oxidation product of hexanal, hexanoic acid, which was only present in S5 after thermal treatment, reduced until it disappeared as the content of hexanal dropped (Lehto et al., 2003). Similar to aldehydes, the concentration of primary alcohols gradually decreased as steaming time as long, which could be due to the minor molecular alcohols with lower boiling points continuously being taken away with water vapor, such as 1-octen-3-ol (Zhang et al., 2022). As an important aroma-active volatile in steaming groups, 1-octen-3-ol gave a mushroom-like odor to steamed sweet corn. As for alcohols with high boiling points, such as 1-hexanol, the reductases could be destroyed during steaming, and the precursors, like hexanal, declined, so the content of 1-hexanol obviously debased. Apart from aldehydes and alcohols, 2-pentyl-furan was also negatively affected by heat treatment. 2-Pentyl-furan was formed from linoleic acid by singlet oxygen (Min et al., 2003). The substantial decrease in 2-pentyl-furan content in thermally processed corn could be due to elevated levels of antioxidant products, such as D-limonene and beta-myrcene. As to the increase of D-limonene, it could be explained by 5-min heat processing causing a significant amount of direct cell damage. Meanwhile, the increased content of D-limonene imparted a more-fruity aroma to S5 than fresh samples. As for beta-myrcene, it was only retained in S15 after steaming, providing a pine-like aroma to S15. The dimethyl trisulfide, which smelled of green onion, was thought to be produced from methionine by Strecker reaction (Pan et al., 2021). In particular, 5-ethylcyclopent-1-enecarboxaldehyde and *n*-caproic acid vinyl ester were found for the first time in sweet corn as characteristic volatiles.

Fatty acids were less affected by steaming treatment. Palmitic acid demonstrated high stability in 5–15-minute steaming treatment. The slight downward trend of stearic acid after steaming was probably due to the thermal degradation of stearic acid. In contrast to stearic acid, the increase in unsaturated fatty acid (oleic acid and linoleic acid) content could be due to the release of fatty acids from cells damaged by high-temperature treatment. Also, 100–110 °C would induce triacylglycerol hydrolysis to produce free fatty acids (Barthet et al., 2002). The correlation between volatiles and fatty acids was shown in Fig. 4A. The volatiles of sweet corn after steaming were positively correlated with stearic acid (R^2 = 0.282–0.989, $p < 0.05$) except for 1 (dimethyl

