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Characterization of volatile organic compounds in walnut oil with various oxidation levels using olfactory analysis and HS-SPME-GC/MS

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ABSTRACT

Walnut oil oxidizes and becomes rancid during storage, that could be significantly affecting flavor and quality. This study aimed to monitor the volatile compounds present in walnut oil during storage, identify the characteristic markers of walnut oil at different oxidation levels, and establish a correlation network analysis based on the relationship between the olfactory analyzer and the characteristic markers to understand their correlation. The results indicated that the oxidation level of walnut oil had a positive correlation with the response of the olfactory analyzer. 219 volatile compounds were identified in walnut oil, with 89 identified as key volatile compounds (VIP >1). Among these, compounds such as (E, E)-2,4-decadienal (6.10%–23.04%) , (E, E)-2,4-heptadienal (2.23%–13.61%) , (E)-2-octenal (0.95%–11.71%) , hexanoic acid (1.63%–4.30%) , 1-octen-3-ol (2.53%–19.01%) , (Z)-2-heptenal (5.95%–25.01%) , 2,3-dihydro-furan (1.08%–3.20%) , 2-pentyl-furan (0.13%–0.54%), pyrazine (0.33%–1.32%), hexanal (24.52%–1.33%) , 3-hethylbutylacetate (12.44%–1.29%) , 2-methyl butyl acetate (7.74%–1.56%) and ethenyl hexanoate (4.39%–0.41%) were found to be characteristic volatile compounds in the oxidation process of walnut oil. Furthermore, the correlation network analysis revealed a strong correlation between the olfactory analyzer sensors and the characteristic volatile compounds. The findings of this study can provide valuable data for the development of rapid determination of the oxidation level of walnut oil.

1. Introduction

As a valuable vegetable oil, walnut oil is widely cultivated and consumed worldwide and is highly respected for its rich nutritional value and unique flavor. Walnut oil contains many unsaturated fatty acids, such as linolenic acid and linoleic acid, which are incredibly beneficial to human health. In addition, walnut oil contains proteins, vitamin E, and minerals such as calcium and magnesium, which make walnut oil an essential part of the daily diet (Gao et al., 2024; Liu et al., 2023). However, due to the high unsaturated fatty acid content of walnut oil, it is susceptible to external environmental factors (e.g., temperature, light, and oxygen) as well as internal factors (moisture and enzymes) during processing and storage, which can lead to complex and diverse chemical reactions such as meladic reactions, thermal degradation of fats, and product crossover of lipids (Huo et al., 2024; Rébufa et al., 2022), leading to a decrease in the safety of walnut oil consumption and even cancer induction (Cai et al., 2021; Huo et al., 2024; Li et al., 2011). Therefore, it is crucial to assess the walnut oil oxidation degree accurately. In addition, although traditional chemical testing methods such as peroxide value and acid value are relatively mature and widely used, they have problems such as complicated operation processes, high requirements for operators' operation experience, high testing costs, and not being environmentally friendly. Therefore, there is a need to further accurately assess the degree of oxidation and flavor characteristics of walnut oil, analyze the oxidation rancidity process, and clarify its oxidation markers, which are of great practical

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significance in applying walnut oil storage, preservation, and quality control.

In recent years, development of olfactory analyzers (Electronic Nose, E-nose) and HS-SPME-GC-MS technologies has provided new methods and tools for food flavor studies (Li et al., 2023). E-nose is a technology that mimics the human olfactory system, employing an array of chemical sensors to detect and identify 'fingerprints' of gases or volatile organic compounds (VOCs). Since the mid-1980s, E-nose technology has developed rapidly. It has been used in food and beverage quality control (Song et al., 2023), environmental monitoring (Moufid et al., 2021), disease diagnosis (Oliveira et al., 2022), agriculture (Kong et al., 2024), and many other fields that show a wide range of potential applications, but are limited by the specificity of their sensor arrays (Rasekh et al., 2022). Headspace solid-phase microextraction gas chromatography-mass spectrometry (HS-SPME-GC/MS) is an efficient and sensitive analytical technique widely used for detecting and quantifying volatile organic compounds (VOCs) in various samples. The method uses solid-phase microextraction (SPME) to extract the target compounds from the sample, followed by separation and identification by gas chromatography-mass spectrometry (GC-MS), which enables rapid and accurate analysis of VOCs in samples (Adelina et al., 2021). Nowadays, the use of these two methods in combination with each other has shown good performance in the analysis of flavor substances in food products, such as fermented chili sauce (Li et al., 2022), Harbin red sausage (Yin et al., 2021). baked soya beans (Cai et al., 2021) and roasted lamb (Cai et al., 2021; Yao et al., 2024). Xu et al. identified 1-octen-3-ol, (E)-2-decenal (OAV = 4.10), γ-dodecalactone, 2-pentyl furan, (E)-2-nonenal and pentanal as the key aroma compounds in walnut oil utilizing the flavor dilution factor (FD), the odor activity value (OAV) as well as recombination and omission experiments (Xu et al., 2023b). Xi et al. to identify the key aroma compounds in walnut oil obtained by pressing walnut raw material at different maturity levels, used chemometrics to identify 1,8-cineole, ethanol as a characteristic marker of early ripened walnut oil, nonanal (E)-2-octenol and hexanal as sources of odor in medium ripened walnut oil. The characteristic flavor of late-ripened walnut oil is caused by nonanal, 1-heptanol, and hexanal (Xi et al., 2024a). Huo et al. showed that hexanal, caproic acid, 1-pentanol, (E)-2-octenal, and 2-heptanenal were significantly different before and after the oxidation of walnut oil and could be used as its characteristic markers (Huo et al., 2024). Overall, although studies such as those mentioned above have studied the characteristic flavors of walnut oil in detail, they have not monitored the changes in flavor during the oxidation process, so it is crucial to clarify the changes in volatile compounds during the oxidation process of walnut oil and to screen for potential characteristic markers during the oxidation process of walnut oil.

