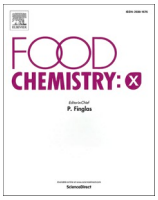
Food Chemistry: X 21 (2024) 101087

Contents lists available at ScienceDirect

Food Chemistry: X

journal homepage: www.sciencedirect.com/journal/food-chemistry-x



Characterisation of flavourous sesame oil obtained from microwaved sesame seed by subcritical propane extraction

Fan Zhang [a], Xue-de Wang [a], Ke Li [a], Wen-ting Yin [a,*], Hua-min Liu [a], Xin-liang Zhu [b], Peng Hu [b]

[a] School of Food Science and Technology, Henan University of Technology, 100 Lianhua Road, Zhengzhou 450001, China
[b] Henan Subcritical Extraction Technology Research Institute Co., Ltd, Anyang 455000, China

ARTICLE INFO

Keywords:
Subcritical extraction
Microwave
Sesame oil
Flavor
Sensory
Bioactive component

ABSTRACT

This study developed a novel and green method to produce fragrant sesame oil using microwaves and subcritical extraction (SBE). Sesame seeds were microwaved at 540 W for 0–9 min before subcritical propane extraction at 40 °C and 0.5 MPa. SBE caused less deformation to the cellular microstructure of sesame cotyledons while dramatically improving oil yield (96.7–97.1 %) compared to screw processing (SP) (53.1–58.6 %). SBE improved extraction rates for γ-tocopherol (381.1–454.9 μg/g) and sesame lignans (917.9–970.4 mg/100 g) in sesame oil compared to SP (360.1–443.8 μg/g and 872.8–916.8 mg/100 g, respectively). Microwaves generated aroma-active heterocyclics and phenolics faster than hot-air roasting in sesame oil with a better sensory profile. SBE had a higher extraction rate for aroma-active terpenes, alcohols, and esters while reducing the concentrations of carcinogenic PAHs and HCAs in sesame oil. The novel combination process of microwaves and subcritical extraction is promising in producing fragrant sesame oil with superior qualities.

Introduction

Sesame (*Sesamum indicum* L.), as an ancient oil crop, is cultivated worldwide, especially abundant in Sudan, India, Tanzania, Myanmar, Nigeria, and China (FAO, 2020). Sesame seeds are good sources of proteins (20–28 %) and oils (48–55 %) (Wei et al., 2022). Sesame oil is recommended by the WHO as a healthy oil for its rich unsaturated fat composition (linoleic acid and oleic acid $\geq$ 80 %) and abundant natural antioxidants including sesamol, sesamin, sesamolin, and tocopherols (Yin, Ma, Li, Wang, Liu, & Shi, 2021). About 50 % of sesame seeds are processed into sesame oil and the global production of sesame oil is approximately one million tons/ year (FAO, 2020).

Originating in ancient China, sesame oil was produced in small family workshops through the traditional aqueous extraction process (TAEP), where roasted sesame seeds are ground by a stone mill before hot water (90–95 °C) is poured into sesame paste to replace sesame oil (Xu, Zhou, & Chen, 2017). The flavour quality of TAEP sesame oil is considered superior to mechanically-extracted sesame oil due to its mild and low-temperature process condition (Yin et al., 2020). However, the TAEP has a low oil extraction rate of between 50 % and 60 % and it is a very time-consuming, labouring, and costly process (Xu et al., 2017). Not to mention that the high-moisture sesame meal as the by-product of TAEP sesame oil is prone to deteriorate and is usually discarded as fertiliser, which is considered a massive waste of good plant protein resources.

Currently, mechanical screw pressing has become the most widely applied industrial practice for the large-scale manufacture of sesame oil (Shi et al., 2018). Compared with the TAEP, the oil extraction rate by screw pressing can be improved remarkably to around 55 %-75 % (Martínez, Bordón, Lallana, Ribotta, & Maestri, 2017). However, the inner chamber temperature of a screw expeller can reach more than 180 °C as a result of heat generation by intense extrusion and friction. High extraction temperature leads to severe protein denaturation, thermal loss of bioactive components, evaporation of volatile flavour compounds, and the generation of undesirable off-flavour and hazardous components in sesame oil (Ji, Liu, Shi, Wang, & Wang, 2019).

The solvent extraction technique using hexane is another common practice in the edible oil industry and its oil extraction rate can reach above 95 % (Lavenburg, Rosentrater, & Jung, 2021). However, to completely remove hexane (boiling point: 69 °C) from the oil phase, high process temperature (280 °C) and vacuum (10^{-4}-10^{-3} MPa) conditions are required (Gharby, 2022). Consequently, most volatile flavour compounds and thermal-sensitive bioactive components will be depleted during the intense solvent removal process (Shi et al., 2018).

* Corresponding author.
E-mail address: yin.wenting@haut.edu.cn (W.-t. Yin).

https://doi.org/10.1016/j.fochx.2023.101087
Received 16 October 2023; Received in revised form 7 December 2023; Accepted 16 December 2023
Available online 21 December 2023

The traditional hexane extraction is apparently not suitable for producing fragrant sesame oil. An alternative greener solvent that can be easily removed from sesame oil at a mild condition while maintaining bioactive compounds and volatile flavour compounds is therefore demanded. Supercritical CO_2 extraction (SPE) has attracted increasing research interest owing to its low process temperature and ability to produce oil with superior quality (Corso et al., 2010). However, the apparent drawbacks of SPE are high process pressure (>25 MPa), expensive equipment, and long extraction time, which hinder its mass application in the edible oil industry (Hrnčič, Cör, Verboten, & Knez, 2018).