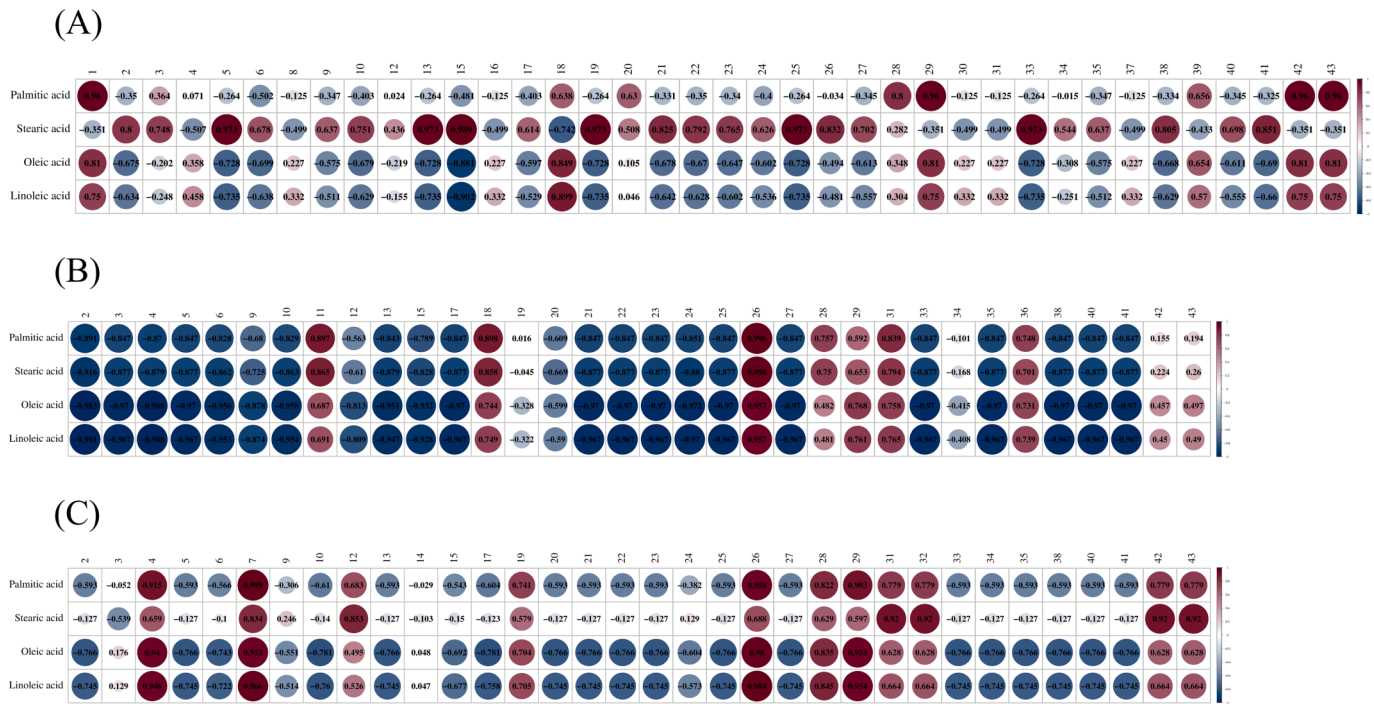

Fig. 4. A correlation heatmap of volatiles and fatty acids. (A) Steaming groups; (B) Blanching groups; (C) Roasting groups. The full name of compounds could be referred to in Table S1 according to No.

disulfide), 4 (D-limonene), 8 ((E)-beta-ocimene), 16 (6-methyl-5-hepten-2-one), 18 (dimethyl trisulfide), 29 (dimethyl sulfoxide), 30 (3,5-octadien-2-one), 31 ((Z)-2-octen-1-ol), 37 (alpha-ionone), 39 (4-methyl-5H-furan-2-one), 42 (2-methoxy-4-vinylphenol), and 43 (4-vinylphenol). Meanwhile, 1 (dimethyl disulfide), 18 (dimethyl trisulfide), 20 (nonanal), 28 (octyl formate), 29 (dimethyl sulfoxide), 39 (4-methyl-5H-furan-2-one), 42 (2-methoxy-4-vinylphenol), and 43 (4-vinylphenol) were significantly positively correlated with palmitic acid (R^2 = 0.630–0.960, $p < 0.05$). 1 (dimethyl disulfide), 18 (dimethyl trisulfide), 29 (dimethyl sulfoxide), 39 (4-methyl-5H-furan-2-one), 42 (2-methoxy-4-vinylphenol), and 43 (4-vinylphenol) had significant positive correlation with both oleic acid (R^2 = 0.654–0.849, $p < 0.05$) and linoleic acid (R^2 = 0.570–0.899, $p < 0.05$). This indicated a close association of volatiles with fatty acids in the steamed samples, which was consistent with the characteristic volatiles of S5-S15 being mostly lipid oxidation products.

