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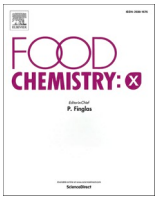

Investigation of aroma characteristics of seven Chinese commercial sunflower seed oils using a combination of descriptive Analysis, GC-quadrupole-MS, and GC-Orbitrap-MS

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ABSTRACT

The aroma characteristics of seven commercial Chinese sunflower seed oils were investigated in this study using descriptive analysis, headspace solid-phase microextraction coupled with GC-quadrupole-MS (LRMS, low-resolution mass spectrometry), and GC-Orbitrap-MS (HRMS, high-resolution mass spectrometry). GC-Orbitrap-MS quantified 96 compounds, including 18 alcohols, 12 esters, 7 ketones, 20 terpenoids, 11 pyrazines, 6 aldehydes, 6 furans, 6 benzene ring-containing compounds, 3 sulfides, 2 alkanes, and 5 nitrogen-containing compounds. Moreover, 22 compounds including 5 acids, 1 amide, and 16 aldehydes were quantified using GC-Quadrupole-MS. To our knowledge, 23 volatile compounds were reported for the first time in sunflower seed oil. All the seven samples were found to have a 'roasted sunflower seeds' note, 'sunflower seeds aroma' note and 'burnt aroma' note and only five of them had 'fried instant noodles' note, three had 'sweet' note and two had 'puffed food' note. Partial least squares regression was used to screen the candidate key volatiles that caused the aroma differences among these seven samples. It was observed that 'roasted sunflower seeds' note was positively correlated with 1-octen-3-ol, *n*-heptadehyde and dimethyl sulfone, whereas the 'fried instant noodles' and 'puffed food' demonstrated a positive correlation with pentanal, 3-methylbutanal, hexanal, (E)-2-hexenal and 2-pentylfuran. Our findings provide information to the producers and developers for quality control and improvement of sunflower seed oil.

Introduction

Sunflower (*Helianthus annulus* L.) is one of the primary oil crops worldwide and is cultivated in Europe, the Middle East, Asia, America and Africa (Pilorgé, 2020). Sunflower seed oil is typically extracted from raw or roasted sunflower seeds. Sunflower seed oil is renowned among consumers worldwide due to its rich nutrient content and distinct flavor. In 2020, the global annual production of sunflower seed oil was approximately 19,158 million tons, rising to 21,796 million tons in 2021 (USDA, 2021). The aroma of vegetable oil is an essential characteristic of its quality and has a considerable impact on consumers' purchasing inclinations. More and more attention has been paid to the research on the aroma of sunflower seed oil.

Both solvent-assisted flavor evaporation (SAFE) and headspace solid-phase microextraction (HS-SPME) pretreatments had been used to concentrate volatile compounds in sunflower seed oil (Guillen, Cabo, Ibargoitia, & Ruiz, 2005; Yin, Shi, Li, Ma, Wang, & Wang, 2022). HS-SPME is one of the most widely reported techniques for the extraction of volatile compounds from sunflower seed oil due to its remarkable advantages such as ease, speed, small sample size, solvent-free system,

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and automation of the entire extraction process (Guillen et al., 2005; Marasca, Greetham, Herring, & Fisk, 2016; Nieva-Echevarria, Goicoechea, & Guillen, 2019; Petersen, Kleeberg, Jahreis, & Fritsche, 2012). Gas chromatography coupled with single quadrupole mass spectrometry (GC–MS) is commonly used to identify volatile compounds in sunflower seed oil (Aydeniz-Guneser & Yilmaz, 2022; Fu, Shen, Peng, Zhou, & Hou, 2019; Hu et al., 2014; Petersen et al., 2012; Valdés García, Beltrán Sanahuja, Karabagias, Badeka, Kontominas, & Garrigós, 2021; Yin, Shi, Li, Wang, et al., 2022). Notably, a previous study reported that two-dimensional comprehensive chromatography-time-of-flight mass spectrometry could identify additional volatile compounds in sunflower seed oil (Hu et al., 2014), indicating that advanced analytical instruments could contribute to a better understanding of the sunflower seed oil aroma.

Recently, GC-Orbitrap-MS has emerged as a compelling substitute for single quadrupole MS because accurate mass measurements can reveal substantially lower background signals, thereby enhancing the signal-to-noise ratio (S/N) of the target analyte and minimizing background ion interference. In 2005, Alexander Makarov developed the first Orbitrap-based mass spectrometer (Eliuk & Makarov, 2015) and it was applied to GC/MS for the first time in 2010 (Peterson, McAlister, Quarmby, Griep-Raming, & Coon, 2010). GC-Orbitrap-MS could provide high quality resolution (upto 120,000 FWHM) (m/z 200) and high-quality accuracy (<3 ppm) (Belarbi, Vivier, Zaghouani, Sloovere, Agasse-Peulon, & Cardinael, 2021). Several GC-Orbitrap-MS methods have been recently developed for the quantitative analysis of pesticide residues in kinds of foods (Belarbi et al., 2021; Mol, Tienstra, & Zomer, 2016), as well as persistent organic pollutants in environmental samples. GC-Orbitrap-MS has been demonstrated as a powerful technique for the qualitative assessment of trace aroma compounds (Dominguez, Arrebola, Martinez Vidal, & Garrido Frenich, 2020). We recently used GC-Orbitrap-MS to analyze aroma compounds in fruit wines (Lin et al., 2022; R. Liu, Liu, Zhu, Kortesniemi, Zhu, & Li, 2022; Y. Liu et al., 2022). Similarly, high-resolution and sensitive instruments were found that could identify more key volatiles in foods such as green tea, fruits and wines (Lim, Gorji, Daygon, & Fitzgerald, 2020; Qian et al., 2020; Yanqin Yang et al., 2020). The application of GC-Orbitrap-MS could contribute to the identification of additional aromatic compounds in sunflower seed oils.

Quantitative descriptive analysis (QDA) is commonly used in food aroma evaluation (Da Costa, Dal Bosco, Ramos, Machado, Garavaglia, & Villasclaras, 2020; Xu, Wu, Huang, Zhang, Jin, & Wang, 2023), and it had already been used in sunflower seed oil. Yin used the QDA method to evaluate one roasted and one cold-pressed sunflower seed oils and their corresponding reconstituted samples, respectively (Yin, Shi, Li, Ma, et al., 2022). This study found that the two samples were very different in aroma sensory and were determined by their volatiles composition.

Roasting is a standard process to enhance and modify the flavor and nutritional value of vegetable oils including sesame, cactus seed, peanut, walnut and other raw materials (Bi et al., 2022; Nounah, Chbani, Matthäus, Charrouf, Hajib, & Willenberg, 2020; K. Yang, Chao, Wu, Ye, & Chen, 2021; Yin, Ma, Li, Wang, Liu, & Shi, 2021; W. Zhang, Cao, & Liu, 2020; Y. Zhang, Li, Lu, Sun, & Wang, 2021). As a processing technique, roasting also has the potential to increase the oil yield and antioxidant activity (Xu, Zhu, Liu, Karrar, Ouyang, & Li, 2022). In China, the demand for heat-treated sunflower seed oil has increased substantially. However, researches on roasted sunflower seed oil were mainly focused on its nutritional properties, safety and physicochemical indexes but not on flavor (Ji, Liu, & Ma, 2022; Kiczorowska et al., 2019; Özcan & Köse, 2022; C.-X. Zhang, Xi, Zhao, Ma, & Wang, 2020). Therefore, it is necessary to investigate the sensory attributes and volatile compounds composition of roasted sunflower seed oil available in the Chinese market.

Partial least squares regression (PLSR) is a widely used statistical analysis method that connects two matrices, X and Y, to create linear multivariate models (Gu et al., 2018). It has been reported to be an effective statistical prediction tool for chemometric datasets with several variables and a relatively small sample size (Wu et al., 2022). Currently, PLSR is mostly used to identify key aroma compounds present in vegetable oils, including onion oil (Tian, Zhan, Tian, Wang, Lu, & Zhao, 2020), A. *tenuissimum* flower oil (C. Zhang, Wang, Ding, Su, & Zhao, 2022), and even cold pressed sunflower seed oils from Italy (Bendini, Barbieri, Moradi, Palagano, Valli, & Toschi, 2014). We believe that, till date, there has been no research on the application of the PLSR model to analyze the aroma of commercial sunflower seed oils that are popular in China.

To above all, seven commercially available sunflower seed oils from China market were analyzed by using both GC-Quadrupole-MS (LRMS, low-resolution mass spectrometry) and GC-Orbitrap-MS (HRMS, high-resolution mass spectrometry) for volatile identification and the QDA method for sensory evolution. We aimed to investigate the aroma characteristics and the potential key aroma compounds of seven commercial sunflower seed oils from Chinese market. Our findings can be useful in quality control and the improvement of sunflower seed oil. We believe that this is the first study to utilize GC-Orbitrap-MS for the identification of volatiles in sunflower seed oils.

Materials and methods

Samples

The samples selected in this study were seven commercially available sunflower seed oils within the shelf life. Most were produced by different manufacturers, and four samples were from the same manufacturer, albeit with differences in the raw materials and processes. The samples were designated HG, JZ, JN, LH, JXP, JXZ and HK. All the products implement GBT10464-2017 standard.