Low-temperature subcritical fluid extraction (SBE) as an emerging green technique brings a technological revolution in the edible oil industry. Compressed propane and butane are the most widely used subcritical solvent in extracting oils, which exist as fluids at temperatures above the boiling points and below the critical points, while at pressures below the critical points (Wang, Zhang, Zhang, & Wang, 2020). At their subcritical conditions, these solvents possess reduced viscosity and density, while dramatically improved diffusivity and solubility (Li et al., 2023). Thanks to their low boiling points (propane: −42.1 °C, butane: −0.5 °C), the subcritical fluid solvents can be easily evaporated from the oil phase and recycled in a closed-loop system at low temperatures (<60 °C) and atmospheric pressure (Liu, Yao, Ma, & Wang, 2020; Wang et al., 2021). Low operation pressure (0.3–1 MPa), energy consumption, and production cost make SBE more economical for large-scale industrial applications than supercritical CO_2 extraction (pressure > 25 MPa) (Li et al., 2023). The application of SBE has been evaluated in extracting oils from red pepper seed (Liu et al., 2020; Zhang, Liu, Ma, & Wang, 2019a), flaxseed seed (Wang et al., 2020), Brazil nut (Zanqui et al., 2020), and sunflower seed (Li et al., 2023; Nimet et al., 2011). These SBE oils have shown strong oxidation stability and high bioactive contents. A couple of studies have also investigated the effect of subcritical extraction on the lipid oxidation, fatty acid composition, and bioactive components of sesame oil (Corso et al., 2010; Shi et al., 2018). However, the insightful understanding of the effects of subcritical extraction on the microstructure of sesame seeds, aroma-active composition, and sensory quality of sesame oil is lacking, which hinders the industrial promotion and application of SBE.

The unique flavour of sesame oil is generated mainly via lipid oxidation, caramelisation, and the Maillard reaction during sesame seed roasting (Yin et al., 2021). In the current sesame oil industry, sesame seeds are roasted by hot air (150–220 °C for 20–50 min) which is highly energy-consuming and causes severe protein denaturation due to excessive heat treatment (Ji et al., 2019). Microwave pretreatment of sesame seeds, as a green technique, has been proven faster in generating flavour with a better sensory profile, while maintaining more bioactive compounds (γ-tocopherol) in sesame oil and causing less thermal damage to sesame seeds owing to the highly efficient volumetric heating mechanism (Yin et al., 2023).

This study developed a novel production of sesame oil combining the two green techniques, i.e. microwave pretreatment for flavour generation and subcritical propane low-temperature oil extraction. It was hypothesised that the combination of microwaves and subcritical extraction could improve the oil yield and bioactive components while giving sesame oil characteristic aroma-active composition and a good sensory profile due to low process temperature and pressure. Comprehensive evaluations of SBE sesame oil, including its oxidative stability, bioactive components (tocopherols and sesame lignans), hazardous components (PAHs and HCAs), aroma-active composition, and sensory perception were characterised by advanced analytical techniques.

Materials and methods

Sesame seed pretreatments

Zhuzhi No. 22 white sesame seeds (moisture content: 5 %) were harvested in 2022 from the Zhumadian city of China. Raw, microwaved, or hot-air roasted sesame seeds were processed into different sesame oil samples. Microwave pretreatment: 250 g sesame seeds were microwaved at 540 W, 2450 MHz for 3, 6, or 9 min (stirred once 3 min) in a G90F25CSLV-*C2* microwave oven (Galanz Co., Ltd., China). Hot-air roasting: 250 g sesame seeds were roasted by hot air at 180 °C for 20 min (stirred once 5 min) in a T7-L384D electronic oven (Midea Co., Ltd., China).

Chemicals

The purities of all chemicals in this study were at least 95 %. Anhydrous ethanol, trichloromethane, glacial acetic acid, hydrochloric acid, petroleum ether, ethyl ether, boron trifluoride, sodium hydroxide, sodium thiosulfate, sodium chloride, potassium iodide, soluble corn starch, anhydrous sodium sulfate, nitrogen, helium, and propane were purchased from Kermel Chemical Reagent Co., Ltd (Tianjin, China). Hexane, acetonitrile, dichloromethane, methanol, and isopropanol were obtained from Sigma Aldrich (Sternheim, Germany). The mixed *n*-alkane standard solution (C_5-C_{30}) was purchased from Agilent Technologies (Palo Alto, California, USA). All aroma standard compounds, 4-nonanol, sesamin, sesamolin, and sesamol were obtained from McLean Biochemicals Ltd (Shanghai, China). Norharman, Harman, polycyclic aromatic hydrocarbons (PAHs), α-, β-, δ-, and γ-tocopherols were purchased from Santa Cruz Biotechnology (California, USA).

Subcritical propane extraction of sesame oil

The subcritical propane extraction was carried out based on previous procedures (Wang et al., 2020) with a few modifications. Raw or microwaved sesame seeds were pulverised and passed through a 20 mesh screen before extraction in a CEB-20L subcritical extraction equipment (Henan Subcritical Machinery Equipment Co., Ltd., An Yang, China). 4 Sesame oil samples were obtained including RS (raw seed-SBE), M3S (3 min microwaved seed-SBE), M6S (6 min microwaved seed-SBE), and M9S (9 min microwaved seed-SBE). For each oil sample preparation, a total of 3 kg sesame powder and 12 L fluid propane were loaded into the system and extracted at 40 °C and 0.5 MPa for 150 min. The majority of propane solvent was initially removed at 45 °C and −0.1 MPa. Any solvent residue in the oil was further removed by distillation at 60 °C for 1.5 h. The sesame oil samples were centrifuged at 4000 rpm for 20 min and briefly stored at −20 °C before analysis.

