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Characterization of aroma-active compounds in sesame hulls at different roasting temperatures by SAFE and GC-O-MS

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ABSTRACT

The study characterized the aroma-active compounds produced by sesame hulls at three roasting temperatures and analyzed the similarities and differences in the aroma profile of sesame hulls with whole seeds and kernels after roasting. Roasting hulls produced mainly furans, aldehydes, and ketones volatiles. 140 Compounds were identified as aroma-active compounds, including 36 key aroma compounds (odor activity value, OAV $\geq$ 1). Among them, furanone (caramel-like, OAV = 80), 3-methylbutanal (fruity, OAV = 124), and 2-methoxy-4-vinylphenol (burnt, smoky, OAV = 160) gave hulls (180 °C) sweet, burnt, and smoky aroma. Due to the contribution of vanillin (fatty, sweet milk, OAV = 45), 2-hydroxy-3-butanone (caramel-like, roast, OAV = 46), and 2-methoxy-4-vinylphenol (OAV = 78), hulls (200 °C) shown strong sweet and roast note. These results identified compounds that contributed significantly to the aroma of sesame hulls and elucidated the contribution of sesame hulls to the flavor of roasted whole seeds and sesame oil.

1. Introduction

As an ancient oilseed crop, sesame (*Sesamum indicum* L.) has been cultivated in China and other Asian countries for its nutrition and flavor. Sesame seeds contain 22–25 % protein and 44–58 % oil (Yin et al., 2021). Sesame oil contains unsaturated fatty acids (more than 80 %) and many bioactive compounds (tocopherols, phytosterols, lignans, etc.). It is confirmed that sesame seeds have exhibited numerous health-promoting characteristics, including anti-lipogenic, hypo-cholesterol, anti-hypertensive, anti-inflammatory, and neurologically beneficial properties (Langyan et al., 2022).

At present, the annual global production of sesame seeds is approximately 6 million tons, of which about 45 % is processed to make sesame oil (Yin et al., 2021). One important feature that distinguishes sesame oil from other vegetable oils is its rich fragrance when made from roasted seeds (Liu, Yao, Ma, & Wang, 2020b). This unique and pleasant aroma makes roasted sesame oil very popular with consumers worldwide (Liu, Han, Wang, Zheng, & Wang, 2020a; Tamura et al., 2010). Many researchers have become interested in sesame oil flavors. Jia et al. (2019) have identified the key aroma-active compounds in sesame oil from microwaved seeds. Yin et al. (2021) have compared the key aroma compounds in hot-pressed and cold-pressed sesame oils. These studies provided valuable information for flavor control of sesame oil products. As Liu, Wang, Tamogami, Chen, and Zhang (2020c) have said, flavor is the most direct and easily understood parameter by which consumers judge the quality of edible oil. Hence, understanding the flavor characteristics of edible oil and how those characteristics are generated is of great value to the oil processing industry. For many years, numerous researchers have been studying the volatile flavor compounds in sesame seeds and have found that the aroma produced by roasted sesame seeds with hulls is stronger than that of hulled sesame seeds. The reason for this is the large number of polysaccharides, especially cellulose, hemicellulose, and pectin, in the hulls. Roasting partially degrades the cellulose and hemicellulose side chains in the crystalline region and degrades the RG-I region and side chain structure of pectin, producing important precursors for the chemical reactions (especially the Maillard reaction and caramelization) that produce aroma and flavor (Yao et al., 2021). In addition to volatile flavor compounds, the oil from roasted sesame seeds has a stronger antioxidant ability (Liu et al., 2020a). The main reason for this is that the lignin in sesame hulls degrades at high temperatures to produce a large number of phenolic compounds (Shahidi, Liyana-Pathirana, & Wall, 2006). It is reasonable to guess that

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sesame hull has a prominent role in the quality of sesame oil. Up to now, most of the literature reports on the flavor constituents of sesame oil and the effects of different heating protocols on the aroma of sesame seeds and sesame oil. However, there is no relevant research on the aroma compounds specifically produced by sesame hulls. This needs to be filled. Therefore, it is necessary to use the relevant flavor extraction method and analysis technology to study the aromatic ingredients produced after roasting sesame hulls and the influence of roasting temperature on flavor.

In this study, the aroma substances of roasted and unroasted whole sesame seeds, sesame kernels, and sesame hulls were analyzed and compared by solvent-assisted flavor evaporation (SAFE) combined with gas chromatography-olfactometry-mass spectrometry (GC-O-MS). External calibration was performed to achieve accurate quantification of aroma-active compounds with relative odor activity values (ROAV) ≥ 1, after which the key compounds (odor activity values ≥ 1) in each sample were identified. Principal Component Analysis (PCA) was performed on all of the key aroma components to determine differences in odor characteristics between individual samples and identify the classes of compounds that had a major impact on aroma. The samples were subjected to sensory evaluation to determine the differences in the overall flavor profile of sesame hulls from that of whole sesame seeds and kernels. Knowing the contribution of sesame hulls to the flavor and aroma profile could fill the gap in the research of sesame hull flavor. This study identified compounds that contributed significantly to the flavor of sesame hulls and elucidated the contribution of sesame hulls to the flavor of roasted whole seeds and sesame oil.

2. Material and methods

2.1. Material

The sesame (Zhuzhi 22) provided by the Henan Academy of Agricultural Sciences (Zhengzhou, China) was used in this study. Raw sesame seeds were selected for high quality, and moldy, rotten, and incomplete seeds were removed. To ensure that the structure of sesame kernels and hulls was not destroyed in separating hulls from seeds, the method of Zhang et al. (2022) was used. The sesame seeds were soaked in water and then rubbed by hand with gauze until the hulls were almost completely separated from the seeds. After drying in an oven at 55 °C, the dried seeds and hulls were passed through a 30 mesh screen to separate hulls and kernels. The hulls and kernels were stored in a desiccator for subsequent tests. Immediately before roasting, the sesame seeds, sesame kernels, and sesame hulls were dried at 55 °C for 2 h to remove any water the sample materials may have absorbed during storage. The moisture content of the raw material was tested to be about 5 %. After that, 10 g of each material was loaded into pressure bottles and heated at three different temperatures (160, 180, and 200 °C) in an oil bath with magnetic stirring for 0.5 h (DF-101 S, YUHUA, China). The samples were then promptly cooled to room temperature, and flavor compounds were extracted with dichloromethane. Untreated raw materials were used as a blank control. The names of the untreated samples were S, K, and H, referring to sesame seeds, kernels, and hulls, respectively. The samples were designated as S160, K160, H160, S180, K180, H180, S200, K200, and H200, with the numbers referring to the roasting temperature.

2.2. Chemicals

Normal alkane mixed standard (C_7–C_{30}), 4-nonanol, dichloromethane (AR), anhydrous sodium sulfate, and all standards used for external calibration (purity $\geq$ 98.0 %) were acquired from Macklin Biochemical Technology Co., Ltd (Shanghai, China). The standards include pyrazines ***1–4***, ***7***, ***8***, ***11***, ***13***, ***15***, ***19***; pyrrole ***23***; pyridine ***41***; furans ***47–49***, ***52***, ***53***, ***56***; thiazole ***73***; thiophene ***75***; aldehydes ***76***, ***78***, ***81***, ***84***, ***86–88***, ***90–92***, ***94***, ***95***; ketones ***104***, ***108***; phenols ***124***, ***125***, ***127***; acid ***129***; S-containing compounds ***138–140***.