4.2. Effect of blanching on volatiles and fatty acids of sweet corn

Alcohols were the first main constituents of volatiles after blanching treatments. Compared with fresh samples, the apparent decrease of alcohols, for instance, 1-pentanol and 1-octen-3-ol, could be explained that these alcohols were lost from the sweet corn to water during blanching process and even be released with water vapor. In the meantime, 1-octen-3-ol was one of the volatiles with top ROAV in blanching groups and could be considered the key odorant. Different ketones responded differently to blanching treatment. 1-(4,5-Dihydro-2-thiazolyl)-ethanone was a completely new substance that appeared in B1 and B3, and could be formed by the reactions of amino acids with sugar degradation products during heat processing (Kumazawa & Masuda, 2002). As the main aroma-active volatiles of B1 and B3, 1-(4,5-dihydro-2-thiazolyl)-ethanone would contribute to their popcorn-like aroma, while dimethyl trisulfide provided an aroma of green onion. Nevertheless, acetoin was affected by blanching, and this could be due to partial dissolution of acetoin in water, and the oxidative degradation of sugars and amino acids which was inhibited by blanching treatment (Lu et al., 2021). Aldehydes likewise tended to decline after blanching treatment, especially hexanal. In boiling water, the autoxidative linoleic acid degradation might be restrained by a lack of oxygen that hexanal of blanching groups dropped much more than that of steaming groups. And in B5, hexanal reached the highest level in blanching groups, and phenols attained the maximum in all samples. Among them, 4-vinylphenol, an aroma with the sweet odor of caramel, was reported to be form by thermal decomposition of p-coumaric acid (Yajima et al., 1978). It was possible that the longer the blanching time, the more degradation of p-coumaric acid, so that 4-vinylphenol increased with longer processing time. Lastly, (E)-4-hexen-1-ol and octyl formate, which lacked report, were found for the first time in sweet corn volatiles.

Moderate blanching seemed to have a positive effect on the fatty acids of sweet corn. The four fatty acid content of sweet corn detected increased significantly after blanching and demonstrated a tendency to decrease with longer blanching times. The enhancement of fatty acid content could be due to the destruction of sweet corn cells by blanching, increasing free fatty acids. During the blanching process, sweet corn had a possible effect of blocking the interaction of oxygen and light with lipids, allowing unsaturated fatty acids to be protected (Chen et al., 2022). However, prolonged blanching time could cause the thermal degradation of fatty acids. According to Fig. 4B, a more significantly positive correlation could be found among 11 (tridecane), 18 (dimethyl trisulfide), 26 (2-ethyl-1-hexanol), 28 (octyl formate), 29 (dimethyl sulfoxide), 31 ((Z)-2-octen-1-ol), and 36 (1-(4,5-dihydro-2-thiazolyl)-ethanone) of the blanching treatment with all four fatty acids (R^2 = 0.481–0.998, $p < 0.05$). It was clear that the volatiles in the blanched samples had a small association with fatty acids.

4.3. Effect of roasting on volatiles and fatty acids of sweet corn

With reference to raw sweet corn, terpenoids with elevated content were the essential contributing products to volatiles in roasted sweet corn. As the most important component, D-limonene was considered to originate from the Maillard reaction and obviously boosted with time (Li et al., 2018). The high temperature significantly affected the synthesis of D-limonene, thus adding a fruity aroma to roasted corn. beta-Myrcene with the typical “woody” and “herbal” notes could be significantly

increased after roasting along with the cleavage of long-chain fatty acids and the metabolism of terpenoids (Hu et al., 2018). The disappearance of beta-myrcene could be attributed to auto-oxidation caused by longer processing time. But alcohols were negatively affected by roasting and a decrease in content occurred, including 1-octen-3-ol and 1-pentanol. The content of 1-octen-3-ol was raised with longer roasting time, but was still lower than fresh samples. The increase in 1-octen-3-ol with rising roasting time, as an oxidative cleavage product of linoleic acid, could be due to the extended treatment time which disrupted the cells, resulting in the release of more 1-octen-3-ol from the cells. Overall, D-limonene with great changes would be the critical volatile to distinguish aroma characteristics of sweet corn after 5 to 10-min roasting.

The four fatty acids in sweet corn were elevated by roasting and tended to be higher with longer roasting time. This was probably due to the damage of more sweet corn cells caused by the prolongation of roasting time. Furthermore, corn was rich in xanthophyll and primarily esterified with fatty acids, which could facilitate the storage and stability of unsaturated lipids. Fig. 4C illustrated the correlation between volatiles and fatty acids after roasting, and it was observed that 4 (D-limonene), 7 ((Z)- 3,7-dimethyl-1,3,6-octatriene), 12 (acetoin), 19 ((E)-4-hexen-1-ol), 26 (2-ethyl-1-hexanol), 28 (octyl formate), 29 (dimethyl sulfoxide), 31 ((Z)-2-octen-1-ol), 32 (acetophenone), 42 (2-methoxy-4-vinylphenol), and 43 (4-vinylphenol) were more significantly positively correlated with the four fatty acids ($R^2 = 0.495–0.984$, $p < 0.05$). This was possibly due to the breakdown of lipid hydroperoxides formed during roasting to produce volatile flavor compounds such as ketones and alcohols (Boge et al., 2009).