Mechanical screw pressing of sesame oil

Raw, microwaved (540 W, 6 min), or hot-air roasted (180 °C, 20 min) sesame seeds were extracted by a ZYJ-9028 screw expeller (BESTDAY Co., Ltd., Germany) to obtain 3 sesame oil samples, i.e. RP (raw seed-screw pressed), M6P (6 min microwaved seed-screw pressed), and HP (hot-air roasted seed-screw pressed), repressively. Sesame oil samples were centrifuged at 4000 rpm for 20 min and briefly stored at −20 °C before analysis. The hot-air roasting condition (180 °C for 20 min) was selected because it was comparable to the commercial practice for sesame oil production (Yin et al., 2021). The microwave condition (540 W, 6 min) was selected because it was the optimal microwave condition for producing flavourous sesame oil according to our previous study (Yin et al., 2023).

Microstructure observation of sesame meal after extraction

The sesame meal samples after different oil extraction processes were observed by an LSM710 inverted confocal laser scanning microscope (CLSM) (CarlZeiss AG, Germany). A sesame meal sample of 0.2 g was stained with 100 μL Nile red, fluorescein isothiocyanate (FITC), and Calcofluor White to label lipids (red fluorescence), proteins (green fluorescence), and cellular walls (blue fluorescence), respectively (Jin

et al., 2022; Yilmaz, Kodama, & Numata, 2020). The excitation wavelengths were 405 nm, 488 nm, and 633 nm. The emission wavelengths for CLSM observation were 410–480 nm, 500–540 nm, and 570–620 nm.

Determination of oil yield

The oil contents of sesame seeds before and after extraction were determined according to ISO 659:2009. The oil yield of an extraction process was calculated based on equation (1) according to Xu et al. (2017).

$$Oil\ yield(\%) = \left(1 - \frac{C_1}{C_0}\right) \times 100 \quad (1)$$

where C_1 represents the oil residue in sesame meal after extraction (g/100 g), and C_0 epresents the oil content of sesame seed before extraction (g/100 g).

Determination of lipid oxidation and colour parametres

Sesame oil samples were analysed for acid value (AV), peroxide value (POV), and colour indexes according to the procedures of ISO 660:2020, ISO 3960:2017, and ISO 27608:2010, respectively. The oxidation stability of sesame oils (5 g) was indicated by oxidation induction time (OIT) which was measured at 110 °C and 20 L/h airflow in the Rancimat 743 apparatus (Metrohm Co. Ltd., Switzerland) according to an established method (Zhang, Liu, Ma, & Wang, 2019b).

Determination of tocopherols in sesame oil

The tocopherol compositions of sesame oil were measured by the E2695 HPLC with the 2475 fluorescence detector (FLD) referring to the method of Yin et al. (2023). The 0.5 g sesame oil sample was mixed with 10 mL hexane and went through a 0.22 μm organic membrane before being injected into HPLC. The HPLC system was equipped with the XBridege BEH Amide column (250 mm × 4.6 mm, 5 μm, Waters, CA, U.S.A.) and set at 40 °C for analysis. The sample injection volume was 10 μL. The mixture of hexane and isopropanol (99:1, v/v) was used as the mobile phase with a flow rate of 1 mL/min. The excitation/emission wavelengths were set at 295 nm/330 nm.

Analysis of lignans in sesame oil

Sesame lignans in sesame oil samples were measured based on the published method (Yin et al., 2023). The mixture of sesame oil sample (0.5 g) and methanol (9 mL) was centrifuged at 4000 rpm for 10 min. The supernatant was filtered by an organic filter membrane of 0.22 μm and fixed to 10 mL. A 10 μL sample was then injected into the E2695 HPLC system with the 2489 UV/visible detector (Waters Co., Ltd., USA) for analysis. The Waters Sun Fire C_{18} column (250 mm × 4.6 mm, 5 μm) was set at 30 °C. The mobile phase was the mixture of methanol and water (7:3, v/v) and the flow rate was controlled at 0.8 mL/min. The wavelength of the UV/Vis detector is 287 nm.

Determination of PAHs in sesame oil

PAHs were extracted from the oil using a 505,048 LC-Si solid-phase extraction (SPE) tube (Agela Tech Co., Ltd., China) and analysed by the E2695 HPLC with the 2475 FLD (Waters) according to the method of Yin et al. (2023). The Waters PAH C_{18} column (250 mm × 4.6 mm × 5 μm) was used at 30 °C. The injected sample volume was 20 μL. The mobile phase was mixed with acetonitrile and water at a flow rate of 1.0 mL/min. The HPLC gradient elution procedure was as follows: 0 to 8 min, 60 % acetonitrile; 8 to 18 min, acetonitrile increased from 60 % to 100 %; 18 to 28 min, 100 % acetonitrile; 28 to 32 min, acetonitrile decreased from 100 % to 60 %.