2.3. Solvent-assisted flavor evaporation (SAFE)

The detailed protocol of SAFE assessments was described in a published paper (Yin et al., 2022) with slight modifications. 10.0 g sample was put into a bottle with a threaded cap; 100 mL dichloromethane was added, followed by 150 μL of 4-nonanol (1 mg/mL). The mixture was shaken for 12 h to improve extraction efficiency and to ensure thorough mixing. The extracted liquid was subjected to vacuum filtration, and volatile substances were extracted by SAFE. Water was removed from the SAFE distillate by adding Na_2SO_4 (10.0 g) and freezing the mixture for 12 h at −20 °C. The distillate was then concentrated to 1 mL using a Vigreux column (50 cm × 1 cm) and a continuous stream of nitrogen.

2.4. Gas chromatography-olfactometry-mass spectrometry (GC-O-MS) analysis

Based on a previous paper (Yin et al., 2022), volatile compounds in SAFE were analyzed by a 7890B GC instrument equipped with a 5977A electron ionization MS (Agilent Co., Ltd., USA). The SAFE extract was injected in a volume of 1 μL. The volatile substances were separated by an HP-5MS column (nonpolar, 30 m × 250 μm × 0.25 μm), and flavor compounds were determined by an olfactometry detection port (ODP-3) (Gerstel Inc., USA). The specific GC heating procedure and the scanning range and speed of the MS were consistent with Yin et al. (2022). Briefly, the GC ramp-up procedure was 40 °C held for 5 min, followed by an increase to 130 °C at 3 °C/min, holding for 5 min, and a final increase to 250 °C at 10 °C/min with another 5-min hold at 250 °C. In 70 eV electronic shock mode, the MS scan range was 33–400 *m/z* at 2.0 scan/s. The volatile substances with aroma were further identified based on odor descriptions by 3 panelists with one year of sniffing experience. They sniffed one or two samples per day for 40 min each, with an interval of more than 4 h between samples. During sniffing, in order to increase the comfort level of panelists, their nasal passages were moistened with nitrogen gas passed through ultrapure water.

2.5. Qualitative analysis

The identification of aroma compounds was based on mass spectrometry (MS), odor description (O), and linear retention index (RI). The initial identification of the aroma compounds was performed using the NIST17 library. By identifying C_7–C_{30} alkanes and calculating the linear retention index (RI), the identities of the scent compounds were verified.

2.6. Quantification analysis

The concentration of each volatile detected by GC-MS in the SAFE extracts was calculated from the peak area ratios of the analyte to 4-nonanol. Quantification of compounds with relative odor activity values $\geq$1 by external standard method (Yin et al., 2021).

2.7. Relative odor activity values (ROAV)

Relative odor activity values (ROAV) calculated using relative concentrations were used to assess the contribution of individual compounds to the overall aroma. ROAV ranges from 0 to 100. The larger the ROAV, the greater the contribution of the component to the overall aroma of the sample. This method avoids the laborious task of absolute quantification of each compound. The formula of ROAV is as follows (Wang et al., 2019).

$$ROAV_A = 100 \times \frac{C_A}{T_A} \times \frac{T_{MAX}}{C_{MAX}} \quad (1)$$

where the maximum ROAV of the compound in the sample is defined as

100, $ROAV_{MAX} = 100$, the threshold of the compound is defined as T_{MAX} and the relative concentration is defined as C_{MAX}, C_A is the relative concentration of compounds to be calculated, T_A is the threshold of compounds to be calculated.

2.8. Odor activity values (OAV)

The odor activity value of a compound is the absolute concentration of the compound divided by its odor threshold (Yin et al., 2021). Compounds with an OAV ≥ 1, which contribute significantly to the overall aroma of the sample, are often considered to be key aroma-active compounds.

2.9. Organoleptic evaluation

10 trained panelists (6 females and 4 males) from the Henan University of Technology, with an average age of 23, assessed the flavor characteristics of sesame seeds, sesame kernels, and sesame hulls. The panelists were informed about the requirements of the experiment and their consent was obtained prior to the study. They volunteered to participate in the study with appropriate protocols to protect their rights and privacy. The evaluations were performed in a comfortable environment without other odors. 3 g of each of the 12 samples were put into brown bottles with lids. Before doing the sensory evaluation, the panelists were trained to identify odors by having them sniff standards. After training, the panelists sniffed, identified, and then scored each of the samples. The average scores of the ten panelists were plotted on a radar map that indicated the intensity of flavor.

2.10. Statistical analysis

All experiments were repeated at least three times. The experimental data were analyzed by ANOVA using IBM SPSS Statistics version 20 software (IBM Corp, Armonk, NY, USA). Significant differences between samples were analyzed using Duncan's multiple range test ($P < 0.05$). Origin 2023b was used for principal component analysis and drawing graphics.

3. Results and discussion

3.1. Comparison of volatile components

A total of 319 volatile substances were detected in hulls, comprising 36 furans, 8 pyrazines, 7 pyridines, 11 pyrroles, 1 thiazole, 47 aldehydes, 52 ketones, 24 alcohols, 14 phenols, 20 acids, 32 esters, 3 S-containing compounds, 53 alkanes, and 11 alkenes. The volatile components of hulls were different from those of sesame kernels and whole seeds, while the latter two were similar (Fig. 1). This is because the production of volatile substances is mainly related to carbohydrate, oil, and protein. Sesame hull mainly contains carbohydrate and the content of oil (about 2 %) and protein (about 7 %) in sesame hulls was very low, while the proportion of oil and protein in sesame kernels and whole seeds is great and similar. In this research, the relative concentration of volatile substances detected in unroasted sesame hulls was 33286 μg/kg, while the relative concentration detected in hulls roasted at 200 °C for 30 min was 818325 μg/kg (Table 1). According to the GC–MS analysis results, the total number and content of volatiles detected in hulls, seeds, and kernels increased with roasting temperature. Roasting not only triggered the oxidation of lipids but also generated the breakdown of sugars and proteins through caramelization and Maillard reactions, resulting in the formation of ketones, aldehydes, and heterocyclic compounds (Krause et al., 2022). The volatile compound content of sesame hulls was significantly higher than whole seeds and kernels, and was about twice or triple that of whole seeds or kernels at the same roasting temperature and weight. This was because sesame hulls, which also have the raw material base to produce flavor compounds, contain a lot of carbohydrates and a small amount of protein and lipids. Sesame hull is thinner than sesame seeds and sesame kernels and can absorb heat faster at the same heating conditions. This made it easier for these raw materials in sesame hulls to interact with each other, resulting in Maillard and caramelization reactions to produce volatile compounds. At 160 °C, the relative content of volatile compounds in sesame seeds was 2854 μg/kg lower than that of sesame kernels. This may be because lignin-carbohydrate compounds are formed by covalent bonds between phenolic components, carbohydrates, and lignin, thereby enhancing the