Therefore, this study aimed to characterize the dynamic changes of volatile compounds in walnut oil during the oxidation process using an olfactory analyzer in combination with HS-SPME-GC/MS to screen out potential characteristic markers of walnut oil during the oxidation process to explore the correlation between its sensors and volatile compounds during the oxidation process, and to provide scientific support for the olfactory analytical technique to be used in the rapid discrimination of the oxidation degree of walnut oil.

2. Materials and methods

2.1. Materials and reagents

The walnuts were purchased from the local market in Wensu County, Aksu, Xinjiang, China, and the raw material was 'Xin 2', which was washed and drained of surface water after degreening immediately after delivery. The walnuts were dried in an AGHD-15 EL C (Foshan Aoyimei Energy Saving Equipment Co., Ltd., China) air-energy heat pump drying oven at 45 °C until the moisture was less than ≤5%. The walnut shells were broken using the 6 PK-600 (Xinjiang Agricultural Engineering Co., Ltd., China) walnut hulling machine developed by the research group. The walnut oil samples were hydraulically cold pressed using a 6YY-270 intelligent hydraulic oil press (Luoyang Luofeng Hydraulic Technology Co., Ltd., China) and stored in cold storage at 4–6 °C. The walnut oil samples were equilibrated in a BPG-9140 A (Shanghai Yiheng Scientific Instrument Co., Ltd., China) precision blower drying oven to accelerate the oxidation of the walnut oil samples. Odor detection of walnut oil samples was carried out using a JSMB-30G (Beijing Jiushihengyi Science and Technology Co., Ltd., China) smart olfactory analyzer. The chromatographic purity of cyclohexanone used for volatile compound analysis was purchased from Sigma-Aldrich (St. Louis, Missouri, USA). (Extraction fiber heads (50/30 μmPDMS/CAR/DVB) were obtained from Supelco (Bellefonte, PA, USA). All other reagents are analytically pure and obtained from Sinopharm Chemical Reagent Co. (Beijing, China).

2.2. Accelerated oxidation test

The accelerated oxidation test was referred to in previous studies with slight modifications (Tinello et al., 2018). Fresh walnut oil samples were placed into 5 L fine-mouth flasks and subjected to an accelerated oxidation test at 100 °C oven and 3 L/min oxygen flux for 48 h. The initial sample was CK, and the samples were taken every 4 h, defined as T4, T8, T12 …, and T48, and three independent replicates of the test were taken for each sample. The flow chart of the experimental design is shown in Fig. 1.

2.3. Olfactory analyzer analysis

Volatile organic compounds (VOCs) in walnut oil with different oxidation levels were analyzed using the JSMB-30 G Intelligent Olfactory Analyzer for Walnut Oil, which was developed independently by the group (Xiong et al., 2023, 2024). The instrument consists of 14 gas sensors, and the gases are passed through a filtering device and a data collection system. Table 1 lists the sensitive gases corresponding to the 14 sensors. Briefly, a 5 mL sample was taken in a 20 mL headspace chromatography injection vial, quickly sealed with a sealing film, and the odor was equilibrated for 30 min in a thermostatic drying oven at 60 °C. The sample was manually injected, and the injection needle of the Smart Olfactory Analyzer was inserted into the top of the injection vial mouth to rapidly aspirate the walnut oil's volatile gases. The detection parameters of the intelligent olfactory analyzer were set at a gas flow rate of 400 mL/min, a detection time of 90 s, an injection time of 30 s, and four independent repetitions for each sample.

2.4. HS-SPME-GC-MS analysis of volatile compounds

We analyzed walnut oil with different degrees of oxidation for volatile compounds using an Agilent 7890 A-5975C series gas chromatography-mass spectrometer (Agilent Technologies, Inc., California, USA) with a slight modification of the method of Xi et al (Jia et al., 2023; Xi et al., 2024b). In the HS-SPME procedure, a weighed 2 g walnut oil sample was placed in a 15 mL extraction vial and equilibrated at 60 °C for 15 min. The sample was adsorbed for 30 min under magnetic stirring at a stirring speed of 500 rpm using a 50/30 μm DVB/CAR/PDMS solid phase microextraction head (2 cm, Supelco, Inc., Bellefonte, PA, USA)., USA) for 30 min.

The GC-MS analysis was performed on a DB-WAX (30 m × 0.32 mm × 0.25 μm) column with high-purity helium (99.999%) as the carrier gas at a 1.0 mL/min-1 flow rate in spitless mode. The inlet temperature was maintained at 270 °C, and the heating program was started at 40 °C for 5 min, then increased to 110 °C at 4 °C/min for 4 min, and then increased to 300 °C at 20 °C/min for 8 min. The ion source temperature was 230 °C, the electron collisional ionization was adjusted to 70 eV, and the scanning mass was 20–500 m/z.