Determination of HCAs in sesame oil

Harman and Norharman in sesame oil were extracted using the 505048 LC-Si SPE tube (Agela Tech Co., Ltd., China) and measured by the Waters E2695 HPLC-2475 FLD according to the method of Zhang et al. (2020). The mobile phase consisted of ammonium formate (10 mmol/L, pH = 6.8, solvent A) and acetonitrile (solvent B). The HPLC gradient elution process was as follows: 0 to 0.2 min, solvent B (0 %-10 %); 0.2 to 1 min, solvent B (10 %-30 %); 1 to 10 min, solvent B (30 %-60 %); 10 to 15 min, solvent B (60 %-90 %). A Waters SunFire C_{18} (250 mm x 4.6 mm, 5 μm) was used at 30 °C and the injection volume of the sample was 10 μL. The wavelengths of excitation and emission in the FLD were selected as 300 nm and 440 nm, respectively.

Volatile extraction by solvent-assisted flavour evaporation (SAFE)

The extraction of volatile compounds from sesame oil samples was performed in a solvent-assisted flavor evaporation (SAFE) apparatus referring to the method of Yin et al. (2021). A total of 100 g sesame oil sample, 225 mL dichloromethane, and 240 μL 4-nonanol (1 mg/mL, internal standard) were mixed at 25 °C for 10 h before being extracted at 50 °C in the SAFE apparatus under 2.0×10^{-3} Pa. The extract was dried with anhydrous sodium sulfate before being concentrated to 5 mL using a Vigreux column (50 cm × ϕ2 cm) at 60 °C and further to 1 mL by nitrogen stream (≥99.99 %). The concentrates went through a 0.22 μm organic microfiltration membrane before being injected into GC-O-MS.

Analysis of aroma-active compounds in sesame oil by GC-O-MS

The volatile extract was analysed by a 7890B gas chromatography (Agilent Technologies, USA) equipped with a 5977B electron ionization mass spectrometer (Agilent) and an ODP-3 olfactometer (Gerstel Inc, Germany), according to the method of Yin, Maradza, Xu, Ma, Shi, & Zhao (2022). The volatiles were separated on an HP-5MS capillary column (30 m × 0.25 mm, 0.25 μm) with helium as the carrier gas (purity of 99.999 %). The temperature of the GC column was initially set at 40 °C (held for 5 min), climbed to 230 °C by 3 °C/min (held for 5 min), and increased up to 230 °C by 10 °C/min. The GC effluent was split equally between the ODP and the MSD. The MSD was set as follows: electron bombardment ion source at 230 °C, ionisation energy of 70 eV, full scan mode, the scan range of 33–350 *m/z*, and the quadrupole at 150 °C. The identifications of odourants were verified by their odour description (O) from three trained panelists (2 females and 1 male) perceived at the ODP, their mass spectra (MS) compared to the NIST17 library, retention indexes (RI), and a comparison to their standard compounds (STD). The relative concentrations of volatile compounds were calculated by the internal standard method (Yin, Shi, Li, Ma, et al., 2022; Yin, Shi, Li, Wang, et al., 2022).

Sensory evaluation of sesame oil

The sensory evaluation of sesame oils was conducted referring to the T/CCOA 29:2020 standard of the Chinese Cereals and Oils Association. A panel of 12 trained participants (6 females and 6 males) evaluated the aroma quality of all samples. All participants were clearly informed about the details of this study and their consent was obtained prior to the study. Appropriate protocols for protecting the rights and privacy of all participants were utilised during the execution of the study. Each sesame oil sample (8 mL) was placed into a 75 mL brown tasting glass labeled with a random two-digit number and evaluated at 35 ± 1 °C. Each participant rated the characteristic aromas of woody, green, raw sesame seed, roasted sesame seed, nutty, caramel, burnt, and meaty on a 10 cm continuous line scale. They were asked to use green apples and warm water to cleanse their palates between samples. Sensory data were

obtained by the Compusense Cloud software (Compusense Inc., Canada) in individual booths of the Sensory Evaluation Centre of Henan University of Technology conforming to ISO: 8589:2007.

Statistical analysis

All experiments were conducted in triplicate and data are presented as mean ± standard deviation. Data analysis was performed using IBM SPSS Statistics 20.0 software (SPSS Inc., Chicago, USA). Analysis of variance (ANOVA) and Duncan's multiple comparisons tests were conducted to identify the presence of any significant difference ($p < 0.05$). All graphs were drawn by the Origin 2023 software (OriginLab Corporation, America), and chemical structures were drawn by the ChemDrew 22.0.0 software (CambridgeSoft Corporation, America).

Results and discussion

Cellular microstructure of sesame meal after extraction and oil yields of different processes

The CLSM images (Fig. 1) of sesame meals after oil extraction demonstrate the micro-distribution of cellular walls, proteins, and lipids in the sesame cotyledon. The green, light-blue, and red areas indicate the protein bodies, structures of cell walls, and oil bodies, respectively. A considerable amount of lipids remained in the sesame meals after screw pressing (RP, M6P, and HP), which was correlated with low extraction rates (53.1 %-58.6 %). The observed aggregation of small and scattered oil bodies (RP) to form large oil droplets (M6P and HP) and the deformation of cellular structures by friction and pressure inside the screw barrel may facilitate the mechanical extraction of sesame oil (Hu et al., 2018). However, the external mechanical forces may not be sufficient to break all the covenant bonds between the solid cellular matrix (e.g. protein bodies) and the oil molecules, which restricts the release of oil from the seed (Hu et al., 2018).