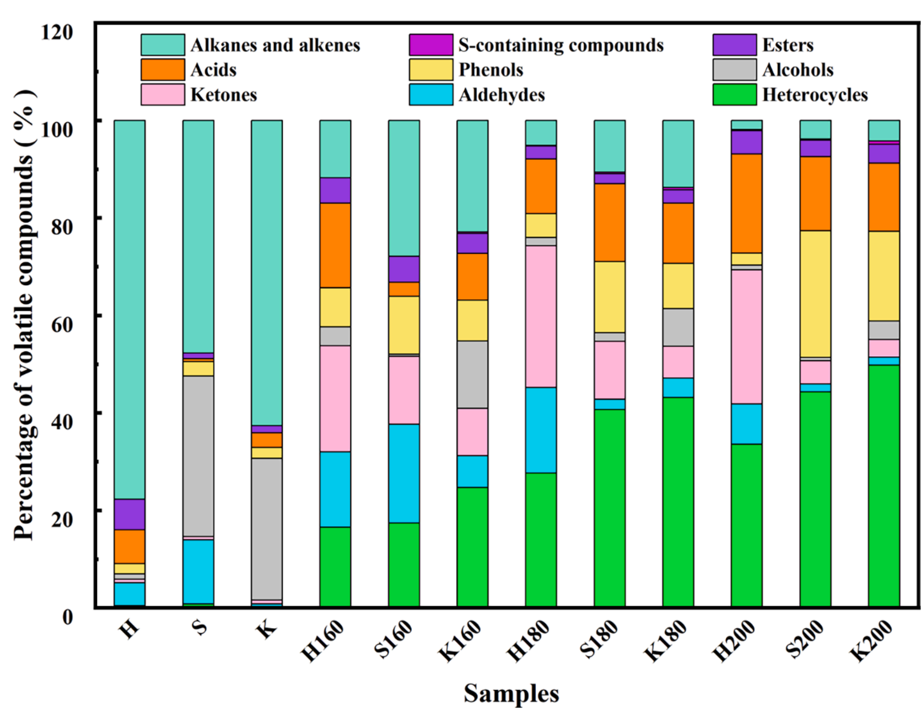

Fig. 1. Percentage of volatile compounds identified in the 12 samples (H, S, and K stand for sesame hulls, whole sesame seeds, and kernels, respectively; 160, 180, and 200 stand for roasting temperatures of 160 °C, 180 °C, 200 °C, respectively, for 30 min).

Table 1
Number of volatile compounds and total relative content of volatiles identified in the 12 samples.

Samples	Number of Volatile Compounds	Total relative Content (μg/kg)[B]
H[A]	78	33286 ± 1833[f]
S	74	28164 ± 798[f]
K	80	29785 ± 1767[f]
H160	146	134077 ± 10051[e]
S160	127	49949 ± 1595[f]
K160	128	52803 ± 2095[f]
H180	147	335100 ± 10785[c]
S180	148	182349 ± 4960[d]
K180	140	117871 ± 3831[e]
H200	161	818325 ± 26036[a]
S200	131	404264 ± 10280[b]
K200	145	321809 ± 17555[c]

[A] Abbreviation: H, sesame hulls; S, whole sesame seeds; K, sesame kernels; 160, 180, and 200 indicate sample roasting temperatures of 160 °C, 180 °C, and 200 °C, respectively.
[B] Data is means ± standard deviation. The different small letters in a row represent the significant difference ($P < 0.05$).

cohesion between the cell wall polymers (He et al., 2022). The tight structure of the hull plays a protective role for the whole seed. The relative content of volatile substances in S180 was higher than 64478 μg/kg in K180. At 200 °C, the relative content of volatiles in sesame seeds was 404264 μg/kg, which was more than those of sesame kernels. This was because the structure of the sesame hulls was damaged by long-term high temperatures, leading to the intensification of reactions such as non-enzymatic browning and lipid oxidation (Guo et al., 2022). Sesame seeds produced more phenols than sesame kernels (Fig. 1). This was because the seeds include hulls, and when heated, the lignin in the hulls cracked and produced many phenolic compounds (He et al., 2022). It should be noted in particular that the concentration of phenols detected in sesame hulls at the same weight was lower than that of whole sesame seeds and sesame kernels. This is inconsistent with previous studies which found that sesame hulls were the main source of phenols in sesame (Shahidi et al., 2006). The reason may be that the detection methods used were different. In the study of Shahidi et al. (2006), all compounds that are miscible with methanol and have reducing properties are categorized as phenols, while only volatile phenolic compounds were detected in this study. Another reason may be that a large amount of vanillin was detected in sesame hulls in this study. Vanillin contains carbonyl and phenolic hydroxyl groups and has a certain antioxidant capacity; here we classified it as an aldehyde.

With increasing heat, the proportion of alkanes and alkenes in the volatiles produced by all three sesame materials gradually decreased, and the proportion of heterocyclic substances gradually increased (Fig. 1). In sesame hulls, the alkanes and alkenes in sesame hulls decreased from 77.7 % to 1.9 %; in whole seeds, the proportion declined from 47.8 % to 3.8 %; and in kernels it decreased from 62.6 % to 4.2 %. The proportion of heterocyclic compounds in sesame hulls increased from 0.5 % to 33.6 %; in whole seeds, it rose from 0.8 % to 44.3 %, and in kernels, it increased from 0.3 % to 49.8 %. The main reason is that the Maillard and caramelization reactions generate heterocyclic compounds, and higher temperature intensifies these reactions (Guo et al., 2022).

3.2. Comparison of aroma-active components

Among the 529 volatile compounds tested, a total of 140 aroma-active compounds were found by sniffing and consulting related information. As shown in Table S1, 101 aroma-active substances were detected in sesame hulls, specifically 6 pyrazines, 8 pyrroles, 3 pyridines, 23 furans, 22 aldehydes, 19 ketones, 3 alcohols, 7 phenols, 2 acids, 6 esters, and 2 S-containing compounds. In comparison, 95 species were detected in sesame seeds, namely 21 pyrazines, 10 pyrroles, 7 pyridines, 17 furans, 4 thiazoles, 2 thiophenes, 9 aldehydes, 11 ketones, 1 alcohol, 7 phenols, 1 acid, 3 esters, and 2 S-containing compounds. 91 species were detected in sesame kernels, which were 21 pyrazines, 8 pyrroles, 8 pyridines, 17 furans, 4 thiazoles, 2 thiophenes, 11 aldehydes, 7 ketones, 2 alcohols, 5 phenols, 1 acid, 2 esters, and 3 S-containing compounds.

Differences in the concentrations of the aroma-active compounds in different samples are shown in Fig. 2A. As the roasting temperature increased, the color of each corresponding substance in the thermogram samples gradually approached red, indicating that the concentration of aroma-active substances gradually increased with the increase of temperature. Sesame hulls mainly experienced an augmentation in the concentration of furans, ketones, aldehydes, esters, and phenolic components, while whole sesame seeds mainly exhibited an increase in the concentration of pyrazines, some furans, and phenols. In sesame kernels, the concentration of pyrazines, thiazoles, and pyrroles was increased. Fig. 2B shows the differences in the content of key aroma-active compounds among the samples. It can be seen that nitrogenous heterocycles (pyrazines and pyridine), sulfur heterocycles (thiophenes and thiazoles), phenols, and sulfides have an important contribution to the aroma characteristics of sesame kernels; nitrogenous heterocycles (pyrazines and pyrroles), sulfur heterocycles (thiophenes and thiazoles) and phenols have a greater influence on the aroma of sesame seeds, while oxygenated heterocycles (furans), aldehydes, ketones, and acids play an important role in the flavor of the hulls.