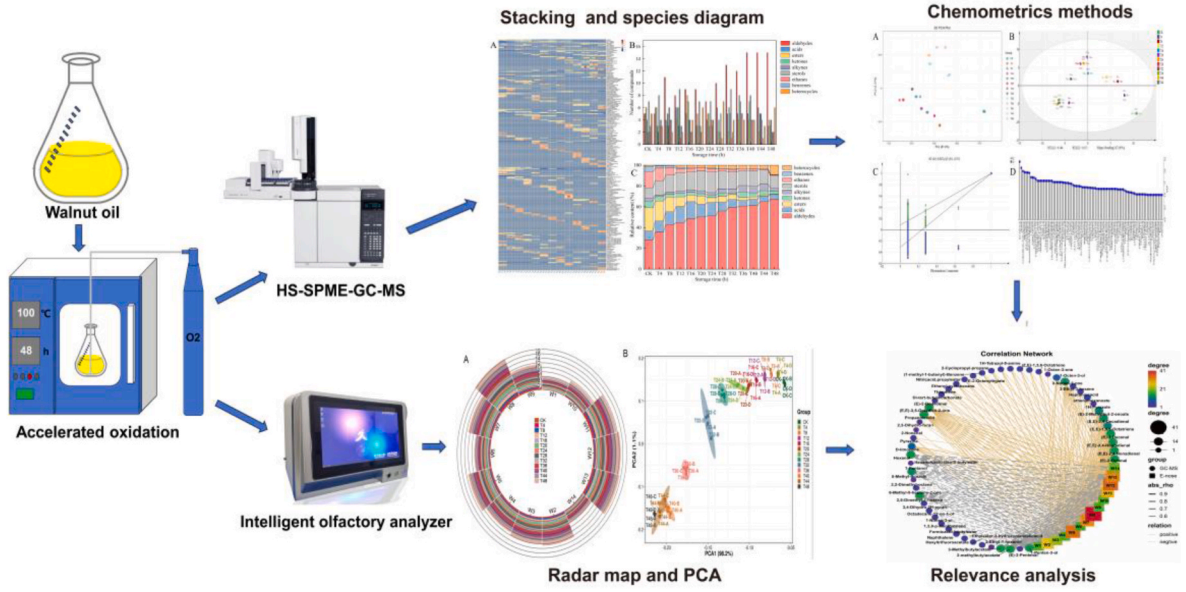

Fig. 1. Processing flow chart for characterization of VOCS in walnut oil with different oxidation degrees and multivariate analysis.

Table 1
Electronic nose array sensors.

Sensor number	Model/ Name	Manufacturer	Detection Object	Detection Concentration Range
W1	MQ136	Figaro	Air pollution (trimethylamine, methyl mercaptan, etc.), garbage, and foul gases in the sewer	1–10 ppm
W2	MP702	Winson	Ammonia (Gas)	0–100 ppm
W3	TGS2615	Figaro	Cigarette smoke, soot, hydrogen, alcohol, methane, carbon monoxide	1–30 ppm
W4	TGS2603	Figaro	Air pollution (trimethylamine, methyl mercaptan, etc.), garbage, foul sewer gas	1–10 ppm
W5	MQ131	Winson	O_3	10–1000 ppm
W6	MP4	Winson	Methane, natural gas, biogas, etc.	300–10,000 ppm
W7	TGS2600	Winson	CO	10–500 ppm
W8	TGS2618	Winson	Methane, natural gas, biogas, etc.	300–10,000 ppm
W9	TGS2612	Winson	Liquefied petroleum gas, natural gas, etc.	300–10,000 ppm
W10	TGS2620	Figaro	Organic solvents such as ethanol	50–5000 ppm
W11	TGS2602	Winson	Ammonia, sulfide, benzene vapors	10–1000 ppm
W12	WSP2110	Figaro	Fluorine, plastic encapsulated, and metal encapsulated	10–300 ppm
W13	MQ137	Winson	nitrogen dioxide	0.1–10 ppm
W14	MP901	Winson	Alcohol, smoke, formaldehyde, toluene, benzene, acetone, lighter gas, paint, etc.	1–50 ppm

2.5. Qualitative analyses

Qualitative analyses were based on previously reported methods (Yu et al., 2024). The identification of volatile compounds in walnut oil was based on gas chromatography retention index (RI) and mass spectrometry (MS) compared with the libraries of Cowan National Institute of Standards and Technology Library (NIST 14, USA) and Wiley Online Library (Wiley 11, USA). Volatile compounds were identified when the match was more significant than 800, i.e., more than 80% match with the database's mass spectral fragments of the candidate components.

2.6. Data processing

Microsoft Excel processed E-nose and HS-SPME-GC/MS data collation. Origin 2021b was used for radar plots, correlation network diagrams, and principal component analysis (PCA), orthogonal partial least squares discriminant analysis (OPLS-DA) was plotted using SIMCA 14.1, and heat maps of HS-SPME-GC-MS data were plotted using TBtools-ll (Toolbox for Biologists) v2.080 for Heat Map Plotting of HS-SPME-GC-MS Data.

3. Results and discussion

3.1. Analysis of volatile components of walnut oil based on olfactory analyzer

An olfactory analyzer (E-nose) is a high-tech product that mimics the olfactory organs of animals or humans and can respond quickly and detect gas components with good repeatability (Pei et al., 2023) . Using E-nose to detect walnut oil volatiles with different oxidation levels, the response values varied among the 14 sensors. As shown in Fig. 2A, the response values of the odor sensors showed a gradually increasing trend, and they all reached the maximum value at T48. There were differences in the response values of W2, W5, W7, W8, W11, W12, and W13. The others were lower, indicating that 7 sensors, such as W2 and W5, played a significant role in the evaluation of the aroma of walnut oil during storage; the response values of the walnut oil in the W2 and W8 sensors had the minimum and maximum response values, respectively, indicating that the aroma of walnut oil under these two sensors varied greatly. These results indicate that the olfactory analyzer can effectively distinguish the volatile compounds of walnut oil, and the magnitude of the response value can judge the oxidation degree of walnut oil. However, the olfactory analyzer has some limitations in identifying the odor and source of volatile substances in different samples. The specific differences in walnut oil volatiles are caused by characteristic volatiles, for