Chemical

All standards were of chromatographic grade, dissolved in ethanol and stored at -20 °C. Except for standard products, the reagents without special instructions were of analytical grade purity, and their details are shown in Table S1. An *n*-alkanes solution (C6-C24) was obtained from Supelco, Bellefonte, PA, USA.

HS-SPME-GC-Quadrupole-MS detection of volatile compounds in sunflower seed oil

Volatile compounds in sunflower seed oil samples were extracted by HS-SPME. Five grams of sunflower seed oil sample was accurately weighed into a 20 mL vial, tightly capped with a PTFE-silicone septum and 10 μL of 4-methyl-2-pentanol (solution dissolved in ethanol, 0.2 g/L) was added.

The chromatographic procedure for HS-SPME-GC-Quadrupole-MS was optimized based on existing studies (Jia et al., 2021; Ni, Wang, Zhan, Tian, & Li, 2021; Ni, Yan, Tian, Zhan, & Zhang, 2022; Zhou et al., 2019). The sample was equilibrated at 60 °C for 30 min. A solid-phase microextraction fiber (50/30 μm DVB/CAR/PDMS; Supelco, Bellefonte, PA, USA, with preheat treatment according to the manufacturer's recommendations before use) was exposed for 30 min (60 °C) in the vial headspace and then desorbed into the GC injection port at 250 °C for 8 min.

The volatile compounds in seven sunflower seed oil samples were analyzed using an Agilent 7890 A GC System (Agilent Technologies, Santa Clara, CA, USA) on a HP-INNOWAX capillary column (60 m × 0.25 μm, 0.25 μm, J & W Scientific, Santa Clara, CA). The temperature increase procedure for chromatography was as follows: 40 °C for 5 min, 5 °C per min up to 240 °C, and held for 5 min. The flow rate of carrier gas (nitrogen with 99.99% purity) was set at 1 mL/min. All mass spectra were acquired in electron ionization mode at 70 eV using full scan with a scan m/z range of 25 to 300. Under the same chromatographic and mass

spectrometric conditions, a C7–C30 *n*-alkane series (500 μg/L, Supelco, Bellefonte, PA, USA) were analyzed to calculate the retention indices (RIs).

The compounds were identified by combining the calculated RI value, the mass spectrum information in NIST13 (S. Liu, Laaksonen, Kortesniemi, Kalpio, & Yang, 2018; Wang et al., 2018) and the retention index. The relative abundances of the compounds in the samples were expressed as peak area ratios (J. Zhang, Li, Gao, Wang, Gao, & Jiang, 2010). The volatile compounds detected were quantified by calculating the peak area ratio using the internal standard method, with 4-methyl-2-pentanol as the internal standard. Three repeated injections were performed for each sample.

HS-SPME-GC-Orbitrap-MS detection of volatile compounds in sunflower seed oil

Sunflower seed oil samples were pretreated by HS-SPME, as described in Section 2.3. A TriPlus RSH autosampler (Thermo Fisher Scientific, Bremen, Germany) was used for automated headspace solid-phase microextraction.

A Thermo Scientific Trace 1300 gas chromatography coupled with a Thermo Scientific Q-Exactive Orbitrap mass spectrometer (GC-Orbitrap-MS, Thermo Scientific, Bremen, Germany) was used to analyze volatile compounds in sunflower seed oil. The chromatographic column is HP-INNOWAX capillary column (60 m × 0.25 μm, 0.25 μm, J & W Scientific, Santa Clara, CA). The temperature increase procedure of chromatography was as follows: 40 °C for 5 min, 150 °C at a rate of 3 °C/min, and then 230 °C at a rate of 5 °C/min. The carrier gas was helium (purity: 99.999%), and the flow rate was 1 mL/min. MS acquisition was performed in profile mode using an *m/z* range of 33–350. The resolution power was set at 60,000 full widths at half-maximum (FWHM) at *m/z* 200. Data acquisition and analyses were performed using the Xcalibur version 4.1 software, with the processing setups Quan Browser and Qual Browser (Thermo Fisher Scientific, Les Ulis, France). Each analysis was performed in duplicate (Y. Liu et al., 2022). The retention indices were obtained by injection of the C6–C24 *n*-alkane series (Supelco, Bellefonte, PA, USA) mixture under the same chromatographic conditions.

The compounds were qualitatively analyzed by combining the calculated RI value, mass spectrum information, and retention index. The relative abundance of the compounds in the samples was expressed the peak area ratio (J. Zhang et al., 2010). Three repeated injections were performed for each sample.

Quantitative descriptive analysis

The sensory characteristics of sunflower seed oil products were evaluated by scoring the intensity of each sensory attribute of sunflower seed oil products. The panelists in our evaluation team all have more than two years of experience in sensory evaluation. The definitions of six sunflower seed oil sensory property descriptors including 'sunflower seeds', 'burnt', 'fried instant noodles', 'sweet', 'puffed food' and 'roasted sunflower seeds' were determined through discussion, and reference samples were prepared for evaluator training. The six sensory descriptors are defined in Table S2. The panel were required to score the intensity of each characteristic sensory attribute, with a score range of 0–10 points (linear scale). After a month of training, they started the evaluating of the samples. The higher the score, the greater the intensity of the representative attribute. The evaluation was repeated twice.

Data analysis

Data analyses were performed using Origin 2021 (MicroCal, Massachusetts, USA) and XLSTAT 19 (Addinsoft, New York, NY). One-way analysis of variance (ANOVA) was used to compare the significant differences between the means using Tukey's post hoc test. Statistical significance was set at $\alpha = 0.05$. A bubble chart was constructed to show the peak area ratios of the compounds jointly detected using these two methods. The compounds detected in this study and their peak areas in each sample were analyzed using a cluster analysis model of agglomerative hierarchical clustering (AHC). Principal component analysis (PCA) was performed on the average intensity values of each sensory attribute obtained from the QDA to determine the sensory characteristics of the samples and their relationships with volatile compounds. Partial least squares regression (PLSR) based modeling was performed to explore the relationship between sensory attributes and volatile composition. In addition, only chemical components with variable importance to projection (VIP) value>1 were retained in the final result of the PLSR models. The data were auto-scaled (mean-centered and divided by the standard deviation of each variable).

Results

Identification of volatiles from sunflower seed oil samples

The volatile compounds were analyzed in seven samples based on HS-SPME-GC-Quadrupole-MS (Table 1) and HS-SPME-GC-Orbitrap-MS (Table 2), respectively.

A total of 46 volatile compounds were detected using GC-Quadrupole-MS. The detected compounds included 8 pyrazines, 3 lower alcohols, 4 furans, 1 compound with benzene ring, 15 aldehydes, 5 acids, 1 ester, 6 terpenes, 1 amide and 2 pyrrole compounds. In contrast, 107 volatile compounds were detected by GC-Orbitrap-MS, including 11 pyrazines, 9 lower alcohols, 10 higher alcohols, 5 furans, 6 compounds with benzene rings, 3 sulfur compounds, 14 aldehydes, 13 esters, 3 acids, 12 terpenes, 11 ketones, 3 alkanes, 2 nitrogen-containing compounds, 1 pyridine compound and 4 pyrrole compounds.

The HRMS specifically detected 72 volatile compounds, whereas 13 compounds were identified by LRMS specifically, and 33 compounds were detected simultaneous (Fig. 1 (a)). It could be seen from Fig. 1 (b) that among the compounds specifically detected by HRMS, there were 3 pyrazines, 14 alcohols, 4 furans, 6 compounds containing benzene rings, 3 sulfides, 7 aldehydes, 12 esters, 12 terpenes, 5 ketones, 2 alkanes, 4 nitrogen-containing heterocycles. The compounds specifically detected by LRMS included two furan compounds, seven aldehydes, two acids, one terpene, and a single amide. Overall, HRMS detected a higher number of volatile compounds in sunflower seed oil than LRMS. HRMS were more suitable for qualitative detection of alcohols, esters, terpenoids and ketones in sunflower seed oil in terms of the types of compounds specifically detected, LRMS might be more suitable for qualitative detection of aldehydes and acids in the samples. It is evident from Fig. 1 (a) that HRMS can characterize more compounds than LRMS, which may be due to the lower mass spectral sensitivity of LRMS. Interestingly, HRMS, which has the advantages of high resolution and high sensitivity, still failed to characterize 13 compounds. This may be due to the relatively low mass charge of the ionic fragments of the compound, which is difficult to match during characterization. For instance, acetamide, which has a molecular weight of 59, probably failed to be characterized by HRMS because it generates major fragments with a mass-to-charge ratio below 50 *m/z* during ionization by the ion source. As far as we know, the present study reports for the first time 23 volatile compounds in sunflower seed oil.