In comparison, there was scarcely any oil remaining in the sesame meals after subcritical extraction (RS, M3S, M6S, M9S), which was in agreement with their high oil extraction rates (96.7–97.1 %) as shown in Fig. 2**A**. In the raw sesame seeds after subcritical extraction (RS), cellular walls were ordered and intact and protein bodies were located inside the cellular structures, whilst more fractures of cellular walls were observed and many protein bodies were scattered outside the cellular structure in the screw-pressed sesame meals (RP, M6P, and HP). This suggests that subcritical propane extraction could extract most lipids from sesame seeds (oil yield ≥ 96.7 %) without causing apparent physical destruction of the cellular wall structures. The dramatically increased diffusivity, density, and permeability of the fluid propane solvent under its subcritical condition (40 °C and 0.5 MPa) are responsible for the significantly improved solubility of lipophilic compounds distributed in the sesame cellular structures (Xu et al., 2017; Zhang et al., 2019a). The rapid penetration and mass transfer of propane through the microchannels of the cellular walls and membranes facilitates the highly efficient extraction of lipophilic compounds (Li et al., 2023). Meanwhile, the subcritical extraction pressure (0.5 MPa) did not exceed the cellular deformation limit so the sesame seed cellular structures were still complete and ordered after extraction (Huang, Hsu, Yang, & Wang, 2013).

With the extension of microwave time, greater disruption of cell walls was observed. After 9 min microwave pretreatment (M9P) or 20 min hot-air roasting (HP), hardly any intact cellular wall was observed, and proteins and lipids were completely released from the cellular structures. Both the microwave pretreatment (M6P = 57.6 %) and hot-air roasting (HP = 58.6 %) of sesame seeds improved the oil yield compared to the cold-pressed sesame oil obtained from raw seeds (RP = 53.1 %) ($p < 0.05$). This is consistent with a few published papers (Wang et al., 2020; Yin, Shi, Li, Ma, et al., 2022; Yin et al., 2023). The increased disruption of cellular walls by microwaves or hot-air roasting may have facilitated the release of the oil droplets. The oil yields did not differ between the four subcritical extracted samples with different microwave parametres (RS, M3S, M6S, and M9S), suggesting that subcritical extraction played a more decisive role in improving oil yield than microwave pretreatment.

Lipid oxidation in differently processed sesame oils

Acid value (AV) is the measure of the free fatty acid content in oil and reflects the degree of hydrolysis and rancidity (Zhang, Li, Lu, Sun, & Wang, 2021). As shown in Fig. 2**B**, the AV of the cold-pressed RP oil was 0.55 mg KOH/g, while the AV of the subcritical-extracted RS oil increased to 0.74 mg KOH/g ($p < 0.05$). A previous study also reported that the AV of subcritical-extracted tiger nut oil was higher than the AV of cold-pressed tiger nut oil (Guo et al., 2021). This was probably because subcritical extraction had a higher extraction rate for free fatty acids in oils. A mild microwave pretreatment (M3S, 3 min) deceased the AV of the sesame oil (0.47 mg KOH/g) slightly compared with the cold-pressed sesame oil (RP) ($p < 0.05$), which could be attributed to the thermal degradation and oxidation of the naturally present free fatty

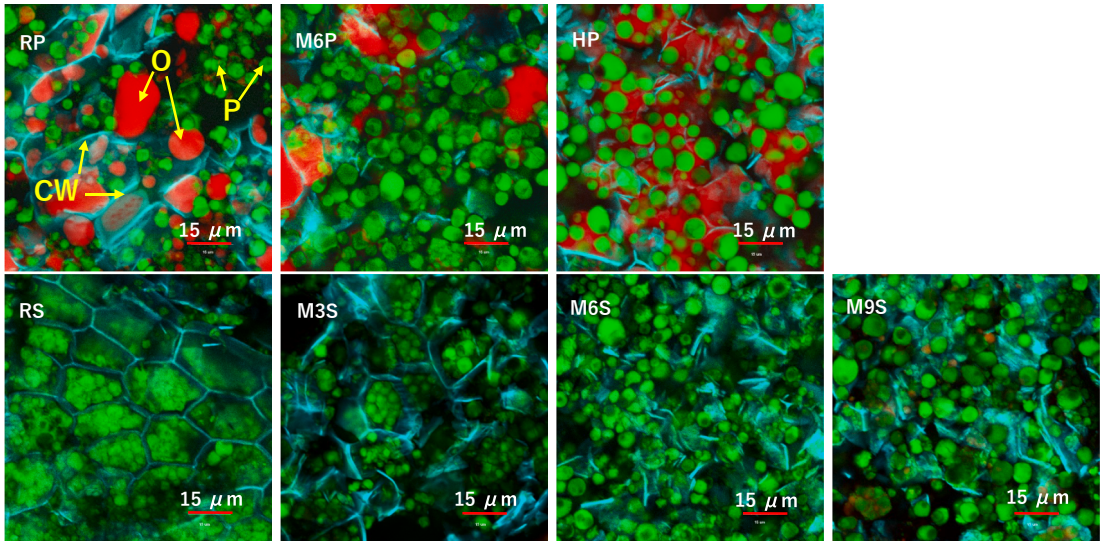


Fig. 1. Confocal laser scanning microscopy of sesame meals produced through different processes.