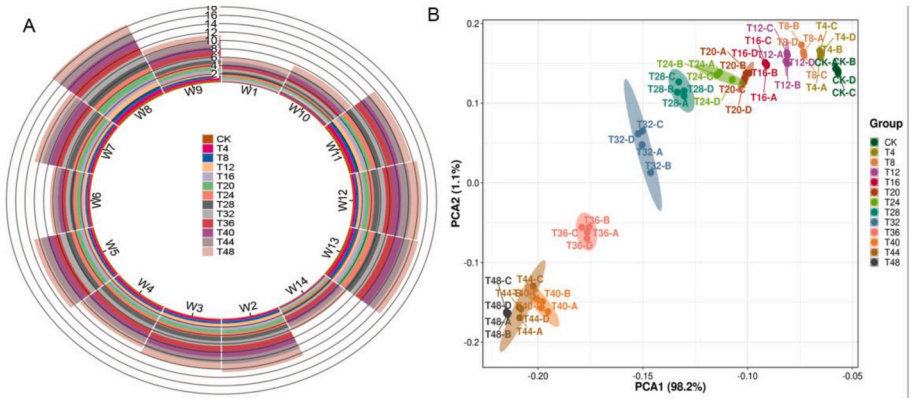

Fig. 2. Olfactory analyzer variation of walnut oil with different oxidation levels. (A) Variation of Olfactory analyzer response values over time. (B) Principal component analysis of olfactory analyzer response scores.

which the phenomenon needs further exploration.

In order to further clarify the variability of volatile compounds in walnut oil with different oxidation levels, the response values of the E-nose sensors were analyzed by PCA, a multivariate statistical method designed to compress multiple variables into significant variables projected onto the axes, reducing the complexity and computational cost of the data (Dong et al., 2019; Song et al., 2023). As shown in Fig. 2B, the results showed that the contribution rates of PC1 and PC2 were 93.7% and 3.4%, respectively. Their total contribution rate was 97.1%, which was higher than 90%, suggesting that PC1 and PC2 could respond to the main informative characteristics of volatile compounds of walnut oil under different oxidation levels. Compared with the control group (CK), all the other groups showed significant differences with the increase of oxidation degree, and the volatiles between the samples increased gradually; however, it is worth noting that there were overlapping phenomena between T20 and T24 and between T40-T48 samples, which indicated that there was not much difference in volatile compounds between samples at this stage or the trend of change was not noticeable. In contrast, the distance between the T44-T48 inter-sample group and the CK group was the furthest, indicating a more significant variation in the volatile compounds of walnut oil at these two storage times.

3.2. Analysis of volatile compounds in walnut oil

3.2.1. Analysis of relative content and species variation of walnut oil

The volatile compounds of walnut oil with different degrees of oxidation were analyzed comprehensively by HS-SPME-GC/MS, and a total of 219 compounds were identified, among which 29 aldehydes, 22 esters, 15 acids, 22 ketones, 33 terpenes, 34 alcohols, 11 benzenes, 28 alkanes, and 25 other heterocyclic substances were identified. As shown in Fig. 3A, the various types of volatile compounds in the oxidation process showed apparent differences in the changes. Specifically, aldehydes showed the most remarkable change, with the relative content increasing from 24.58% to 64.9% and the number rising from 5 to 15. Aldehydes in walnut oil originate from the conversion of its high content of unsaturated fatty acids to hydroperoxides by lipoxygenase (LOX) and their degradation to aldehydes under the catalyzing conditions of hydro peroxidase lyase (HPL) (Kalogiouri et al., 2021; Xu et al., 2023b). In

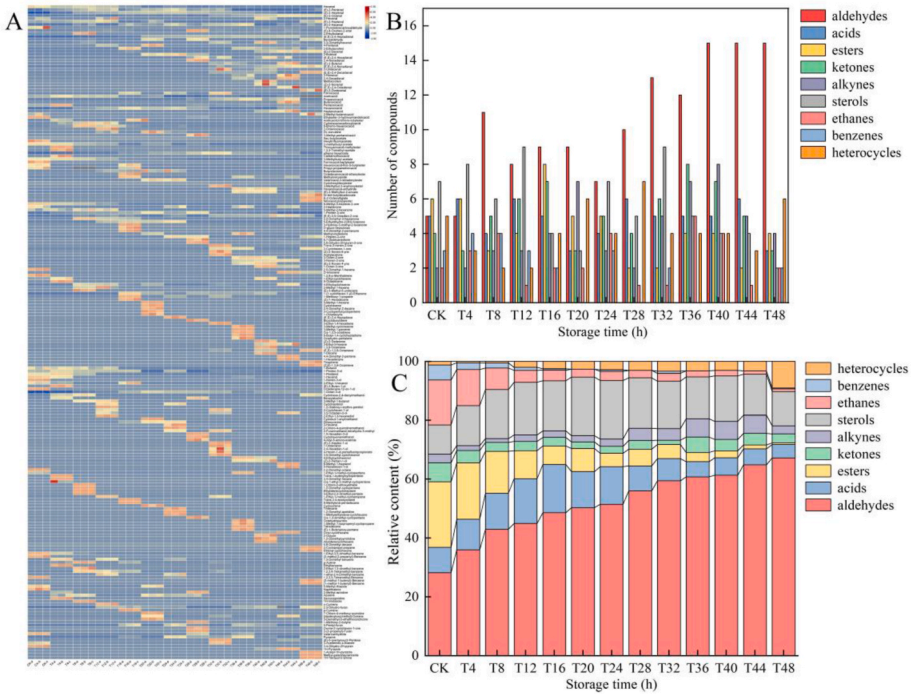

Fig. 3. Relative content and species analysis of volatile compounds in walnut oil (A. Clustered heat map; B. Species map; C. Stacking).