Esters are generally considered aroma-rich compounds and it has been demonstrated in a variety of media that they have major synergistic and aroma-presenting effects (De-la-Fuente-Blanco, Saenz-Navajas, Valentin, & Ferreira, 2020; Jia et al., 2021; Xiao, Luo, Niu, Wang, Wang, & Sun, 2019). In contrast, the reports of esters in studies on sunflower seed oil were rare. In this study, 8 of the 13 esters including methyl acetate (E1), isoamyl acetate (E2), butyl acrylate, (E4), ethyl lactate (E5), ethyl (S)-(-)-lactate (E7), citronellyl acetate (E10), myrtenyl acetate (E11), methyl salicylate (E12) were first detected in sunflower seed oil by HRMS. These compounds were firstly detected in sunflower seed oils. The esters were probably produced by the decomposition of

Table 1
List of volatile compounds identified in the seven sunflower seed oils studied by GC-Quadrupole-MS.

Categories	Compound	Structure	CAS	Quantitative ion	Actual RI	Ident[a]
Pyrazines	Pyrazine	$C_4H_4N_2$	290–37-9	80	1357	St, RI, QMS
	2-Ethyl-6-methylpyrazine	$C_7H_{10}N_2$	13925–03-6	121	1389	St, RI, QMS
	2,3,5-Trimethylpyrazine	$C_7H_{10}N_2$	14667–55-1	42	1401	St, RI, QMS
	Methylpyrazine	$C_5H_6N_2$	109–08-0	94	1418	St, RI, QMS
	3-Ethyl-2,5-dimethylpyrazine	$C_8H_{12}N_2$	13360–65-1	135	1440	St, RI, QMS
	2-Ethyl-5-methyl pyrazine	$C_7H_{10}N_2$	13360–64-0	121	1441	St, RI, QMS
	2,5-Dimethylpyrazine	$C_6H_8N_2$	123–32-0	108	1482	St, RI, QMS
	2,6-Dimethylpyrazine	$C_6H_8N_2$	108–50-9	108	1488	St, RI, QMS
Alcohol	Methyl Alcohol	CH_4O	67–56-1	31	968	St, RI, QMS
	1-Pentanol	$C_5H_{12}O$	71–41-0	55	1253	St, RI, QMS
	1-Octen-3-ol	$C_8H_{16}O$	3391–86-4	57	1440	St, RI, QMS
	2-Furanmethanol	$C_5H_6O_2$	98–00-0	98	1653	St, RI, QMS
Furan	2-Methylfuran	C_5H_6O	534–22-5	82	936	St, RI, QMS
	Dihydro-2-methyl-3(2H)-furanone	$C_5H_8O_2$	3188–00-9	43	1271	St, RI, QMS
	2-Pentylfuran	$C_9H_{14}O$	3777–69-3	81	1376	St, RI, QMS
	2(5H)-Furanone	$C_4H_4O_2$	497–23-4	84	1967	St, RI, QMS
Aldehyde	2-Methylpropanal	C_4H_8O	78–84-2	43	814	St, RI, QMS
	Butanal	C_4H_8O	123–72-8	44	878	St, RI, QMS
	Pentanal	$C_5H_{10}O$	110–62-3	44	981	St, RI, QMS
	3-Methylbutanal	$C_5H_{10}O$	590–86-3	44	982	St, RI, QMS
	α-Methylbutanal	$C_5H_{10}O$	96–17-3	57	995	St, RI, QMS
	Hexanal	$C_6H_{12}O$	66–25-1	44	1095	St, RI, QMS
	n-Heptaldehyde	$C_7H_{14}O$	111–71-7	70	1193	St, RI, QMS
	(E)-2-Hexenal	$C_6H_{10}O$	6728–26-3	41	1228	St, RI, QMS
	(E)-2-Octenal	$C_8H_{14}O$	2548–87-0	41	1329	St, RI, QMS
	n-nonanal	$C_9H_{18}O$	124–19-6	57	1390	St, RI, QMS
	Furfural	$C_5H_4O_2$	98–01-1	96	1463	St, RI, QMS
	(Z)-2-Heptenal	$C_7H_{12}O$	57266–86-1	83	1485	St, RI, QMS
	Decyl aldehyde	$C_{10}H_{20}O$	112–31-2	43	1493	St, RI, QMS
	Benzaldehyde	C7H6O	100–52-7	106	1523	St, RI, QMS
	(E,E)-2,4-decadienal	$C_{10}H_{16}O$	25152–84-5	81	1814	St, RI, QMS
Acid	Acetic acid	$C_2H_4O_2$	64–19-7	43	1442	St, RI, QMS
	Propanoic acid	$C_3H_6O_2$	79–09-4	74	1526	St, RI, QMS
	Butanoic acid	$C_4H_8O_2$	107–92-6	60	1616	St, RI, QMS
	4-Hydroxybutanoic acid	$C_4H_8O_3$	591–81-1	86	1829	St, RI, QMS
	Hexanoic acid	$C_6H_{12}O_2$	142–62-1	60	1838	St, RI, QMS
Easter	Methyl acetate	$C_3H_6O_2$	79–20-9	43	828	St, RI, QMS
Terpene	α-Pinene	$C_{10}H_{16}$	80–56-8	93	1022	St, RI, QMS
	α-Phellandrene	$C_{10}H_{16}$	99–83-2	93	1123	St, RI, QMS
	α-Thujene	$C_{10}H_{16}$	2867–05-2	93	1131	St, RI, QMS
	Camphene	$C_{10}H_{16}$	79–92-5	93	1174	St, RI, QMS
	α-terpineol	$C_{10}H_{18}O$	98–55-5	93	1249	St, RI, QMS
	γ-Terpinene	$C_{10}H_{16}$	99–85-4	93	1249	St, RI, QMS
Amide	Acetamide	C_2H_5NO	60–35-5	59	1962	St, RI, QMS
Pyrrole	1-(1H-pyrrol-2-yl)1-ethanone	C_6H_7NO	1072–83-9	94	1978	St, RI, QMS
	1H-Pyrrole-2-carboxaldehyde	C_5H_5NO	1003–29-8	95	2035	St, RI, QMS

[a]Basis for identification: St, standard compound; QMS, quadrupole GC–MS; RI: RI agreed with data base of NIST11.

the hydroperoxides generated by the oxidation of oils (W. Zhang et al., 2020).

In recent years, small-molecule sulfides have been detected that can provide 'sulfur', 'fatty' and 'cabbage' flavors to vegetable oils (Yini Yang et al., 2022). Among the three sulfides detected in this study using HRMS, dimethyl sulfone (S3) was reported for the first time in sunflower seed oil. This compound has been detected in rapeseed oil (Zhou et al., 2019) and raw cabbage and has been described as 'sulfurous' and 'fatty' (Marcinkowska, Frank, Steinhaus, & Jeleń, 2021). A rencent research generally suggested that the sulfur compounds in vegetable oils came from the oilseed crop itself or that sulfur-containing precursors react with enzymes during processing (Yu et al., 2022).

To our knowledge, this study reported 23 volatiles in sunflower seed oil for the first time, including the 8 esters and 1 sulfur-containing compound mentioned above in addition to verbenol (T14, HRMS), myrtenol (T17, HRMS), α-phellandrene (T5, LRMS and HRMS), p-cymene (T7, HRMS), menthol (T13, HRMS), azulene (ALK2, HRMS), 5-ethyl-cyclopent-1-enecarboxaldehyde (AL12, HRMS), 1-ethyl-cyclohexene (ALK1, HRMS), phenol (B6, HRMS), styrene (B1, HRMS), 1-methyl-2-(2-propenyl)benzene (B2, HRMS), 1-methyl-naphthalene (B3, HRMS), 2-methyl-naphthalene (B4, HRMS), and butylated hydroxytoluene (B5, HRMS).

Quantitative comparison of volatiles among sunflower seed oil samples studied

The volatile compounds were quantified using an internal standard method, and the peak area ratios were calculated for each compound in the samples. The quantitative results from these two methods are plotted in a bubble diagram for the 33 substances detected simultaneously using HRMS and LRMS (Fig. S1). It can be observed from Fig. S1 that some compounds may be effectively detected in the samples by LRMS, e.g. Acetic acid, which was detected by LRMS in all seven samples, whereas in HRMS the compound was quantified in only two samples; this observation was similar for compounds including *n*-heptaldehyde and α terpineol. Conversely, some compounds may be better detected in samples by HRMS, such as 2-Ethyl-6-methylpyrazine, which was detected by HRMS in all seven samples but only quantified in four samples by LRMS. The occurrence of the aforementioned may be a result of variations or inherent qualities of the device; however, we decided to quantify these compounds using a more efficient mass spectrometry method. Among these 33 compounds, some compounds could be quantified in LRMS, but with very small relative concentrations and also without differences between samples, whereas these compounds were well represented in HRMS, such as γ-Terpinene and (E)-2-Hexenal. This

Table 2
List of volatile compounds identified in the seven sunflower seed oils studied by GC-Orbitrap-MS.