4.4. Evaluation of aroma markers and compositions of main aroma in heat-treated sweet corn aroma

To further understand the overall effect of heat treatment on sweet corn aroma and to investigate which treatment yielded the sweet corn aroma closest to fresh corn, and since the “main body” of the aroma, which represents the overall odor, is defined by the main aroma through the key aroma active compounds (Gu et al., 2022), the ROAV of 28 aroma-active volatiles were analyzed using PLS-DA. The score plot (Fig. 5A) showed that S5 and fresh samples leaned close together, but at a significant distance from the other samples. The variable importance in projection (VIP) score plot was shown in Fig. 5B, with the VIP scores listed in Table S3. There are five volatiles with VIP scores greater than one, including 1-(4,5-dihydro-2-thiazolyl)- ethenone (36), beta-myrcene (3), 1-octen-3-ol (24), dimethyl trisulfide (18), and (E)-2-nonenal (27). Compounds with VIP scores higher than one were considered to be aroma markers that could be used to distinguish between ten different types of processed sweet corn. In addition, the ROAV value of D-limonene (4) varied significantly in sweet corn with different processing methods, thus including an additional aroma compound. The main aroma components of the steaming group were a combination of beta-myrcene, D-limonene, dimethyl trisulfide, and 1-octen-3-ol which tended to increase with increasing steaming time (Fig. 5C). And the main aroma components of the hot group were dominated by dimethyl trisulfide and 1-octen-3-ol (Fig. 5D). As for the roasted group, the main aroma components showed a combination of beta-myrcene which abruptly disappeared with increasing roasting time, and D-limonene, 1-octen-3-ol, which tended to increase with longer roasting time (Fig. 5E). (E)-2-Nonenal appeared only in the fresh sample and in S5, both reaching a ROAV of 100. 1-(4,5-Dihydro-2-thiazolyl)-ethanone occurred exclusively in B1 and B3. Steaming, blanching and roasting at different