addition, aldehydes have a low odor threshold, and their relative content showed a gradually increasing trend during the oxidation of walnut oil (Fig. 3C), suggesting that they contribute more to the overall aroma of walnut oil. Ester compounds mainly conferred fruity and floral aroma to walnut oil, which mainly originated from the esterification reaction occurring between alcohols and acids (Xu et al., 2023a), and their relative contents gradually decreased from 22.57% in the early stage (CK) to 0.55% in the late stage (T48), while there was no significant difference in the species (Fig. 3B). The ester compounds throughout the oxidation process gradually decreased with storage time under high-temperature conditions, which is similar to the results reported in the literature (Song et al., 2023; Zhu et al., 2022). The source of alcohol was mainly the decomposition of fatty acids (Ma et al., 2024), and the relative content reached a maximum of 20.46% at the oxidation time (T24), which was 2.04 times higher than that of the initial (CK). Alcohols are an essential product of walnut oil's oxidation process, giving it a vegetal and aromatic odor (Huo et al., 2024; Song et al., 2021). A total of 63 hydrocarbons (terpenes, alkanes) were detected in the oxidation process of walnut oil. Unsaturated fatty acids with low odor thresholds of d-limonene and 1,3,8-p-menthatriene were detected in the pre-production phase (CK), conferring herbal and citrus aromas to walnut oil. In contrast, alkanes, which are mainly formed by the decomposition of alkoxy radicals in fatty acids, can provide walnut oil with an inherent fresh aroma but have little effect on the aroma profile of walnut oil due to their high odor threshold (Maggi et al., 2010). A total of 15 acids were detected during the oxidation of walnut oil. Among them, most of the saturated acids were detected, such as formic acid, acetic acid, pentanoic acid, hexanoic acid, and heptanoic acid, which may be produced by the oxidation of the corresponding aldehydes and present a sour, cheesy, sweet, and pungent flavor, which are essential volatile compounds affecting the overall walnut oil They are important volatile compounds that affect the overall aroma of walnut oil (Xu et al., 2023a). Ketones are due to the degradation of amino acids and the production of Meladic reaction under the conditions of high temperature and high oxygen content of walnut oil. Most of the ketones present butter and fruity flavors, and their relative content is relatively low during the oxidation process of walnut oil. However, it has been shown that their presence contributes to the generation of pyrazines in walnut oil and will gradually decrease along with the prolongation of the oxidation process of walnut oil (Pei et al., 2023). In addition to this, some complex heterocyclic substances such as furan, thiazole, pyrazine, etc., which are produced as a result of free radical cyclization of linoleic acid with oxygen (Ma et al., 2024), are also produced, which confer odors such as roasted nut, coffee and caramel to walnut oil and are essential aroma compounds in walnut oil (Zhang et al., 2021).

In summary, walnut oil is interspersed with numerous complex oxidation, degradation, and polymerization reactions during the oxidation process, accompanied by the dynamic changes of numerous aldehydes, acids, lipids, and heterocyclic compounds. Therefore, the monitoring and identification of these volatile compounds are crucial.

3.2.2. *Multivariate statistical analysis of volatile compounds in walnut oil*

To further assess the changes in the characteristics of walnut oil volatiles, 219 compounds detected by HS-SPME-GC/MS were subjected to multivariate statistical analysis (MSA). Overall, the trend of volatile compounds in walnut oil with storage time was apparent (Fig. 4A), and PCA provided a visual distinction between different groups (CK~48 h) of walnut oil samples. As shown in Fig. 4A, the distance between the three parallel samples is short, indicating the data's reproducibility. Fig. 4A also shows a clear trend of separation for each group of samples, indicating significant differences in volatile compounds between samples. In short, the PCA indicates significant separation between the 13 groups of samples and good reproducibility within each group. The PCA plot shows that the first two principal components explain 89.67% of the total variance, with PC1 and PC2 accounting for 87.40% and 2.27%, respectively, representing the main informative characteristics of the samples. In addition, to better construct the characteristic differences between these groups, we employed OPLS-DA analysis. This model can effectively differentiate the differences between samples with different oxidation levels in walnut oil and accurately reflect the degree of contribution to the changes in volatile compounds of the samples (Kong et al., 2024). Fig. 4B shows that the score plots of OPLS-DA showed significant results, with significant group separation detected and each group showing tighter clustering, suggesting that the OPLS-DA method is reproducible. In addition, the model was cross-validated with 200 iterations, and the model prediction index Q^2 was 0.994, with Q^2 above 0.5 indicating good model prediction and above 0.9 indicating excellent prediction. This indicates that the model is not overfitted (Wang et al., 2024) and can be used for the discrimination of volatile compounds in walnut oil, which is similar to the results of the PCA analysis of the