Categories	Compound	Structure	CAS	Quantitative ion	Actual RI	Ident[a]
Pyrazines	Pyrazine	$C_4H_4N_2$	290–37-9	80.036942	1200	O MS
	Methylpyrazine	$C_5H_6N_2$	109–08-0	94.05249	1249	St, RI, O MS
	2,5-dimethylpyrazine	$C_6H_8N_2$	123–32-0	108.06814	1300	St, RI, O MS
	Pyrazine, 2,6-dimethyl-	$C_6H_8N_2$	108–50-9	108.06822	1307	O MS
	Pyrazine, ethyl-	$C_6H_8N_2$	13925–00-3	107.06043	1312	St, RI, O MS
	Pyrazine, 2,3-dimethyl-	$C_6H_8N_2$	5910–89-4	108.06822	1324	O MS
	2-Ethyl-5-methyl pyrazine	$C_7H_{10}N_2$	13360–64-0	121.07604	1364	St, RI, O MS
	2-Ethyl-6-methylpyrazine	$C_7H_{10}N_2$	13925–03-6	121.07604	1370	St, RI, O MS
	2,3,5-Trimethylpyrazine	$C_7H_{10}N_2$	14667–55-1	122.08369	1383	St, RI, O MS
	3-Ethyl-2,5-dimethylpyrazine	$C_8H_{12}N_2$	13360–65-1	135.09175	1426	St, RI, O MS
	5-Ethyl-2,3-dimethylpyrazine	$C_8H_{12}N_2$	15707–34-3	135.09184	1443	St, RI
Alcohol	Methyl Alcohol	CH_4O	67–56-1	33.033463	905	O MS
	2-nonanol	$C_9H_{20}O$	628–99-9	45.033535	932	St, RI
	2-Propanol, 1-methoxy-	$C_4H_{10}O_2$	107–98-2	45.033535	1127	O MS
	1-Propanol, 2-methyl-	$C_4H_{10}O$	78–83-1	41.038399	1147	St, RI
	1-Pentanol	$C_5H_{12}O$	71–41-0	41.03841	1245	St, RI
	Acetone alcohol	$C_3H_6O_2$	116–09-6	43.017773	1277	St, RI
	3-Cyclohexene-1-methanol, 6-methyl-	$C_8H_{14}O$	5259–31-4	93.069931	1317	O MS
	1-Pentanol, 4-methyl-	$C_6H_{14}O$	626–89-1	41.038399	1342	O MS
	1-Butanol, 2-ethyl-	$C_6H_{14}O$	97–95-0	43.017803	1367	O MS
	1-Octen-3-ol	$C_8H_{16}O$	3391–86-4	57.033539	1440	St, RI, O MS
	1-Heptanol	$C_7H_{16}O$	111–70-6	41.038399	1446	St, RI
	2,3-Butanediol	$C_4H_{10}O_2$	513–85-9	45.033535	1531	St, RI
	2,3-Butanediol, [S-(R*,R*)]-	$C_4H_{10}O_2$	19132–06-0	45.033535	1571	O MS
	Propylene Glycol	$C_3H_8O_2$	57–55-6	45.033535	1583	O MS
	2-Furanmethanol	$C_5H_6O_2$	98–00-0	97.02845	1654	St, RI
	2-Furanmethanol, 5-methyl-	$C_6H_8O_2$	3857–25-8	112.05201	1718	O MS
	Benzenemethanol, α,α-dimethyl-	$C_9H_{12}O$	617–94-7	121.06496	1754	O MS
	Benzyl alcohol	C_7H_8O	100–51-6	79.054298	1870	O MS
Furan	Furan, 3-methyl-	C_5H_6O	930–27-8	82.041397	885	O MS
	2-Pentylfuran	$C_9H_{14}O$	3777–69-3	81.033531	1221	St, RI, O MS
	3(2H)-Furanone, dihydro-2-methyl-	$C_5H_8O_2$	3188–00-9	43.017803	1247	O MS
	Acetylfuran	$C_6H_6O_2$	1192–62-7	95.012772	1483	St, RI
	1-Pentanone,1-(2-furanyl)-	$C_9H_{12}O_2$	3194–17-0	110.03626	1749	St, RI
Benzenes	Styrene	C_8H_8	100–42-5	104.06213	1239	St, RI
	Benzene, 1-methyl-2-(2-propenyl)-	$C_{10}H_{12}$	1587–04-8	117.07002	1413	O MS
	Naphthalene, 1-methyl-	$C_{11}H_{10}$	90–12-0	141.07001	1829	O MS
	Naphthalene, 2-methyl-	$C_{11}H_{10}$	91–57-6	141.07001	1865	O MS
	Butylated Hydroxytoluene	$C_{15}H_{24}O$	128–37-0	205.15872	1908	O MS
	Phenol	C_6H_6O	108–95-2	94.041405	1991	St, RI
Sulfur	Carbon disulfide	CS_2	75–15-0	75.943642	713	O MS
	Dimethyl Sulfoxide	C_2H_6OS	67–68-5	78.013474	1556	O MS
	Dimethyl sulfone	$C_2H_6O_2S$	67–71-0	78.984879	1885	O MS
Aldehyde	2-Methylpropanal	C_4H_8O	78–84-2	41.03841	859	St, RI
	Hexanal	$C_6H_{12}O$	66–25-1	41.03841	1078	St, RI
	n-Heptaldehyde	$C_7H_{14}O$	111–71-7	55.054306	1176	St, RI
	(E)-2-Hexenal	$C_6H_{10}O$	6728–26-3	41.03841	1204	St, RI
	Octanal	$C_8H_{16}O$	124–13-0	41.038399	1271	St, RI
	2-Heptenal, (E)-	$C_7H_{12}O$	18829–55-5	83.04921	1300	St, RI
	n-nonanal	$C_9H_{18}O$	124–19-6	41.03841	1372	St, RI
	5-Ethylcyclopent-1-enecarboxaldehyde	$C_8H_{12}O$	36431–60-4	67.054298	1389	O MS
	(E)-2-Octenal	$C_8H_{14}O$	2548–87-0	41.03841	1405	St, RI
	Furfural	$C_5H_4O_2$	98–01-1	95.012825	1442	St, RI
	Trans −2- nonanal	$C_9H_{16}O$	18829–56-6	41.03841	1520	St, RI
	5-Methyl-2-furancarboxaldehyde	$C_6H_6O_2$	620–02-0	109.02845	1553	St, RI
	Benzeneacetaldehyde	C_8H_8O	122–78-1	91.054245	1621	St, RI
	Benzaldehyde, 2-hydroxy-	$C_7H_6O_2$	90–02-8	121.02856	1658	O MS
	2E,4E-Decadienal	$C_{10}H_{16}O$	25152–84-5	81.033554	1798	St, RI
	1H-Pyrrole-2-carboxaldehyde	C_5H_5NO	1003–29-8	94.028748	2001	St, RI, O MS
Esters	Methyl acetate	$C_3H_6O_2$	79–20-9	43.017773	867	St, RI
	Isoamyl acetate	$C_7H_{14}O_2$	123–92-2	43.054199	1054	St, RI
	Butyl propionate	$C_7H_{14}O_2$	590–01-2	75.044151	1138	O MS
	Butyl acrylate	$C_7H_{12}O_2$	141–32-2	55.017899	1170	O MS
	Ethyl lactate	$C_6H_8N_2$	97–64-3	45.033535	1336	St, RI
	Allyl tiglate	$C_8H_{12}O_2$	7493–71-2	83.04921	1347	O MS
	Ethyl (S)-(-)-lactate	$C_5H_{10}O_3$	687–47-8	45.033535	1385	O MS
	trans-3-Hexenyl butyrate	$C_{10}H_{18}O_2$	53398–84-8	67.054298	1481	O MS
	(-)-Bornyl acetate	$C_{12}H_{20}O_2$	5655–61-8	95.08564	1562	O MS
	Citronellyl acetate	$C_{12}H_{22}O_2$	150–84-5	95.04924	1641	St, RI
	Myrtenyl acetate	$C_{12}H_{18}O_2$	1079–01-2	91.054306	1643	O MS
	Methyl salicylate	$C_8H_8O_3$	119–36-8	120.02071	1757	St, RI
Acid	Propanoic acid	$C_3H_6O_2$	79–09-4	74.03624	948	St, RI
	Acetic acid	$C_2H_4O_2$	64–19-7	43.017757	1428	St, RI
	Hexanoic acid	$C_6H_{12}O_2$	142–62-1	73.028442	1893	St, RI

(*continued on next page*)

Table 2 (*continued*)