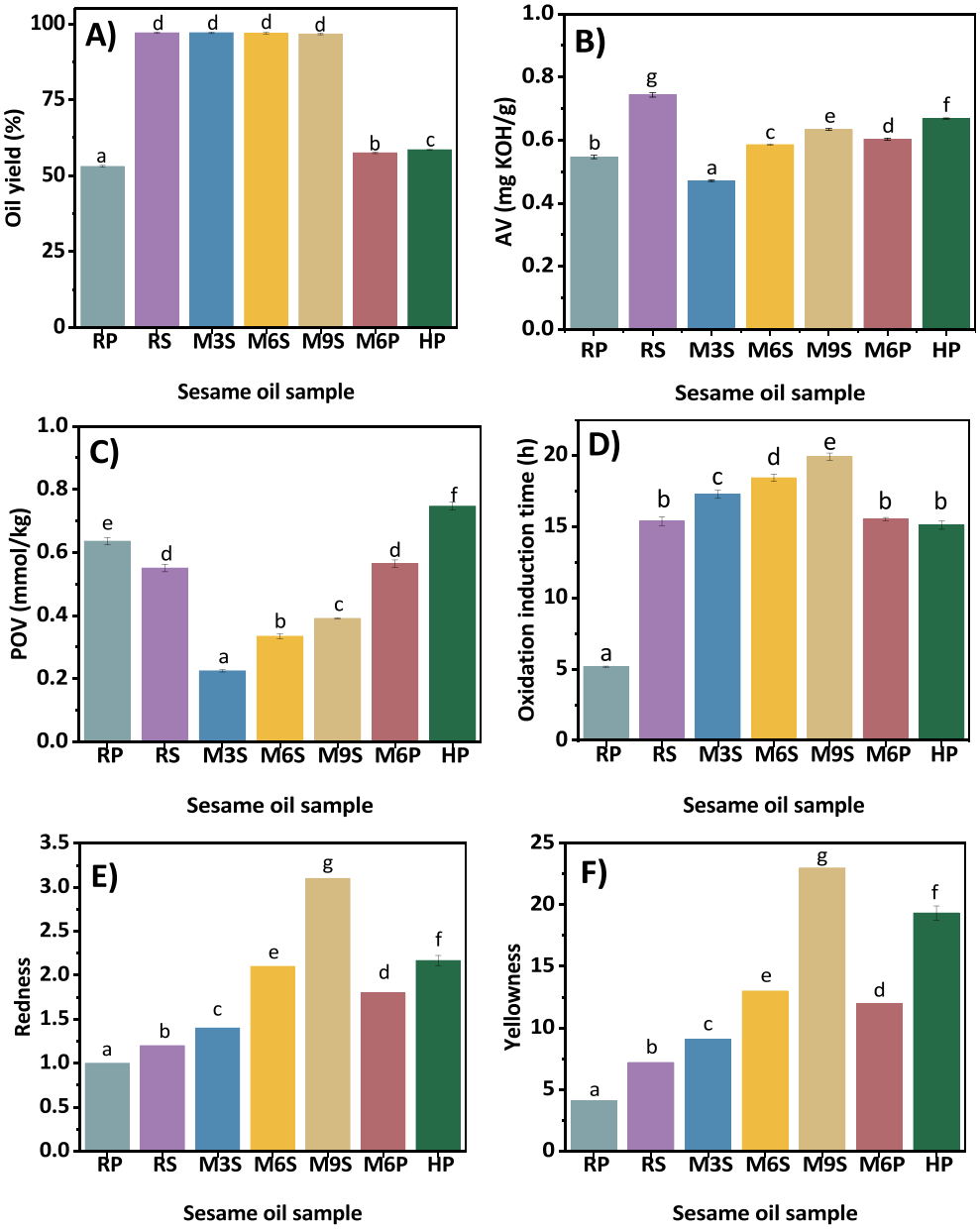


Fig. 2. The oil yield (A), acid value (B), peroxide value (C), oxidation induction time (D), redness index (E), and yellowness index (F) of sesame oil samples produced through different processes.

acids in sesame oil upon the mild thermal effect of microwaves (Qin, Han, Wang, Liu, Zheng, & Wang, 2020). With the increase of microwave time from 3 min to 9 min, the AVs of subcritical-extracted sesame oils (M3S, M6S, M9S) increased slightly ($p < 0.05$). It was probably due to the hydrolysis of triglycerides to free fatty acids upon the accumulated thermal effect of microwaves (Ji et al., 2019).

The peroxide values (POVs) indicate the levels of peroxides and hydroperoxides in sesame oils (Zhang et al., 2021) (Fig. 2**C**). The POV of the RS oil (0.55 mmol/kg) was lower than that of the RP oil (0.63 mmol/kg) ($p < 0.05$). Gao et al. (2022) also reported that the POVs of walnut oils obtained by subcritical extraction were significantly lower than screw pressing. It was possibly due to the low processing temperature and anaerobic environment in the subcritical extraction equipment which inhibited lipid oxidation. When moderate microwaves (3 min and 6 min) were applied to sesame seeds, the POVs of the subcritical-extracted sesame oils (0.23–0.33 mmol/kg) were reduced significantly compared to the RS oil ($p < 0.05$). It was likely because the naturally present peroxides and hydroperoxides in sesame seeds rapidly decomposed to aldehydes and ketones upon the initial mild to moderate microwave treatment (Ahmed et al., 2021). The HP oil (0.75 mmol/kg) had the highest POVs among all samples, which may be due to the excessive heat generated by hot air, decomposing fatty acids to form more peroxides and hydroperoxides (Yin et al., 2023).