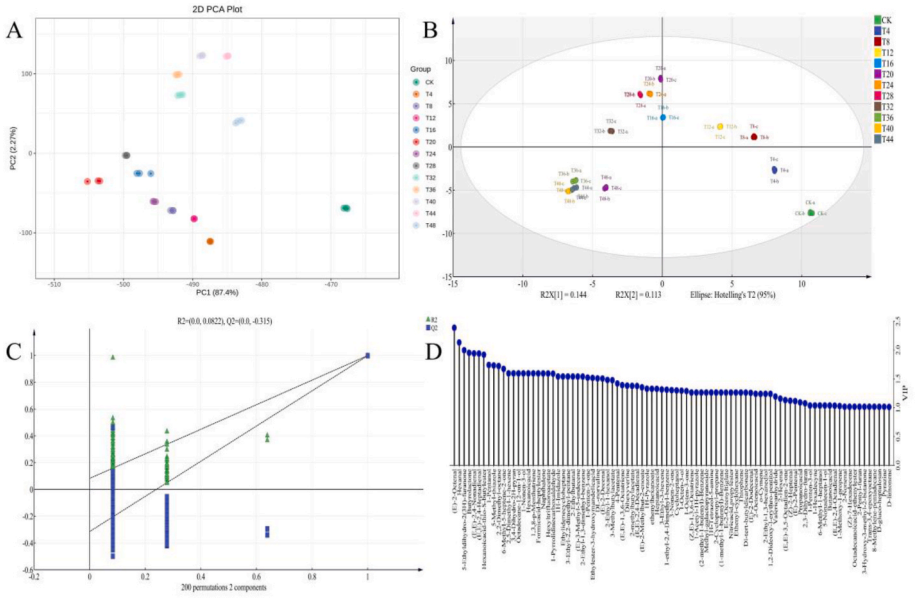

Fig. 4. Multivariate statistical analysis of walnut oil (A. Principal component analysis; B. OPLS-DA score plots; C. 200 permutation test plots; D. Volatile compounds with VIP >1).

electronic nose.

The VIP value filters out the variables contributing most to model classification (Huo et al., 2024; Tapp and Kemsley, 2009). Specifically, VIP values are used to assess the importance of a variable in a model, and variables with values greater than 1 are considered to be those that significantly influence the model classification results and are, therefore, often used as important metrics to examine for screening differential metabolites (Wang et al., 2020). Using variable importance projections allowed the characterization of volatile-specific markers between different oxidation levels in walnut oil, as shown in Fig. 4D, and a total of 89 volatile compounds with VIP values greater than 1 were screened.

3.2.3. *Cluster analysis of volatile organic compounds in walnut oil with different degrees of oxidation*

To further visualize the relationships between the characteristic volatiles filtered by the VIP values in 3.2.2, a clustered heat map based on Euclidean distances was used, with hierarchical cluster analysis based on the normalized data. Thermal clustering provided an average configuration profile for each volatile compound, with color intensities normalized from maximum (red) to minimum (blue), corresponding to the relative content of volatile compounds from high to low. The thermal clustering results graphically demonstrate the distribution of each volatile substance in the samples, and the darkest-colored substances can be considered characteristic volatiles of different oxidation levels in walnut oil. As shown in Fig. 5, complex biochemical reactions such as the oxidative decomposition of unsaturated fatty acids and the degradation of amino acids were generated during the oxidation of walnut oil, which led to dynamic changes in the oxidation process of volatile compounds.

Specifically, the volatiles whose relative contents changed significantly with increasing oxidation in walnut oil were (E, E)-2,4-decadienal (6.10%–23.04%), (E, E)-2,4-heptadienal (2.23%–25.60%), (E)-2-octenal (0.95%–11.71%), hexanoic acid (1.63%–4.30%), 1-octen-3-ol (2.53%–19.01%), (Z)-2-heptenal (5.95%–25.01%), 2,3-dihydrofuran (1.08%–3.20%), 2-pentyl furan (0.13%–0.54%), pyrazine (0.33%–1.32%), hexanal (24.52%–1.33%), 3-methyl butyl acetate (12.44%–1.29%), 2-methyl butyl acetate (7.74%–1.56%) and ethenyl hexanoate (4.39%–0.41%). Among them, (E, E)-2,4-decadienal appeared in sample T32 and reached the highest relative amount in sample T48. (E, E)-2,4-heptadienal appeared in sample T8, and the relative content tended to increase with oxidation time, reaching a maximum in sample T44. (E)-2-octenal appeared in sample T4 and reached the highest relative content in sample T44. (Z)-2-heptenal appeared in the T4 samples and reached the highest value at an oxidation time of 24 h. The highest values were found in the T4 samples. In conclusion, the relative content of these aldehydes gradually increased with oxidation time. It occupied an important position in the walnut oil oxidation process, and its formation was mainly attributed to the Meladic reaction and Strecker oxidative degradation (Hao et al., 2020). Lower concentrations of aldehydes impart green, grassy, floral, fruity, and fatty aromas to walnut oil, but high concentrations of aldehydes are one of the main reasons for the unpleasant halcyon odor of walnut oil (Cui et al., 2024; Xi et al., 2024a) It is used as an aging marker for the oxidative deterioration of walnut oil (Xi et al., 2024b). In addition, Hexanal was the most abundant aldehyde in the CK samples, gradually decreasing with oxidation time, reaching a minimum in the T44 sample. It has a strong nutty flavor and is mainly degraded by lipid oxidation and amino acids (Wang et al., 2022). It has been shown in existing reports (Xu et al., 2018) that the relative content of Hexanal increases with increasing oxidation levels, which is in contrast to the results of our experiments. This situation may have occurred because our experiments were carried out at high temperatures and high oxygen content, lipid-derived radicals accelerated the Maillard reaction, or aldehydes during lipid degradation promoted an increase in furans and pyrazines, which depleted Hexanal in walnut oi (Ma et al., 2024; Zhang et al., 2021). Hexanoic acid was present throughout the oxidative process, presenting a cheesy and fishy odor for walnut oil. Specifically, it increased from 1.74% to 12.33% during the oxidation