3.2.1. Heterocyclic compounds

Heterocyclics, an important class of aromatic compounds, were the main aroma-active substances identified by GC-O-MS. They included pyrazines, pyrroles, pyridines, furans, and small amounts of thiophenes and thiazoles (Table S1). Studies have shown that the content of heterocyclics is positively correlated with roasting temperature (Jia et al., 2019; Mao, Zhao, Huyan, Liu, & Yu, 2019). Among the heterocyclics, those containing oxygen occupied the main position in sesame hulls, and heterocyclics containing nitrogen were of the highest concentration in sesame seeds and kernels. Sulfur-containing heterocyclic compounds are mainly produced by the roasting of sesame seeds and kernels.

Pyrazines, nitrogen-containing heterocyclic compounds, mainly exhibit roasted and nutty aromas (Yin et al., 2021). The unroasted samples had no pyrazines, while S200 had the highest concentrations (accounting for 38.8 % of all aroma-active compounds). Compounds ***1*–*4*** were the main pyrazines in sesame hulls. Their concentrations increased greatly at 200 °C; in particular, the concentration of 2-methyl-pyrazine (***2***) grew from 0 to 1289 μg/kg (Table S3). Ethyl-pyrazine (***3***) is one of the compounds used to identify fraud involving sesame oil raw materials and production processes (Liu et al., 2022). 2,6-Dimethyl-pyrazine (***5***) was detected only in H160, and 2-ethyl-6-methyl-pyrazine (***6***) was found in H200. These substances have been reported as aroma-active substances of sesame oil (Yin et al., 2021). The pyrazine compounds with high concentrations in S160 were pyrazines ***7***, ***8***, ***11***, and ***13***. Among them, pyrazine ***13*** was identified as a key aroma-active compound in S160. Pyrazines ***3***, and ***15*–*20*** were new compounds in S180 relative to S160. Pyrazines ***21*** and ***22*** were new aroma-active substances presented in S200. There was no significant difference in the concentration of pyrazines among aroma-active compounds of whole seeds and kernels at 160 °C and 180 °C. Compared with S160, the slight difference was that the substances of aroma contribution in K160 included ethyl-pyrazine (***3***), 2,3-diethyl-5-methyl-pyrazine (***11***), (E)-2-methyl-5-(1-propenyl)-pyrazine (***16***), 2-ethyl-3-methyl-pyrazine (***17***), 2-ethenyl-6-methyl-pyrazine (***18***) and 2,3-dimethyl-5-ethylpyrazine (***21***). 2,6-Diethyl-pyrazine (***9***), (E)-2-methyl-6-(1-propenyl)-pyrazine (***10***), and 2-ethenyl-6-methyl-pyrazine (***18***) were detected in S180. However, they were not detected in K180. The content of pyrazines in sesame hulls was significantly lower than that in whole seeds and kernels at 200 °C. The fewest pyrazines were found in hulls, and the content of pyrazines in whole sesame seeds and kernels was similar. At 160 °C, most pyrazines

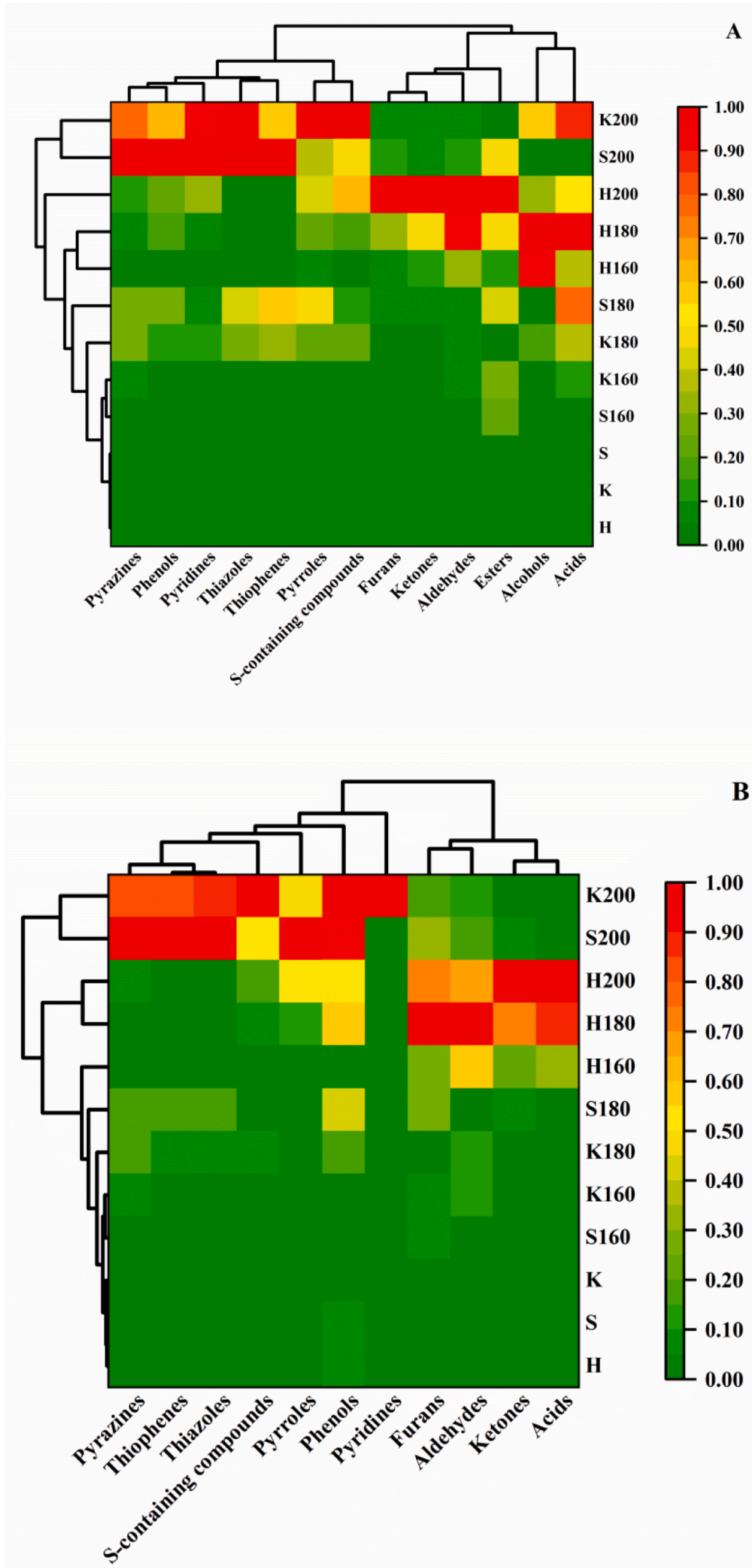

Fig. 2. Content of aroma-active compounds (A) and key aroma-active compounds (B) identified in the 12 samples (H, S, and K stand for sesame hulls, whole sesame seeds, and kernels, respectively; 160, 180, and 200 means that the corresponding sample was roasted at 160 °C, 180 °C, 200 °C, respectively, for 30 min).

were produced in kernels, while at 200 °C they were the highest in seeds. The reason may be that pyrazines are mainly produced through the Maillard reaction, which is a non-enzymatic browning reaction that involves the hydroxyl groups of sugar compounds and the carbonyl groups of amino acids (Yu, Seow, Ong, & Zhou, 2018). In our test, the protein content in hulls was only 6.8 %, which was far lower than in kernels and whole seeds. At relatively low temperatures, there were relatively lower levels of reactions in whole seeds due to the protection of the hulls; at the high temperature of 200 °C, the hulls were cracked such that the degree of reactions greatly increased. Eight of the 22 pyrazines were identified as key aroma-active compounds in all samples (Table 2), including pyrazines ***1***, ***3***, and ***11*** (in whole sesame seeds and kernels at 200 °C); ***2*** (in all samples at 180 and 200 °C); ***8*** (in S200); ***13*** (in roasted whole sesame seeds and kernels); ***15*** (in K180 and K200); ***19*** (in whole sesame seeds and kernels at 180 and 200 °C); ***8*** (in S200). These substances mainly affect the aroma of the whole seeds and kernels, giving them a strong roasted and nutty odor.