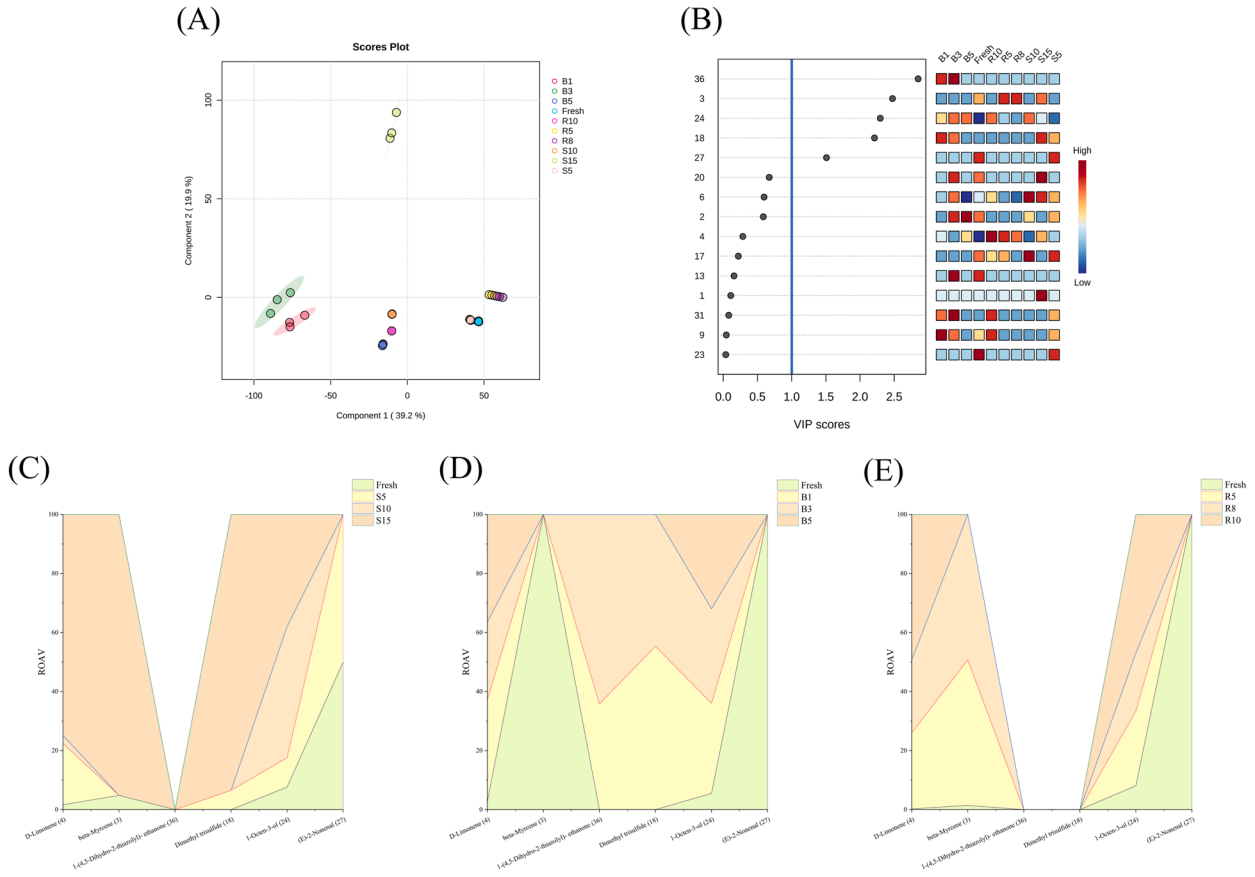

Fig. 5. (A) PLS-DA score plot; (B) VIP score plot; (C-E) ROAV of six aroma markers representing aroma boundary compositions of fresh sweet corn with steaming, blanching, and roasting groups, respectively. The full names of compounds could be referred to in Table S1 according to No.

times apparently had a significantly different effect on the main sweet corn aromas. Meanwhile, the ROAV values for the main aroma markers, including 1-octen-3-ol (fresh sample: 17.22 ± 0.87; S5: 22.42 ± 0.23) and (E)-2-nonenal (1 0 0), were very close for the fresh sample and S5. In combination with the PLS-DA analysis, the results showed that S5 had the closest major aroma profile to the fresh sample.

5. Conclusion

The present study investigated that steaming, blanching, and roasting significantly altered aroma volatiles and effectively increased the fatty acid content of sweet corn. Twenty-seven volatiles were identified in fresh samples, and after the steaming, blanching, and roasting, the number of volatiles increased by six and decreased by six and eight, respectively. Characteristic aroma-active volatiles of sweet corn after thermal treatments included: (E)-2-nonenal (green, tallow), 1-octen-3-ol (mushroom-like), beta-myrcene (pine-like), dimethyl trisulfide (savory, green onion), 1-(4,5-dihydro-2-thiazolyl)-ethanone (popcorn-like, roasty), and D-limonene (citrus-like, fruity). Besides, after thermal treatments, unsaturated fatty acids (oleic acid and linolenic acid) were increased by 110 to 183%. In parallel, the oxidative cleavage of fatty acids produces most of the characteristic volatiles found in sweet corn. The aroma of S5 was thought to be closest to that of fresh corn. The investigation revealed the effects of different heat treatments on aroma volatiles and fatty acids of sweet corn and provided guidance for subsequent research on the sources of flavor changes in processed sweet corn.

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CRediT authorship contribution statement

Bing Zhang: Software, Validation, Data curation, Writing – original draft, Visualization. **Kun Li:** Methodology, Formal analysis, Data curation, Funding acquisition. **Hao Cheng:** Software, Methodology. **Jianguang Hu:** Resources, Project administration. **Xitao Qi:** Conceptualization, Investigation, Resources. **XinBo Guo:** Conceptualization, Project administration, 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.

Data availability

Data will be made available on request.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.fochx.2023.100743.

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