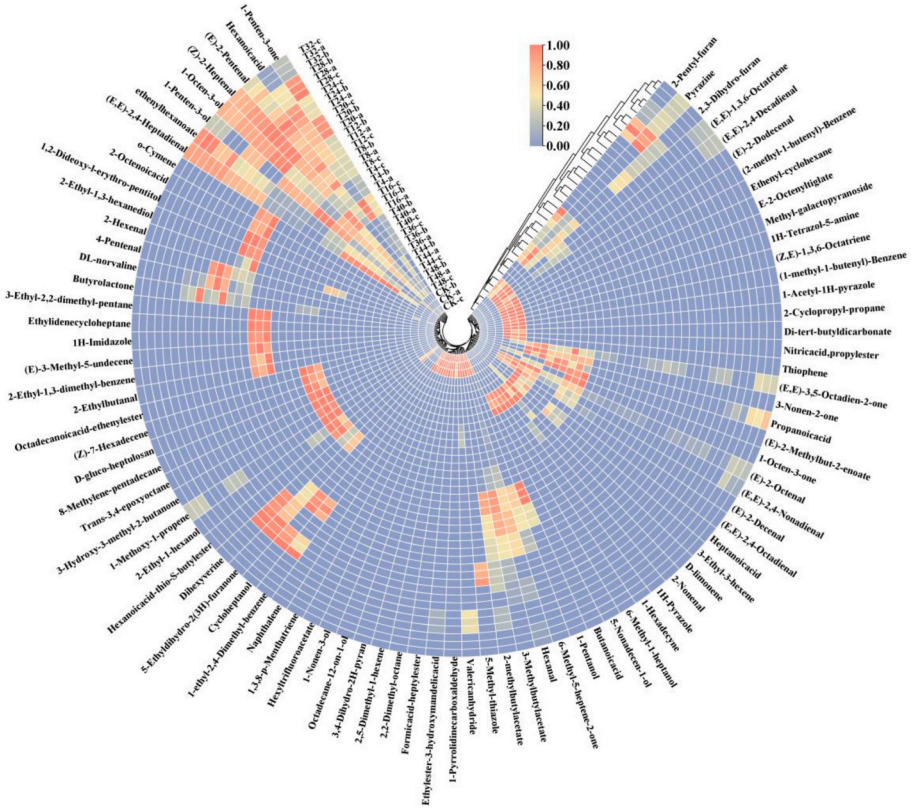

Fig. 5. Clustering heat map of characteristic volatile compounds of walnut oil.

process from the 0th to the 16th h and gradually decreased until 4.30% at the late oxidation stage. The increase of saturated acids in walnut oil was attributed to the further oxidative decomposition of secondary oxides such as aldehydes (Xi et al., 2024a), and the decreasing tendency after 16 h could be due to the esterification reaction with the corresponding aldehydes in walnut oil (Ghorbani Gorji et al., 2019). 1-octen-3-ol, which exhibits a mushroom aroma, showed a gradual increase during the oxidation of walnut oil, with the relative content increasing from 2.53% at T4 to 10.25% at T48. It has been shown that it is mainly produced by the oxidation and degradation of arachidonic acid (AA) in polyunsaturated fatty acids (PUFA), which is considered one of the key aroma compounds in walnut oil (Ghorbani Gorji et al., 2019; Grilo and Wang, 2021). Meanwhile, the concentration showed a gradually increasing trend during oxidation and was the most abundant alcohol compound in aged walnut oil, which is like our results (Xi et al., 2024a). The aromatic compound 2,3-dihydro-furan, belonging to the heterocyclic compound class, exhibited a gradual increase in its relative content with the progression of oxidation time, reaching a maximum of 48 h (3.2%). Additionally, 2-pentyl furan and pyrazine, significant aroma compounds in walnut oil, were identified. It is postulated in existing reports that it originates from the oxidative decomposition of linoleic acid, which provides caramel, butterscotch, and baking flavors to the overall aroma of walnut oil (Xu et al., 2023a). 3-methyl butyl acetate, 2-methyl butyl acetate, the relative content of ethenyl hexanoate, 2-methyl butyl acetate, and 3-methyl butyl acetate undergo a significant change throughout the oxidation process of walnut oil. These esters are primarily produced through the esterification reaction between alcohols and organic acids and are predominantly found in fresh walnut oil. However, with the oxidation of lipids, their concentration gradually decreases. Most of these esters exhibit a fresh floral and fruity aroma. Furthermore, it has been demonstrated that these volatile compounds effectively attenuate fatty acids' pungency and the bitterness of amino acids (Chen et al., 2024; Li et al., 2023a). Therefore, these compounds play a significant role in distinguishing the oxidation process of walnut oil, which is the primary source of oxidative deterioration of walnut oil and can be used as potential characteristic markers of walnut oil oxidative rancidity.

3.3. Correlation analysis of response values of walnut oil olfactory analyzer with GC-MS

As shown in Fig. 6, a correlation network diagram was constructed with the characteristic volatile compounds of walnut oil with VIP >1 in the HS-SPME-GC-MS results (89) and the sensor response values of the olfactory analyzer (14). The square blocks in the graph are the sensors of each E-nose; the size and color shade of the squares represent the importance of the sensors, the size and color shade of the dots indicate the importance of the volatile compounds, and the thickness of the curves indicate the degree of correlation. 1246 correlations were established between the 89 volatile compounds and the 14 olfactory analyzer sensors, and the correlations were based on the Pearson correlation coefficients $p < 0.05$ and $|r| > 0.8$ to create a correlation network. The network plot showed 358 correlations, including 228 positive ones (gold curve) and 130 negative ones (silver curve). Specifically, 14 sensors had strong correlations with 53 volatile compounds. Olfactory analyzer sensors W8, W13, W11, W12, W5, W7, and W2 were the central nodes associated with most volatile compounds. Pyrazine, (E, E)-3,5-octadien-2-one, propanoic acid, (E)-2-octenal, (E, E)-2,4-nonadienal, (E, E)-2,4-heptadienal, (E)-2-decenal, (E, E)-1,3,6-octatriene, (E, E)-2,4-decadienal, (E)-2-methylbut-2-enoate, and 1H-pyrazole showed significant positive correlation. In contrast, hexanal, 1-pentanol, 6-pethyl-5-heptene-2-one, 3-pethylbutylacetate, and 2-methylbutylacetate showed a significant negative correlation, which is consistent with the change in the characterized volatiles in the clustered heat map of 3.2.3. This change suggests that the olfactory analyzer sensor can distinguish the oxidation process of walnut oil based on the specific response values of the main volatiles in walnut oil. In summary, the correlation analysis of the HS-SPME-GC/MS and E-nose results provided visualization of the volatile components, which provided an effective means of indicating the characteristic compounds of walnut oil with different degrees of oxidation, and the network modelling analysis further confirmed the correspondence between the volatile compounds and the E-nose, which helps to provide a certain This helps to provide a certain basis for the development of rapid detection of walnut oil.