Furans, which are oxygen-containing heterocyclic compounds, mainly contribute caramel, sweet and fruity aromas. Furan content depends on the original material and the heating temperature (Ramírez et al., 2022). A small number of furans was detected in unroasted sesame hulls and seeds (Fig. 2). The content of furans rose as the roasting temperature increased. As shown in Table 2, furans ***47*** (sour and delicate fragrance, OAV = 2), and ***53*** (caramel-like and burnt, OAV = 1) were largely responsible for the overall flavor of H160. For H180, the important contributions to flavor were made by furans ***47*** (OAV = 2), ***52*** (caramel-like and smoky, OAV = 1), ***53*** (OAV = 3), and ***56*** (caramel-like, OAV = 80). Furans ***47*** (OAV = 2), and ***52*** (OAV = 2) played a major role in the flavor of H200. Furan ***47*** is produced by the oxidation of 2-acetylbutyrolactone (Ragoussis, Lagouvardos, & Ragoussis, 1998). Albouchi and Murkovic (2017) noted that ***52*** is produced from the products of oxidative cleavage of glucose by further dehydration cyclization. Furan ***53***, a common product of the Maillard reaction, is produced by the degradation of maltose (Wietstock, Baldus, Öhlschläger, & Methner, 2017). Furan ***56*** is formed directly from D-fructose, probably employing dehydration and reduction reactions (Schwab, 1998). Compared to seeds and hulls, sesame kernels had a relatively low content of furans. Formation of furans usually occurs through the degradation and interaction of carbohydrates and amino acids in the Maillard reaction (Kim, Park, Moon, Lee, & Kim, 2023) and caramelization reactions (Srivastava et al., 2018). Saccharide is an important raw material in these reactions. Compared with sesame seeds and kernels, hulls had the highest ratio of saccharides (about 43.0 % polysaccharides) (Zhang et al., 2022). The content of furans in sesame seeds was higher than that in kernels under the same heat treatment condition because of the existence of hulls.

The pyrrole and pyridine compounds detected in sesame hulls were also detected in the other two raw materials (Fig. 2). Among them,

Table 2
The key aroma-active compounds with odor activity values (OAV) ≥ 1 in 12 samples.

NO.	Compounds	OT[A] (μg/kg)	OAV[B]											
			H[C]	S	K	H160	S160	K160	H180	S180	K180	H200	S200	K200
1	2,3-Dimethyl-pyrazine	100[D]	ND	ND	ND	<1	<1	<1	ND	<1	<1	<1	2	1
2	2-Methyl-pyrazine	200[E]	ND	ND	ND	<1	<1	<1	1	1	1	1	6	5
3	Ethyl-pyrazine	200[D]	ND	ND	ND	<1	ND	<1	<1	<1	<1	<1	1	1
8	2,5-Dimethyl-pyrazine	2000[E]	ND	ND	ND	ND	<1	<1	ND	<1	<1	ND	1	<1
11	2-Ethyl-5-methyl-pyrazine	320[G]	ND	ND	ND	ND	<1	<1	ND	<1	<1	ND	1	1
13	3-Ethyl-2,5-dimethyl-pyrazine	79[E]	ND	ND	ND	ND	1	2	ND	3	4	ND	9	11
15	2,3-Diethyl-5-methyl-pyrazine	0.5[E]	ND	ND	ND	ND	ND	<1	ND	<1	6	ND	ND	42
19	Acetyl-pyrazine	16[F]	ND	ND	ND	ND	ND	ND	ND	5	1	ND	12	7
23	1-(2-Furanmethyl)-1H-pyrrole	20[E]	ND	ND	ND	ND	ND	ND	<1	ND	ND	1	1	1
41	2-Pentyl-pyridine	0.6[E]	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	65
47	2-Methyltetrahydrofuran-3-one	23[E]	ND	ND	ND	2	ND	<1	2	<1	<1	2	2	ND
52	2-Furan-methanol	400[E]	ND	ND	ND	<1	<1	<1	1	<1	<1	2	2	2
53	Furfural	700[E]	ND	ND	ND	1	<1	<1	3	<1	<1	<1	1	<1
56	Furaneol	4[E]	ND	ND	ND	ND	40	103	80	201	ND	ND	52	ND
73	4-Methylthiazole	20[E]	ND	ND	ND	ND	ND	ND	ND	1	<1	ND	6	5
75	Dihydro-3-(2H)-thiophenone	10[E]	ND	ND	ND	ND	ND	ND	ND	1	<1	ND	2	1
76	Pentanal	150[E]	ND	ND	ND	2	ND	ND	3	ND	ND	ND	ND	<1
78	2-Methyl-butanal	23[E]	ND	ND	ND	23	4	13	21	5	15	22	15	14
81	Phenylacetaldehyde	9[G]	<1	<1	<1	3	5	1	5	1	<1	11	1	ND
84	Nonanal	260[E]	<1	<1	<1	1	<1	<1	2	<1	<1	3	1	ND
86	Vanillin	50[E]	ND	ND	ND	13	2	ND	34	4	1	45	ND	ND
87	2-Methyl-propanal	3.4[E]	ND	ND	ND	42	ND	ND	36	ND	ND	41	ND	ND
88	3-Methyl-butanal	10.8[E]	ND	ND	ND	36	2	11	124	ND	7	35	12	8
90	Heptanal	50[E]	ND	ND	ND	1	ND	ND	2	ND	ND	2	ND	ND
91	Hexanal	100[E]	1	<1	ND	3	<1	<1	5	<1	<1	3	<1	<1
92	Octanal	56[E]	ND	ND	ND	2	ND	ND	4	ND	ND	5	ND	ND
95	(E,E)-2,4-Decadienal	70[F]	ND	ND	ND	ND	ND	ND	2	ND	ND	ND	ND	ND
104	3-Hydroxy-2-butanone	120[E]	ND	ND	ND	3	1	<1	19	1	<1	46	2	<1
108	Hydroxyacetone	800[E]	ND	ND	ND	1	<1	<1	2	<1	<1	<1	ND	<1
124	2-Methoxy-phenol	13[E]	ND	ND	ND	2	ND	ND	6	9	5	36	109	84
125	2-Methoxy-4-vinylphenol	5[G]	ND	7	6	ND	ND	<1	160	85	31	78	ND	95
127	Phenol	10[E]	9	9	ND	ND	ND	ND	ND	9	8	ND	ND	ND
129	Pentanoic acid	600[E]	ND	ND	ND	11	ND	ND	31	ND	ND	35	ND	ND
138	Dimethyl disulfide	4.2[G]	ND	ND	ND	1	ND	<1	2	1	3	ND	20	29
139	Dimethyl trisulfide	3[E]	ND	ND	ND	ND	ND	<1	1	<1	<1	10	ND	6
140	2-Methyl-1-butanethiol	0.1[E]	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	170

[A] OT, Odor threshold of aroma compounds in oil or water, the lowest concentration that people can perceive.