Categories	Compound	Structure	CAS	Quantitative ion	Actual RI	Ident[a]
Terpene	α-Pinene	$C_{10}H_{16}$	80–56-8	91.054245	1013	St, RI
	Camphene	$C_{10}H_{16}$	79–92-5	93.069878	1055	St, RI
	β-Pinene	$C_{10}H_{16}$	127–91-3	93.069878	1096	St, RI, O MS
	Sabinene	$C_{10}H_{16}$	3387–41-5	91.054245	1112	St, RI, O MS
	α-Phellandrene	$C_{10}H_{16}$	99–83-2	91.054245	1154	St, RI
	γ-Terpinene	$C_{10}H_{16}$	99–85-4	91.054245	1230	St, RI
	p-Cymene	$C_{10}H_{14}$	99–87-6	119.08569	1251	St, RI
	α-Campholenal	$C_{10}H_{16}O$	4501–58-0	93.069931	1466	O MS
	5,7-Octadien-4-one, 2,6-dimethyl-, (Z)-	$C_{10}H_{16}O$	3588–18-9	95.08564	1487	O MS
	(±)-Linalool	$C_{10}H_{18}O$	78–70-6	93.069878	1540	St, RI
	Cyclosativene	$C_{15}H_{24}$	22469–52-9	161.13246	1541	O MS
	Calarene	$C_{15}H_{24}$	17334–55-3	161.13246	1569	O MS
	(±)-Terpinen-4-ol	$C_{10}H_{18}O$	562–74-3	93.069878	1588	St, RI
	Menthol	$C_{10}H_{20}O$	89–78-1	81.069984	1632	St, RI
	Verbenol	$C_{10}H_{16}O$	473–67-6	79.054298	1671	O MS
	Bicyclo[3.1.1]hept-3-en-2-one, 4,6,6-trimethyl-	$C_{10}H_{14}O$	80–57-9	107.08566	1686	O MS
	α-terpineol	$C_{10}H_{18}O$	98–55-5	121.10121	1694	St, RI
	Myrtenol	$C_{10}H_{16}O$	515–00-4	79.054298	1787	St, RI
Ketone	3-Penten-2-one, (E)-	C_5H_8O	3102–33-8	69.033562	1121	O MS
	Acetoin	$C_4H_8O_2$	513–86-0	45.033497	1265	St, RI, O MS
	4-Cyclopentene-1,3-dione	$C_5H_4O_2$	930–60-9	68.025772	1563	O MS
	2(3H)-Furanone, dihydro-4,5-dimethyl-	$C_6H_{10}O_2$	6971–63-7	42.046307	1602	O MS
	2(5H)-Furanone	$C_4H_4O_2$	497–23-4	55.017818	1733	St, RI, O MS
	Pantolactone	$C_6H_{10}O_3$	599–04-2	71.04921	2006	St, RI
Alkanes	Cyclohexene, 1-ethyl-	C_8H_{14}	1453–24-3	81.069984	1481	O MS
	Azulene	$C_{10}H_8$	275–51-4	128.06216	1717	O MS
Nitrogen-containing heterocycles	Pyridine	C5H5N	110–86-1	79.04174	1173	O MS
	Pyrrole	C_4H_5N	109–97-7	67.041733	1498	O MS
	1H-Pyrrole, 3-methyl-	C_5H_7N	616–43-3	80.049561	1558	O MS
	1H-Pyrrole-2-carboxaldehyde, 1-methyl-	C_6H_7NO	1192–58-1	108.0445	1599	O MS
	1-(1H-pyrrol-2-yl)1-ethanone	C_6H_7NO	1072–83-9	94.028748	1958	St, RI, O MS

[a]Basis for identification: St, standard compound; O MS, Orbitrap-MS; RI: RI agreed with data base of NIST11.

is probably because the concentrations of these compounds in the samples were below the minimum detection limit of LRMS and outside the linear range of quantification by LRMS. Therefore, they could only be quantified by HRMS, which has a much lower detection limit. According to the aforementioned observations, among these 33 compounds, 23 were quantified using HRMS and the remaining 10 using LRMS.

Finally, 96 compounds, including 18 alcohols, 12 esters, 7 ketones, 20 terpenoids, 11 pyrazines, 6 aldehydes, 6 furans, 6 benzene ring-containing compounds, 3 sulfides, 2 alkanes, and 5 nitrogen-containing compounds, were quantified using HRMS, whereas 22 compounds including 5 acids, 1 amide, and 16 aldehydes were quantified using LRMS. The mean values were calculated and 108 out of 118 compounds were found to have significantly different contents among the samples based on ANOVA ($p \leq 0.05$ considered to indicate significantly different) and the results were shown in Table S3. The relative contents did not differ significantly between samples ($p > 0.05$) for 2-methylfuran, dihydro-2-methyl-3(2H)-furanone, α-thujene, benzaldehyde, α-phellandrene, *trans*-2-nonanal, 5-methyl-2-furancarboxaldehyde, benzeneacetaldehyde, benzaldehyde, 2-hydroxy-, and α-campholenal, therefore, they were excluded from the subsequent statistical analysis.

After normalization, a cluster heat map was created to observe the distribution of the 108 compounds among the samples (Fig. 2). These compounds could be divided into two major categories. There were 60 compounds in Group 1 (G1), including 11 terpenoids. In sunflower seed oil, these compounds were usually from sunflower seeds. Terpenoids have also been identified as key aromatic compounds in cold-pressed sunflower seed oils (Yin, Shi, Li, Ma, et al., 2022). It should be noted that terpenoids might also be converted into other components during heat treatment (Yin, Shi, Li, Ma, et al., 2022). Group 2 (G2) consisted of 48 compounds, including 10 pyrazines and all 3 sulfur-containing compounds detected in this study.

Among the 108 compounds that qualified in JXZ, 37 were considerably higher in the samples than the other six, mainly aldehydes and terpenoids. Among these 37 compounds, 3-ethyl-2,5-dimethylpyrazine (NH10) was 2.8 times higher than the content of this compound in JN, two alcohols (Al-ol4 and Al-ol18) were 10 times higher than the samples with the lowest content on average, while seven other compounds (Al-ol7, Al-ol8, Al-ol10, Al-ol18, T14, T17, and Al-ol20) were 12 times higher than the samples with the lowest content on average, and one furan (F5, with a relative concentration of 0.002) was only detected in this sample. Five compounds with benzene rings (B1, B2, B4, B5, B6) were 14 times higher than the samples with the lowest content on average, the four esters (E8, E9, E11, E12) were 19 times higher than the samples with the lowest content on average, the concentration range of 11 terpenoids (T1-7, T9, T11-13) was 0.003–9.114, and the four other compounds (AL9, AL10, T8, T15) were 6 times higher than the samples with the lowest content on average. One alkane (ALK1, 0.088) was only detected in this sample.

Twenty-nine compounds in LH, mainly pyrazines, were significantly more abundant than those in the other samples. Among them, 10 pyrazines (NH1-9 and NH11) were 32 times higher than the samples with the lowest content, 2 alcohols (Al-ol15 and Al-ol17), 3 furans (F1, F3 and F4), 1 amide (AM1), 2 sulfides (S1, S2), 3 ester compounds (E1, E5, and E7), 4 ketone compounds (K2, K4, K5, and K6), 1 pyridine compound (N1), 3 pyrrole compounds (N2, N4, and N5). 19 compounds in JZ that were significantly more abundant than other samples, including 4 alcohols (Al-ol1, Al-ol3, Al-ol4, Al-ol14), 5 aldehydes (AL1, AL5, AL13, AL14, AL16), 1 acid (A4), 3 compounds with benzene rings (B3-5), 2 esters (E2, E3), 1 ketone (K3), and 2 other compounds (T10, ALK2).

The compounds in HK that were significantly higher than those in other samples were: Al-ol5 (relative concentration is 0.122), which was 6 times the content of this compound in LH, F2 (0.349, relative concentration, the same below) was 7 times the content of this compound in LH, AL4 (0.337) was 5.8 times the content of this compound in JXZ, and AL6 (1.664) was 3.8 times the content of this compound in JXZ. There were 12 compounds in HG that are significantly higher than other samples, including 4 alcohols (Al-ol1, Al-ol2, Al-ol12, Al-ol13), 3 aldehydes (AL7, AL13, AL16), 1 acid (A4), 1 compound with benzene ring