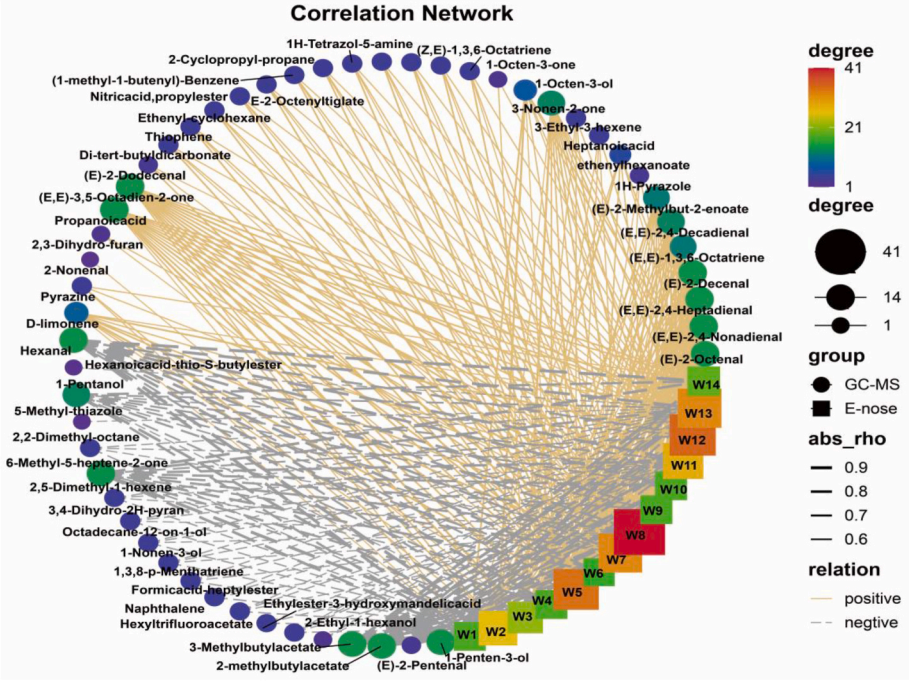

Fig. 6. Correlation network diagram of E-nose's sensor with characteristic volatile compounds in GC-MS.

4. Conclusion

In this study, the volatile compounds of walnut oil during the oxidation process were comprehensively analyzed using HS-SPME-GC-MS and olfactory analyzer techniques. The results of the olfactory analyzer showed that the volatile organic compounds in the walnut oil samples presented a good differentiation as the degree of oxidation advanced, and the content of volatile compounds differed significantly from each other. HS-SPME-GC-MS identified 219 volatile compounds, and 89 key volatile compounds were screened (VIP > 1). The thermal clustering results visualized the distribution of each volatile substance in the samples. (E, E)-2,4-decadienal (6.10%–23.04%), (E, E)-2,4-heptadienal (2.23%–25.60%), (E)-2-octenal (0.95%–11.71%), hexanoic acid (1.63%–4.30%), 1-octen-3-ol (2.53%–19.01%), (Z)-2-heptenal (5.95%–25.01%), 2,3-dihydrofuran (1.08%–3.20%), 2-pentyl furan (0.13%–0.54%), pyrazine (0.33%–1.32%), hexanal (24.52%–1.33%), 3-methyl butyl acetate (12.44%–1.29%), 2-methyl butyl acetate (7.74%–1.56%) and ethenyl hexanoate (4.39%–0.41%) can be defined as characteristic markers in the walnut oil during oxidation processing, elucidating its dynamic changes. The correlation network plot strongly correlated the E-nose sensors and the characterized compounds. Although 219 volatile compounds were identified in this study, more compounds may be produced during the oxidation of walnut oil. More advanced analytical techniques, such as ultra-performance liquid chromatography-mass spectrometry (UPLC-MS) or nuclear magnetic resonance (NMR), will be considered in the future study to identify and analyze the volatile compounds in walnut oil, and to obtain a more comprehensive volatile composition profile. Meanwhile, future studies can explore the antioxidant mechanisms and activities of these volatile compounds and how they can be used to improve the antioxidant properties and extend the shelf life of walnut oil. Finally, based on the strong correlation between the E-nose sensors and the characteristic compounds, a device dedicated to the rapid detection of the oxidation level of walnut oil could be developed in the future for real-time monitoring of oxidation level and quality changes of walnut oil, which could provide a convenient detection method for quality control and preservation of walnut oil.

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CRediT authorship contribution statement

Lina Sun: Investigation, Writing – original draft, Formal analysis, Data curation, Funding acquisition. **Guowang Wang:** Investigation, Software, Formal analysis, Writing – review & editing. **Lijian Xiong:** Methodology, Modelling. **Zhongqiang Yang:** Methodology, Funding acquisition. **Yan Ma:** Funding acquisition. **Yanlong Qi:** Methodology, Validation. **Yongyu Li:** Methodology, 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.

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