[B] Odour activity values (OAV) = $\frac{C}{T}$, C is the absolute concentration of the relevant compound quantified by external calibration. T is odor threshold.

[C] H, sesame hulls; S, whole sesame seeds; K, sesame kernels. 160, 180, and 200 respectively indicate that the sample roasting temperatures were 160 °C, 180 °C, and 200 °C. ND, not detected.

[D, E, F, G] Odour thresholds (OT) from the following works and literatures: [D] Yin et al., 2021; [E] the book Odor Thresholds: Compilations of flavour threshold values in Water and Other Media (second enlarged and revised edition) (van Gemert, 2011); [F] Matheis & Granvogl, 2016; [G] Jia et al., 2019.

pyrrole ***23*** (caramel-like and nutty, OAV = 1 in samples at 200 °C), the Maillard reaction product from glucose and aspartic acid, has been identified as an aromatic substance of roasted sesame oil (Yin et al., 2021). Zhou and Boatright (2000) use of the stable isotopes 13C-2,4-decadienal and 15 *N*-ammonia indicated that both compounds are precursors for the formation of pyridine ***41*** (fatty and burnt, OAV = 65 in K200). Pyrroles ***31***, ***32*** and pyridines ***36***–***41*** were found only in sesame seeds and kernels. Thiazoles and thiophenes were not found in seeds and kernels roasted at 160 °C and were not identified in any of the roasted sesame hulls. Thiazole ***73*** (roast and smoky) and thiophene ***75*** (roast and caramel-like) were identified as key aroma substances in S200 and K200 (Table 2).

3.2.2. Aldehydes

Aldehydes are common volatile compounds, usually produced through lipid oxidation reactions (Ivanova-Petropulos et al., 2015); they typically have green, nutty, and fatty odors (Dun et al., 2019). For all groups, the content of aldehydes in hulls was generally higher than that in whole seeds and kernels. It is worth mentioning that vanillin (fatty and sweet milk, OAV > 10 in roasted sesame hull), produced by oxidative cracking of phenolic lignin, which has a unique flavor, and which has attracted much attention in recent years, was found in this research. The data in Table 2 shows that the concentration of vanillin produced in sesame hulls was vastly higher than that in sesame kernels and whole sesame seeds. As the roasting temperature rose, vanillin content in sesame hulls increased gradually. A total of 12 aldehydes were identified as key aroma-active compounds in all samples of this study. It was worth noting that these compounds mainly affected the flavor characteristics of sesame hulls (Table 2). Among them, aromatic aldehyde ***81*** (almond, fatty, and cocoa-like, OAV ≥ 3 in hulls) is a typical Strecker aldehyde that is degraded by phenylalanine in the Maillard reaction (Gu, Jin, Schwarz, Rao, & Chen, 2022). Branched-chain aldehydes ***87*** (green and fruity, OAV > 30 in hulls) and ***88*** (fruity, OAV > 30 in hulls) are breakdown products of leucine and ***88*** can also be generated by Amadori rearrangement in the Maillard reaction (Smit, Engels, & Smit, 2009). In contrast, there were smaller amounts and lower OAVs of key aroma-active aldehydes in whole seeds and kernels. Aldehydes contributed much less to the overall aroma of whole sesame seeds and kernels than sesame hulls.

3.2.3. Ketones and alcohols

For the unroasted samples, only one ketone was detected in the sesame hull. At 160 °C, ketones ***101***, ***102***, and ***105*** were detected, and again, only in the sesame hull. Prior research has shown that 2-heptanone (***102***) is the primary product of oxidizing linoleic acid and that it is an aromatic component of microwaved sesame oil (Jia et al., 2019). The fatty acids of sesame hulls are about 37 % linoleic acid, which provides the material basis for the production of 2-heptanone. Because sesame hulls absorb heat faster than either seeds or kernels, as a Maillard reaction product (Huang et al., 2011), 2,3-pentanedione (***101***) could be produced at low temperatures. Compared to H160, H180 contained 2-hydroxy-3-pentanone (***103***), acetone (***106***), 2-butanone (***107***), and 4-methyl-2-pentanone (***110***), a newly detected ketone compound. S180 had 6 ketones more than S160, and sesame kernels had 2,3-pentanedione (***101***) at 180 °C. The content of ketones in H200 was about ten times that of S200. From 160 °C to 180 °C, the content of hydroxyacetone (***108***) in samples increased. Hydroxyacetone, a derivative of natural cellulose (Fukutome, Kawamoto, & Saka, 2014), is responsible for caramel and burnt flavors (Reichel et al., 2021). The OAVs of the ketones identified as key aroma-active compounds in seeds and kernels were lower than those of the hulls at all roasting temperatures. Therefore, compared with whole seeds and kernels, the flavor of sesame hulls was most affected by ketones. Three alcohols were detected in all samples, namely 1-dodecanol (***119***), 1-octanol (***120***), and 1-pentanol (***121***). Alcohols ***120*** and ***121*** have been detected in sesame oil before (Jia et al., 2019). None of the alcohols were identified as key aroma-active compounds, suggesting that the alcohols contributed weakly to the odor of the samples.

3.2.4. Phenols

Phenols generally have a smoky smell. At 160 °C, the content of phenols in sesame hulls was the highest, and at 180 °C, it was the highest in seeds. At 200 °C, the content of phenolic compounds detected in hulls was the lowest. These phenolic compounds are mainly produced through lignin degradation. On the whole, the concentration of phenols gradually increased with increased roasting temperature. Only 2-methoxy-4-vinylphenol (***125***) and phenol (***127***) were detected in the unroasted samples. With increasing roasting intensity, the concentrations of 2-methoxy-phenol (***124***), which has a characteristically smokey flavor, increased and the concentrations of 2-methoxy-4-vinylphenol (***125***) declined in sesame seeds. This phenomenon was consistent with a previous study (Dong et al., 2012). Phenols ***124*** and ***125*** were determined in a previous study as aromatic substances vital to the flavor and aroma of roasted sesame oil and seeds (Cadwallader & Heo, 2001). In the study, these two phenols were identified as key aroma-active compounds in roasted sesame hulls, whole seeds, and kernels (Table 2). Sesamol was also detected in the roasted samples. Sesamol is produced from the degradation reaction of sesamolin at high roasting temperatures (Tamura et al., 2010). 2,3-Dimethoxyphenol was detected in sesame hulls and seeds. Maltol is an aromatic substance, newly found in sesame; it has a flavor like caramel.

3.2.5. S-containing compounds

The sulfides have roasted, meat-like, and fat flavors (Jo, Carter, Barbano, & Drake, 2019). It is assumed that the amino acids such as cysteine, cystine, and methionine in sesame seeds are matrixes for the formation of many S-containing aromatic active compounds by Strecker degradation or the Maillard reaction (Jia et al., 2019; Yin et al., 2022). S-containing compounds generally have a low threshold of perception, so humans can readily detect them even at low concentrations. Therefore, when present, they contribute noticeably to the overall flavor (Sun, Wang, & Sun, 2021). In addition to the heterocyclic compounds (thiophene, thiazole) introduced earlier, dimethyl disulfide (***138***), dimethyl trisulfide (***139***), and 2-methyl-1-butanethiol (***140***) were sulfides identified in this study. 2-Methyl-1-butanethiol was detected only in K200 at a concentration of 17 μg/kg (Table 2S). Sulfides including thiophene and thiazole heterocyclic compounds were more likely to be produced from sesame seeds and kernels, followed by sesame hulls, at the same temperature.