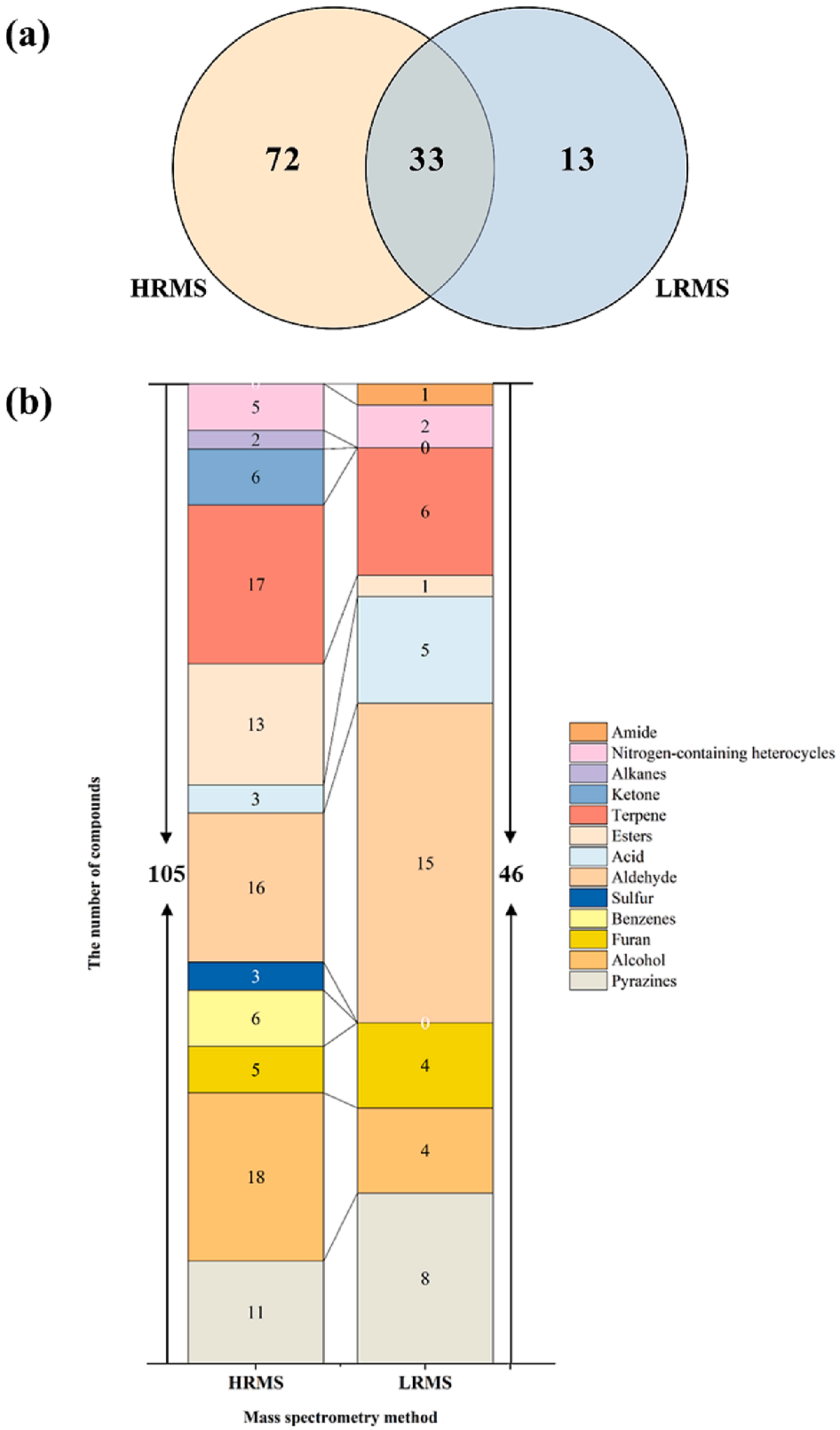

Fig. 1. Comparison of the qualitative effects of HRMS and LRMS.

(B5), 1 sulfide (S3).

Ten compounds with no significant difference ($p > 0.05$) in content as mentioned previously were excluded from the subsequent statistical analysis.

Sensory characteristics of sunflower seed oil samples

QDA was conducted to quantitatively describe the aroma characteristics of seven commercial sunflower seed oils by a well-trained panel. One-way ANOVA was performed on the quantitative description of the

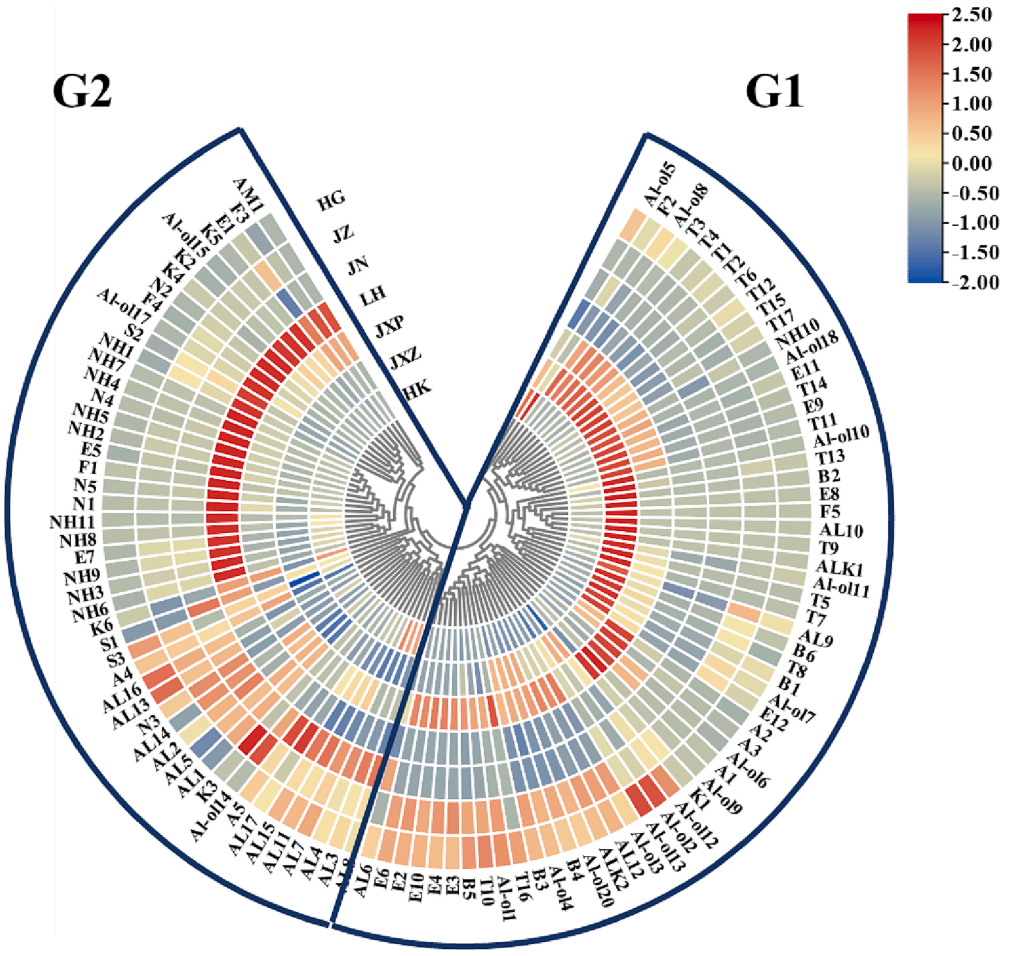

Fig. 2. The cluster Heatmap showing the relative content of compounds in seven sunflower oil samples.

results, and all the six specific notes were found to be significantly different ($\alpha < 0.05$) among the samples. HCA was performed, as shown in Fig. S2.

The intensity of 'roasted sunflower seeds' aroma ranged from 2.8 to 4.9 in the samples, with the intensity of this attribute scoring 4.0 and above in all samples except JXZ. The intensity of 'sunflower seeds' aroma ranged from 0.9 to 5.9, reaching the highest value in JXZ and the lowest value in LH. The intensity distribution of 'burnt' aroma in the samples ranged from 2.5 to 5.5. The 'fried instant noodles' aroma had an intensity distribution in the samples between 0.3 and 4.3, reaching a minimum of 0.3 and 0.4 in JXP and LH. Only JXZ, JZ and HG were described as a 'sweet' aroma with intensities of 1.0, 2.0 and 2.7, respectively. The 'puffed food' aroma was only identified in JXP and LH, with intensities of 5.3 and 6.3, respectively. Overall, all samples had 'sunflower seeds' aroma, 'roasted sunflower seeds' aroma and 'burnt' aroma with generally high scores.

'Fried instant noodles' aroma, 'puffed food' aroma and 'sweet' aroma were only present in part of the samples or very weakly in some samples. The intensity of 'fried instant noodle' aroma in the samples ranged from 0.3 to 4.3, reaching a maximum in HK and a minimum in the JXP, and in fact LH also exhibited a very weak 'fried instant noodle' aroma (0.4). A 'sweet' aroma was only found in JXZ, JZ and HG, with distributions between 1.0 and 2.7. In parallel, only JXP and LH were described as 'puffed food' aroma, with scores of 5.3 and 6.3, respectively.

Principal component analysis on QDA results

The QDA results of these seven samples were used for the principal component analysis, and the peak areas of the compounds were introduced as supplementary variables. The results are shown in Fig. 3. The first two principal components together contributed 80.80% of the variance, and the seven samples were well-differentiated. JXP and LH were identified as 'puffed food'. JXZ had a more pronounced 'sunflower seeds', while the rest of the samples were closer to 'fried instant noodles' and 'sweet'. The panel described LH as having higher 'puffed food' and 'burnt' scores. As can be seen from Fig. 3, the seven samples can be classified into three categories according to their aroma characteristics, including JXZ, which is closer to 'sunflower seeds' aroma; HG, JZ, HK and JN, which are closer to 'sweet' and 'fried instant noodles'; and JXP and LH, which are closer to 'puffed food' and 'burnt'. Undoubtedly, the aroma of sunflower seed oil is altered by thermal processing, and sunflower seed oil exhibits different aroma characteristics depending on the process and the degree of thermal processing.

The distribution pattern of the complementary variables showed that some of the detected compounds may be closely related to the sensory properties of the sample. For example, the 'puffed food' aroma was more closely located to several pyrazines (NH2, NH1, NH3, NH8, NH9, etc.), and ketones such as K2, K4, and K5, in addition to AM1, Al-ol6, Al-ol15, F3, and F4. Moreover, S3 was correlated with 'roasted sunflower seeds' due to its close location. 'Sweet' was closer to AL16, Al-ol13 and other compounds, which may also be correlated.