The oxidative stability, measured as the oxidation induction time (OIT) by Rancimat, is an important quality indicator for oils, mainly determined by the amounts of antioxidants and fatty acids in oil (Zhang et al., 2019b). As shown in Fig. 2**D**, compared with the OIT of the RP oil (5.16 h), the OITs of M6P oil (15.37 h) and HP oil (15.09 h) improved ($p < 0.05$). The generation of anti-oxidative Maillard reaction products (MRPs) upon the thermal effect of microwaves or hot-air roasting may be responsible for the increased OIT (Qin et al., 2020). The OITs of all subcritical extracted sesame oils were significantly improved (15.5 h-19.9 h) than any of the screw-pressed sesame oil (RP, M6P, or HP) ($p < 0.05$). Subcritical extraction may have higher extraction rates for the

natural and synergistic antioxidants including tocopherols, sesame lignans, and the MRPs in sesame oils than screw pressing, hence increasing the oil stability.

Colour indexes in differently processed sesame oils

The colour of the RP oil was the lightest as it had the lowest red and yellow indexes **(**Fig. 2**E & F)**. The red and yellow indexes of the RS oil increased compared with the RP oil ($p < 0.05$). After the same microwave pretreatment (540 W, 6 min), the subcritical-extracted oil (M6S) had higher red and yellow indexes than the screw-pressed oil (M6P). These results suggested that the subcritical propane was more efficient in extracting sesame oil pigments than the mechanical screw pressing. Previous studies reported that subcritical propane improved the concentrations of phytochemicals and pigments in oils even more than supercritical CO_2 extraction (Li et al., 2023).

The red and yellow colour indexes increased continuously with the increasing microwave pretreatment time ($p < 0.05$). It is probably because of the accumulation of browning pigments generated via the Maillard reaction and caramelisation during microwaves (Yin et al., 2023). In addition, the M9S oil had higher red and yellow indexes than the HP oil ($p < 0.05$), suggesting that microwave pretreatment was more time-efficient in generating oil pigments than hot-air roasting.

Bioactive compounds in differently processed sesame oils

Tocopherols

γ-Tocopherol was the only tocopherol detected in sesame oil samples (Fig. 3**A**). Compared with the RP oil (443.8 μg/g), the RS oil had a higher content of γ-tocopherol (455.0 μg/g) ($p < 0.05$). Meanwhile, more γ-tocopherol was detected in the M6S oil (402.9 μg/g) than in the M6P oil (394.1 μg/g) ($p < 0.05$). It suggests that subcritical propane extraction is more efficient in extracting γ-tocopherol in sesame oil compared to screw pressing.

The content of γ-tocopherol in the subcritical-extracted sesame oils decreased with the increasing microwave pretreatment time ($p < 0.05$). This was probably due to the thermal degradation of γ-tocopherol by microwaves (Ji et al., 2019). All microwave-pretreated sesame oils contained more γ-tocopherol than the HP oil (360.1 μg/g) ($p < 0.05$). This is consistent with other studies showing that microwave pretreatment causes less thermal damage and increases the tocopherol content in oils than traditional hot-air roasting (Gao et al., 2022; Yin, Shi, Li, Ma, et al., 2022).

Sesame lignans

Sesamin and sesamolin are two naturally occurring sesame lignans (Wei et al., 2022). Both the concentrations of sesamin **(**Fig. 3**B)** and sesamolin **(**Fig. 3**C)** in the RS oil were higher than in the RP oil ($p < 0.05$), suggesting that the subcritical propane extraction is more efficient in extracting sesamin and sesamolin in sesame oil than screw pressing. With the increasing microwave pretreatment time, the concentrations of sesamin and sesamolin in the subcritical-extracted sesame oils gradually decreased ($p < 0.05$). It was likely due to the thermal degradation of sesamin and sesamolin to form sesamol during the roasting of sesame seeds. The antioxidant activity of sesamol is reported stronger than sesamin and sesamolin (Wei et al., 2022). Sesamol was not detected in the RP, RS, and M3S oils, but appeared in the M6S, M9S, M6P, and HP oils **(**Fig. 3**D)**. The M6S oil (1.01 mg/100 g) contained more sesamol than the M6P oil (0.50 mg/100 g), suggesting that the subcritical propane extraction is more efficient in extracting sesamol in sesame oil than screw pressing. In addition, the significantly higher sesamol content in the M9S oil (1.49 mg/100 g) than in the HP oil (1.23 mg/100 g) indicated that microwave was more efficient in generating sesamol with a shorter process time than hot air roasting.

Hazardous components in differently processed sesame oils

PAH4 in oils

PAH4 including benzo[*a*]anthracene, chrysene, benzo[*b*]fluoranthene, and benzo[*a*]pyrene have been identified as having carcinogenic toxicity by the European Food Safety Authority (Ji, Jiang, Zhang, Hou, & Sun, 2022). They are produced by incomplete thermal degradation of carbohydrates and fats at high temperatures and are often found as hazardous components in oilseeds (Potočnik & Košir, 2016). No PAH4 was detected in the RP and RS oils which were extracted from raw sesame seeds **(**Fig. 3**E).** Only two of PAH4 including benzo[*a*]anthracene and chrysene were detected in the sesame oils extracted from microwaved or hot-air roasted sesame seeds. In subcritical-extracted sesame oils, the sum concentration of PAH4 in subcritical-extracted sesame oils continuously increased from 0.71 ng/g to 3.61 ng/g with the increasing microwave time ($p < 0.05$). The accumulation of PAH4 in oils is believed a result of the thermal effect of microwaves (Yin et al., 2023). The