3.2.6. Acids and esters

Acids and esters are aroma-active compounds that generally contributed less to the overall flavor of samples in this study. Results here showed that the aroma-active pentanoic acid (***129***) was detected only in roasted sesame hulls, and as the roasting temperature rose, the concentration also rose. Acid ***129*** typically contributes sweaty, pungent, and vinegar-like aroma notes (Matheis & Granvogl, 2016). Acid ***129*** (OAV > 10 in roasted sesame hulls) was confirmed as a key aromatic compound only in hulls. There was a tremendous increase after roasting (0 μg/kg for unroasted sesame hulls, 6870 μg/kg at 160 °C, 18702 μg/kg at 180 °C and 21272 μg/kg at 200 °C). As the roasting temperature rose, the acetic acid content first increased and then decreased in hulls and seeds, but consistently increased in kernels. Acetic acid was present in all materials. Only esters ***134*** and ***137*** were detected in sesame kernels. In addition to the above two esters, 3-hydroxy-methyl-propanoate (***131***) was also detected in whole sesame seeds. Esters ***131***–***133*** and ***136*** were monitored in sesame hulls. In terms of ester concentration under different conditions, as Chai et al. (2019) and Mao, Zhao, Huyan, Liu, and Yu (2019) said heating contributed to ester formation.

3.3. PCA analysis of main aroma-active compounds

PCA is a statistical analysis based on multiple variables, and, in the case of flavor compounds, it can depict the relationship between samples using differences in flavor substance concentration or odor strength as variables (Wang et al., 2023). The key aroma active compounds of sesame hulls, sesame kernels, and whole sesame seeds were classified at different roasting levels and then analyzed by PCA to explore the differences in aroma between sesame hulls and the other two materials at different roasting temperatures. As shown in Fig. 3, the cumulative contribution of PC1 and PC2 was over 85.0 %. It indicated that most of the information for characterizing the flavor components of the samples can be obtained by PCA. The parallel samples were close, which indicated that the samples were reproducible. As can be seen in Fig. 3A, the samples after roasting at 160 °C were relatively close to the unroasted samples, which suggested that there was less accumulation of flavor substances at this point, which had relatively little effect on the odor of the samples. The differences in flavor profile between K160 and S160 were less significant, and H160 was significantly different from the other two about aroma (Fig. 3B). This is mainly because sesame seeds and kernels produced very low levels of phenols, acids, and sulfides (which affect PC1 positively). Differences in flavor profiles of sesame hulls, whole seeds and kernels were not significant under 180 °C roasting conditions (Fig. 3C). At 200 °C, the flavor profiles of sesame seed hulls were somewhat similar to those of both whole seeds and kernels, while the seeds and kernels showed significant differences in flavor presentation, with the substances responsible for such differences mainly including pyrroles and furans in S200 (Fig. 3D).

3.4. Comparison of aroma profiles

Because different compounds have different odor thresholds, simply comparing the content of key flavor compounds does not accurately reflect the aroma profile of a sample. Therefore, sensory evaluation was applied in this study. The sensory properties of sesame hulls, seeds, and kernels under different roasting conditions were determined and rated for intensity by 10 trained panelists. It can be seen that at the same temperature, Roasted seeds had a stronger caramel-like aroma than kernels, and sesame hulls had a weaker fatty and roasted odor (Fig. 4). The S and K had a strong green relish, and mild nutty overtones, together with a somewhat woody and raw sesame seed odor. The H had a medium-intensity woody odor, a weak green, and a raw sesame seed

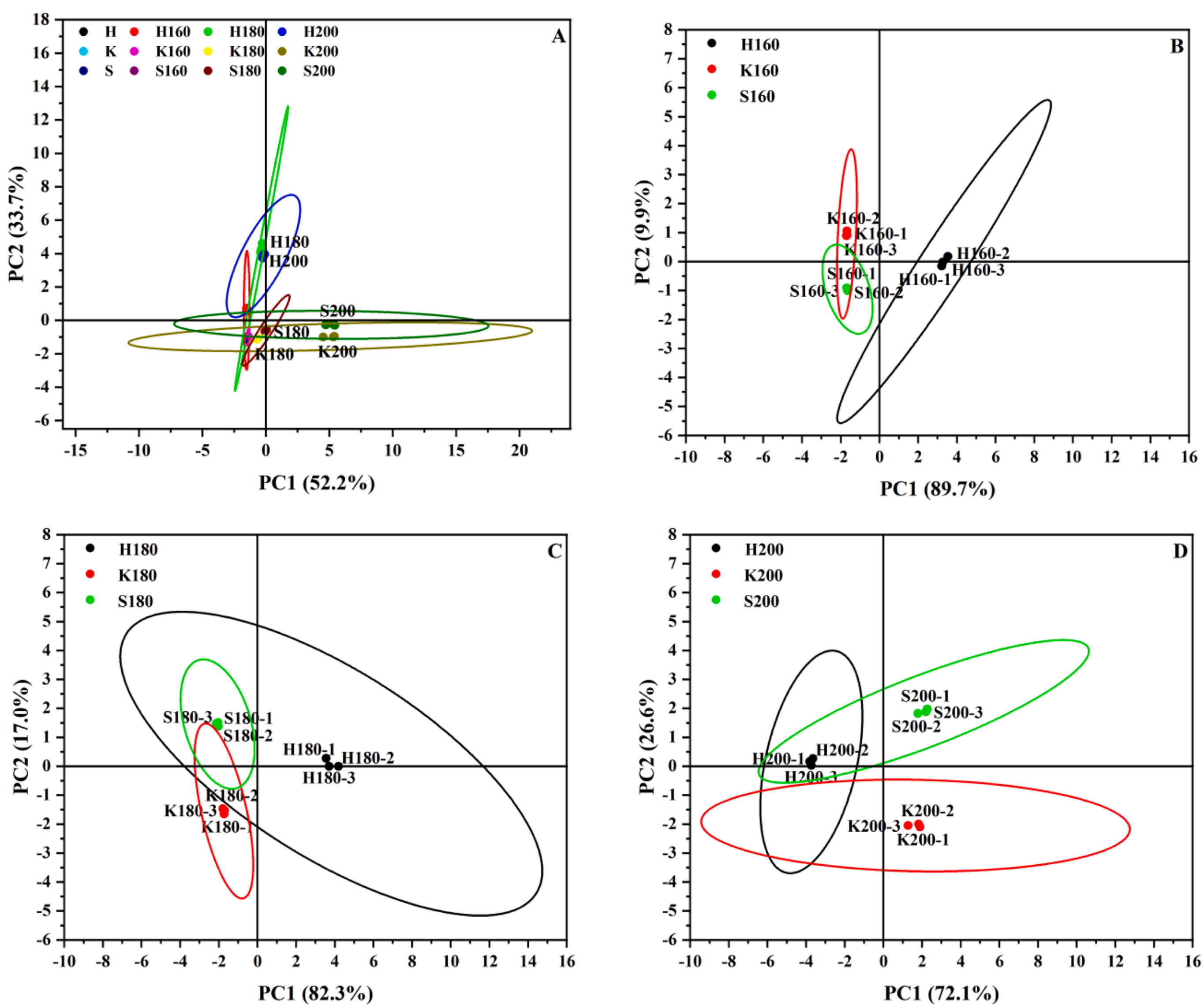

Fig. 3. PCA analysis of key aroma-active components of sesame hulls (H), whole seeds (S), and kernels (K) with different roasting degrees (160, 180, and 200 means that the corresponding sample was roasted at 160 °C, 180 °C, 200 °C, respectively, for 30 min).