PLSR analysis

To further understand the relationship of volatile compounds and the six sensory attributes of sunflower seed oils, PLSR models were constructed and the results were shown in Fig. 4, and all models were optimized to $Q^2 > 0.4$ and $R^2Y > 0.6$. Compounds with VIP > 1 and

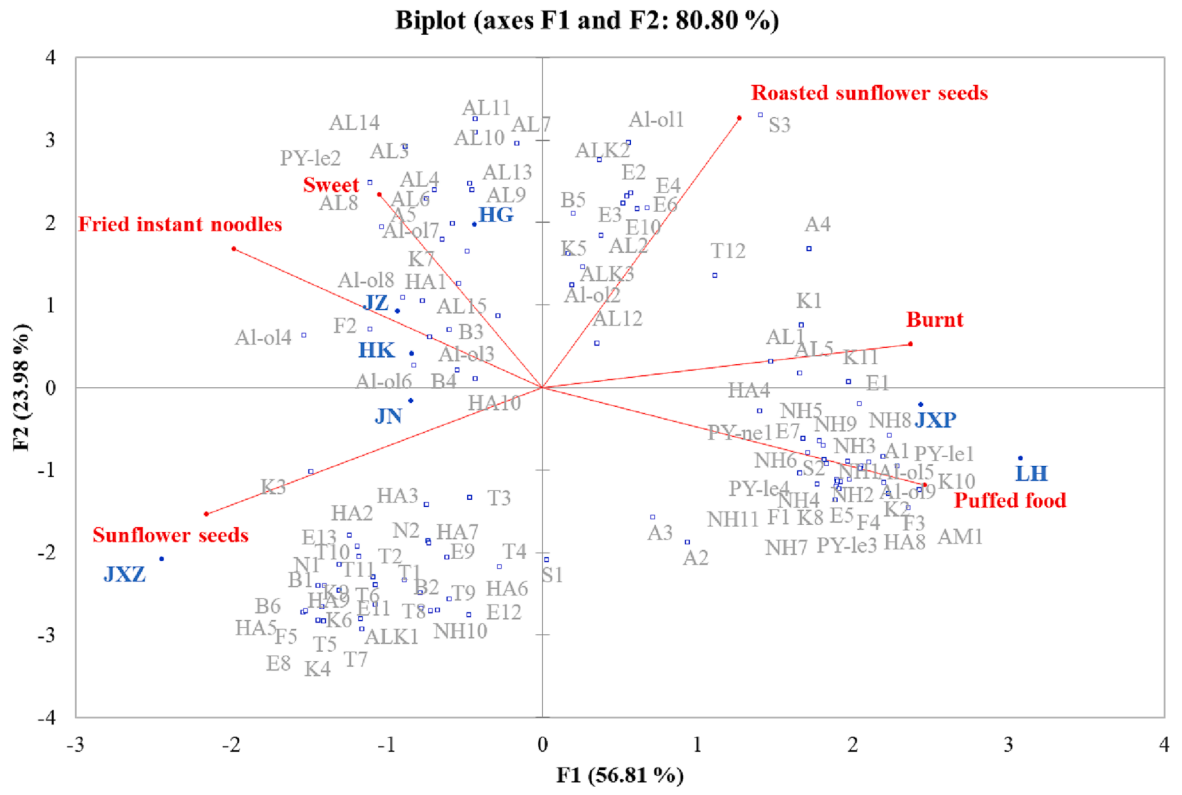

Fig. 3. The principal component analysis model showing the sensory attribute scores of six sunflower oil samples.

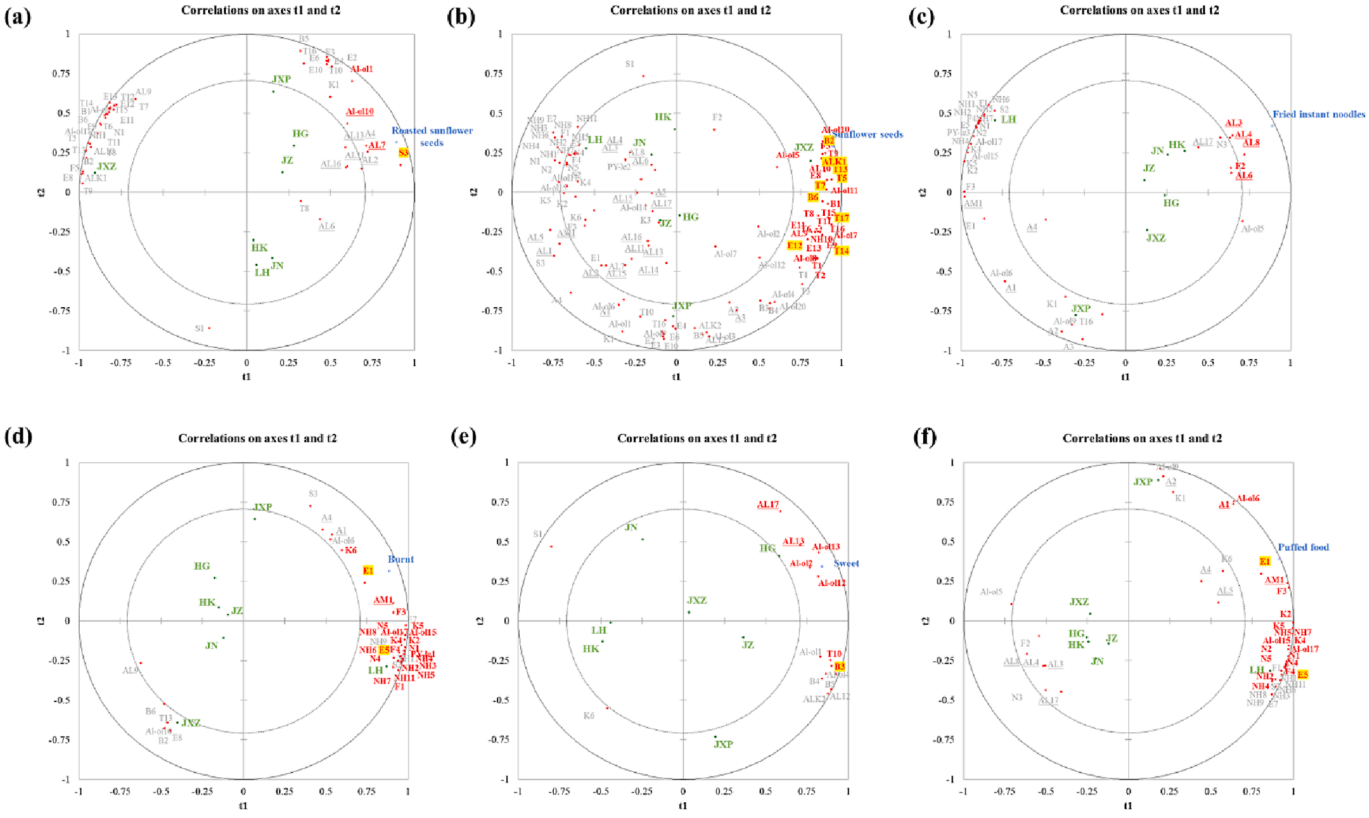

Fig. 4. Partial least squares regression analysis for aroma intensity variables and volatile compounds content variables in six sunflower oils.

correlation coefficient > 0 in each model were marked in red in Fig. 4 and listed in Table S4.

As shown in Table S4 and Fig. 4 (a), the 'roasted sunflower seeds' aroma of sunflower seed oil was positively correlated with 1-octen-3-ol (mushroom-like, 0.045) (Neugebauer, Schieberle, & Granvogl, 2021), *n*-heptaldehyde (citrus-like, fatty, 0.031) (Neugebauer, Granvogl, & Schieberle, 2020) and dimethyl sulfone (sulfurous, fatty, 0.045) (Marcinkowska et al., 2021). Among them, 1-octen-3-ol was also sniffed in GC-O-MS by SAFE pre-treatment method only in a study by Yin et al (Yin, Shi, Li, Ma, et al., 2022). In addition, the 23 compounds reported for the first time in this study with dimethyl sulfone correlated with this aroma. Xu et al. also mentioned in their study on flavor improvement of fried foods that *n*-heptaldehyde may provide flavor associated with frying or heating (Xu, Mei, Wu, Karrar, Jin, & Wang, 2022).

From Table S4 and Fig. 4 (b), it was evident that the number of aroma compounds correlating tightly with the 'sunflower seeds' was very high, including phenol (ink-like, phenolic, 0.021) (Zhai & Granvogl, 2020) and three other benzene compounds, p-cymene (terpene-like, 0.021) (Steinhaus & Schieberle, 2005), menthol (mint-like, 0.025) (Zhai & Granvogl, 2020) and nine other terpenes. Cyclohexene, 1-ethyl- (0.026), 1-pentanone,1-(2-furanyl)- (0.026), (E)-2-heptenal (green, fatty, 0.026) (Neugebauer et al., 2020) and four other aldehydes and five other esters, 1-heptanol (flowery, soapy, fruity, 0.025) (Polster & Schieberle, 2015) and six other alcohols. One alkane and one furan were also found to be highly correlated with the 'sunflower seeds' aroma of sunflower seed oil, and the detailed list of compounds is shown in Table S4. Among them, 1-pentanol, α-pinene, γ-terpinene, octanal, (±)-linalool, 2-heptenal, (E)- had been sniffed in GC-O. Xu et al. have mentioned in their study on green plum seed oil that alkenal compounds are potentially associated with fatty aroma (Xu, Wang, et al., 2023). In addition to the reconstituted sunflower seed oil solution, two compounds, 3-ethyl-2,5-dimethylpyrazine and α-phellandrene, were added to the reconstituted solution (Yin, Shi, Li, Ma, et al., 2022). Yin et al. reported (±)-terpinen-4-ol in sunflower seed oil for the first time using the pretreatment method of SAFE (Yin, Shi, Li, Ma, et al., 2022). In the present study, this compound was identified using HS-SPME, which is a simpler pretreatment method combined with HRMS. In addition, 9 of the 23 compounds reported for the first time in this study were correlated with this sensory property, and these compounds are marked with yellow color in Fig. 4 (b).