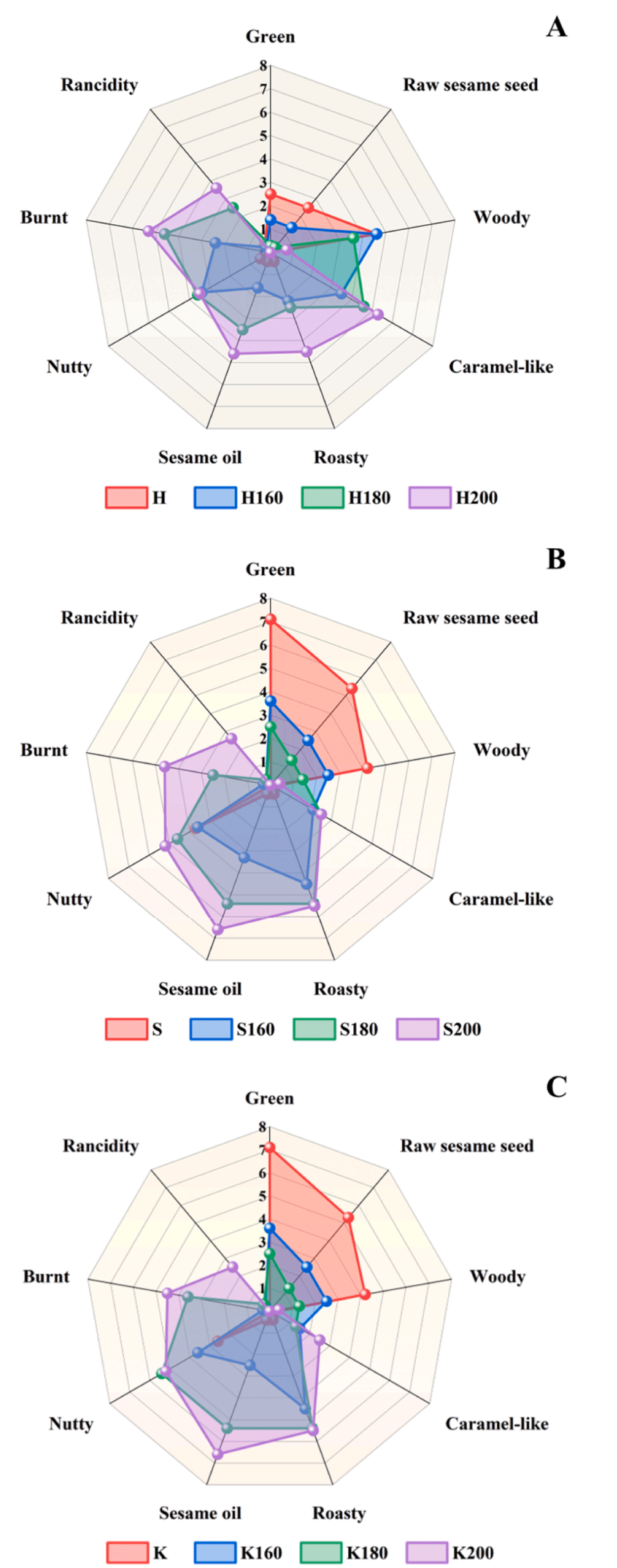

Fig. 4. Aroma profiles of sesame hulls (H), whole seeds (S), and kernels (K) under different roasting conditions (160, 180, and 200 means that the corresponding sample was roasted at 160 °C, 180 °C, 200 °C, respectively, for 30 min). The number indicates the subjective evaluation of the flavor by the average score of the panelists, with 0 indicating that the odor was not smelled and 8 indicating that the odor was given as very strong.

flavor. After roasting at 160 °C, the green, raw sesame seed and woody notes of both seeds and kernels gradually weakened, as the roasted and fatty aroma increased. This is the result of the action of the heterocyclic compounds produced by the roasting of the samples. It mainly showed the flavors of fat, roast, and nut in S180 and K180. In comparison, H180 had a distinct caramel and burnt flavor. It is mainly the result of the large accumulation of furans in the roasted sesame seed hulls and the highest phenolic content of the sesame seed hulls. Phenolic compounds have a low threshold and can still exhibit phenolic odor characteristics (burnt, smoky) even at low concentrations differences. And the OAV of phenol ***125*** in H180 was 160 which explains the pronounced burnt flavor of sesame hulls. At 200 °C, sesame hulls had a medium-strength fatty aroma, with a relatively strong caramel and burnt smell which was mainly the contribution of aldehydes, phenols, and acids (Table 2). Due to the effect of heterocycles and phenols, whole sesame seeds had strong roasted and fatty flavors, as well as medium-intensity burnt flavors. Sesame kernels had a strong fatty and roasted aroma because of the influence of *N*-containing heterocycles and sulfides.

4. Conclusion

In this study, the aroma-active compounds produced by sesame hulls after roasting at different temperatures were identified, and the whole sesame seeds and sesame kernels under the same conditions were used as blank controls. The results showed that compared with the whole seeds and kernels, sesame hulls could produce the most volatile compounds after roasting under the same conditions, but the composition of these compounds in hulls was different from the other two. This difference was mainly manifested in the production of furans and aldehydes after sesame hulls roasting, while the production of *N*-containing heterocycles (pyrazines, pyrroles, and pyridines) after whole seeds and kernels roasting. Among the 36 compounds identified with OAV $\geq$ 1, *N*-containing heterocycles were all present in sesame whole seeds or kernels, among which only pyrazine ***2*** and pyrrole ***23*** (containing furan rings) were identified as the key aroma compound of H200. Furans mainly existed in sesame hulls and whole seeds, and furan ***56*** (caramel-like note, OAV > 40) contributed the most to the aroma. Aldehydes, ketones, and acids mainly affect the flavor of sesame hulls. Aldehydes ***76***,***87***,***90***–***92***, ***95***, ketone ***108***, and acid ***129*** were identified as the key aroma substances of sesame hulls. Phenolic compounds and sulfides contributed to the aroma of all three materials. Phenol ***124*** contributed more to the odor of sesame seeds, phenol ***125*** played a major role in the aroma of sesame hulls, and sulfide ***140*** had a great effect on the flavor of sesame kernels. These results identified compounds that contributed significantly to the flavor of sesame hulls and elucidated the contribution of sesame hulls to the flavor of roasted whole seeds and sesame oil by comparing sesame hulls with whole seeds and kernels.

CRediT authorship contribution statement

Rui Wang: Writing – original draft, Validation, Investigation, Formal analysis, Data curation. **Lin-Xuan Wu:** Investigation, Formal analysis. **Bing-Xin Guo:** Investigation. **Peng-Hao Zhao:** Investigation. **Wen-Ting Yin:** Supervision, Resources, Formal analysis. **Hua-Min Liu:** Writing – review & editing, Supervision, Resources, Funding acquisition. **Hong-Xian Mei:** Resources, Funding acquisition. **Ying-Hui Duan:** Resources, Funding acquisition.

Declaration of competing interest

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

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.fochx.2024.101203.

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