The 'fried instant noodle' aroma of sunflower seed oil was greatly associated with aldehydes, including pentanal (fatty, green, 0.065) (Mallia, Escher, Dubois, Schieberle, & Schlichtherle-Cerny, 2009), 3-methylbutanal (malty, 0.070) (Pollner & Schieberle, 2016), hexanal (green, grassy, 0.051) (Poehlmann & Schieberle, 2013) and (E)-2-hexenal (green apple-like, bitter almond-like, 0.061) (Poehlmann & Schieberle, 2013) (Table S4 and Fig. 4 (c)). In addition to these aldehydes, 2-pentylfuran (vegetable-like. 0.061) (Neugebauer et al., 2020) also demonstrated a positive correlation with the aroma of 'fried instant noodles' from sunflower seed oil. The positive correlation between the above compounds, which were described as having a fruity or grassy aroma in most studies, and the 'fried instant noodle' aroma in the present study might be due to the interaction between the compounds. Among them, four compounds, pentanal, 3-methylbutanal, hexanal and 2-pentylfuran, were all components of the sunflower seed oil reconstituted solution in the study of Yin et al. Among them, pentanal and 2-pentylfuran were also sniffed in this study by SAFE-GC-O (Yin, Shi, Li, Ma, et al., 2022).

From Table S4 and Fig. 4 (d), it can be observed that there were a large number of aroma compounds positively correlated with the 'burnt' aroma of sunflower seed oil, mainly including 5-ethyl-2,3-dimethylpyrazine (0.024), 2-ethyl-5-methyl pyrazine (0.023), 2-ethyl-6-methylpyrazine (0.027) and 8 other pyrazines, 1-(1H-pyrrol-2-yl)1-ethanone (0.026) and 3 other pyrroles, pyridine (0.03), acetamide (0.054), pantolactone (0.091) and 5 other aldehydes and ketones and acetic acid, methyl ester (0.068) and 2 esters, etc. A detailed list of compounds is provided in Table S4. The nitrogenous compounds mentioned above are usually considered to be generated from the Maillard reaction during sunflower seed oil processing, and aldehydes and ketones and esters from the decomposition of oils and fats, with only the difference being whether they were oxidized or not. It could be seen that the 'burnt' aroma in sunflower seed oil might come from the processing procedure. Among them, the two compounds detected by HRMS in this study (2-furanmethanol and pyrrole) were also first detected in sunflower seed oil by LRMS using the SAFE pretreatment by Yin et al (Yin, Shi, Li, Ma, et al., 2022). In addition, 2-furanmethanol, acetyl furan, 2(5H)-furanone, 5-ethyl-2,3-dimethylpyrazine, methylpyrazine, 2,5-dimethylpyrazine, pyrazine, 2,6- dimethyl-, pyrazine, ethyl-, pyrazine, 2,3-dimethyl-, 2-ethyl-5-methyl pyrazine, 2-ethyl-6-methylpyrazine and 1H-pyrrole-2-carboxaldehyde, 1- methyl- have also been reported to be aroma-contributing compounds in sunflower seed oil because they were either smelt in GC-O or were present above their threshold values (Yin, Shi, Li, Ma, et al., 2022). Two esters, acetic acid, methyl ester and propanoic acid, 2-hydroxy-, ethyl ester, were identified for the first time in this study and were correlated with the above sensory properties.

From Fig. 4 (e), it could be seen that the 'sweet' aroma of sunflower seed oil mainly associated alcohols and aldehydes, including 2 aldehydes decyl aldehyde (0.219) and *n*-nonanal (citrus-like, soapy, 0.120) (Neugebauer et al., 2021), 2,3-butanediol (butter-like, 0.078) (Kubícková & Grosch, 1998) and 2,3-butanediol, [S-(R*,R*)]- (0.141) 2 lower alcohols, one alcohol 2-nonanol (fruity, green, 0.179) (Polster & Schieberle, 2015). Additionally, one alkane and one benzene compound (naphthalene, 1-methyl-) were positively correlated with the sweet aroma of sunflower seed oil, and detailed compound information, VIP values and correlation coefficients were shown in Table S4. Among them, naphthalene, 1-methyl- is the first compound reported in sunflower seed oil in this study.

As seen from Fig. 4 (f), the compound base of the 'puffed food' aroma of sunflower seed oil was more similar to that of 'fried instant noodles', including four aldehydes and ketones such as 2(5H)-furanone (0.038), acetoin (0.04), four pyrazines such as pyrazine, ethyl- (0.019), 2-ethyl-5-methyl pyrazine (roasty, nutty, 0.019) (Angeloni et al., 2020), four pyrrole and pyridine compounds such as pyridine (0.024), pyrrole (0.021), in addition to acetic acid (vinegar-like, 0.085) (Neugebauer et al., 2021), two alcohols such as acetone alcohol (0.085), acetamide (0.059), two esters such as acetic acid, methyl ester (0.049), and two furans such as 3(2H)-furanone, dihydro-2-methyl- (0.056). Two of these esters were detected in sunflower seed oil for the first time in this study. Most of the above compounds were nitrogenous and are mainly derived from the processing of sunflower seed oil. Among them, nine compounds, methylpyrazine, 1-(1H-pyrrol-2-yl)1-ethanone, acetyl furan, pyrazine, 2,6-dimethyl-,2-ethyl-5-methyl pyrazine, pyrazine, ethyl-,1H-pyrrole-2-carboxaldehyde, 1-methyl-2-furanmethanol, 2(5H)-furanone, were also detected in GC-O or at levels above their thresholds and were reported to be the compounds with aroma contribution in sunflower seed oil (Yin, Shi, Li, Ma, et al., 2022).

In conclusion, PLSR was successfully screened for compounds that contributed to aroma among a variety of compounds, some of which were identified in earlier research and, more importantly, some of which had not. The results above demonstrate that HS-SPME-GC-HRMS was capable of detecting a wide range of significant volatiles. 15 of the 23 compounds discovered by HRMS for the first time in sunflower seed oil appeared to contribute to the five distinctive aroma characteristics of sunflower seed oil—'roasted sunflower seeds', 'sunflower seeds', 'burnt', 'sweet' and 'puffed food'—that could not have been identified without HRMS. However, the GC-LRMS specifically identified 13 chemicals that were important to the PLSR model. In order to explore the important fragrance components in sunflower seed oil, it is therefore required to combine these two GC–MS techniques.

Conclusions

In this study, we characterized 46 and 105 volatile compounds in seven commercial sunflower seed oils in Chinese market by using GC-Quadrupole-MS and GC-Orbitrap-MS respectively. 96 compounds including 18 alcohols, 12 esters, 7 ketones, 20 terpenoids, 11 pyrazines, 6 aldehydes, 6 furans, 6 benzene ring-containing compounds, 3 sulfides, 2 alkanes, and 5 nitrogen-containing compounds were quantified using HRMS, and 22 compounds including 5 acids, 1 amide, and 16 aldehydes were quantified using LRMS. QDA was used to characterized the aroma profile of seven sunflower seed oil samples, and the results revealed that all the studied samples had 'roasted sunflower seeds,' 'sunflower seeds', and 'burnt' aroma with generally high scores. A few samples were observed to contain 'Fried instant noodles, 'sweet', and 'puffed food' aroma. Positive correlations were observed between 'Roasted sunflower seeds' and 1-octen-3-ol, *n*-heptaldehyde, and dimethyl sulfone. The primary constituents of 'Sunflower seeds' included a variety of aldehydes, ketones, benzenes, terpenoids, and esters. Pentanal, 3-methylbutanal, hexanal, (E)-2-hexenal, and 2-pentylfuran positively correlated with 'fried instant noodles' and 'puffed food' aroma. Pyrazines, pyrroles, pyridines, amides, aldehydes, ketones and esters might collectively contribute to the burnt flavor. On the other hand, the 'sweet' aroma was primarily correlated with alcohols and aldehydes. GC-Orbitrap-MS can detect additional key volatiles that may have been overlooked during GC-Quadrupole-MS detection. This study provided comprehensive information on key aroma components of sunflower seed oils and further supports the quality control and processing modification of sunflower seed oil.

CRediT authorship contribution statement

Jiani Liu: Data curation, Writing – original draft. **Huimin Zhao:** Formal analysis. **Xiaomin Chang:** Data curation. **Xiaolong Li:** Visualization. **Yu Zhang:** Writing – review & editing. **Baoqing Zhu:** Conceptualization, Methodology, Resources. **Xiangyu Wang:** Conceptualization.

Declaration of Competing Interest

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

Data availability

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.2023.100690.

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