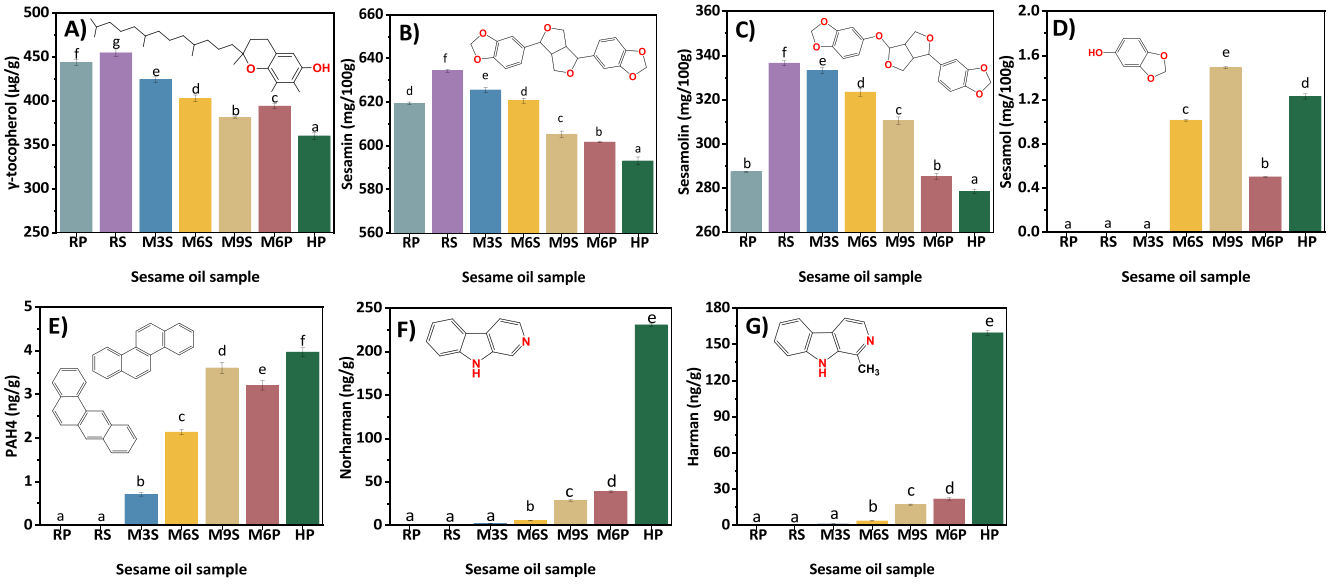


Fig. 3. Contents of γ-tocopherol (A), sesamin (B), sesamolin (C), sesamol (D), PAH4 (E), Norhaman (F), and Harman (G) in sesame oil samples produced through different processes.

Table 1
Relative concentrations of aroma-active compounds in sesame oils extracted from differently processed sesame seeds.

RI [A]	Aroma-active compounds	Identification Method [B]	Odour Description [C]	Relative concentration (μg/kg) in sesame oil sample						
				RP	RS	M3S	M6S	M9S	M6P	HP
934	α-Thujene	MS	green	1 ± 0^{a}	192 ± 14^{c}	67 ± 3^{b}	72 ± 4^{b}	74 ± 4^{b}	N.D.	N.D.
944	(1R)-(+)-α-Pinene	MS/O/STD	minty	N.D.	579 ± 76^{b}	622 ± 19^{c}	188 ± 10^{a}	N.D.	N.D.	N.D.
983	Sabinene	MS/RI	woody	N.D.	1,599 ± 65^{d}	481 ± 22^{a}	675 ± 39^{c}	527 ± 25^{b}	N.D.	N.D.
985	(1S)-1-β-Pinene	MS/O/STD	woody, green	N.D.	418 ± 21^{c}	301 ± 7^{b}	N.D.	277 ± 15^{a}	N.D.	N.D.
1002	β-Myrcene	MS/O/RI/STD	fatty	N.D.	510 ± 65^{d}	374 ± 12^{a}	434 ± 15^{c}	391 ± 11^{a}	N.D.	N.D.
1016	α-Phellandrene	MS/O/RI/STD	sweet	N.D.	225 ± 61^{b}	185 ± 5^{a}	N.D.	N.D.	N.D.	N.D.
1026	α-Terpinene	MS/O/STD	sweet	N.D.	36 ± 10^{c}	17 ± 2^{a}	18 ± 2^{a}	22 ± 2^{b}	N.D.	N.D.
1051	(E)-β-Ocimene	MS	flower	N.D.	67 ± 10^{d}	61 ± 5^{c}	53 ± 6^{b}	39 ± 6^{a}	N.D.	N.D.
1097	Terpinolene	MS/O	sweet, fatty	N.D.	61 ± 17^{c}	49 ± 3^{b}	28 ± 2^{a}	N.D.	N.D.	N.D.
1389	Copaene	MS/O/STD	green, woody	4 ± 1^{a}	70 ± 19^{d}	35 ± 3^{c}	15 ± 1^{b}	37 ± 3^{c}	N.D.	5 ± 1^{a}
	Total Terpenes			**5 ± 1^{a}**	**3,755 ± 370^{e}**	**2,192 ± 71^{d}**	**1,483 ± 49^{c}**	**1,367 ± 51^{b}**	**N.D.**	**5 ± 1^{a}**
707	3-Penten-2-ol	MS/O	green	N.D.	N.D.	N.D.	N.D.	N.D.	25 ± 2^{a}	N.D.
809	(S, S)-2,3-Butanediol	MS	milk	N.D.	N.D.	196 ± 42^{b}	97 ± 11^{a}	N.D.	N.D.	N.D.
815	2,3-Butanediol	MS/O	fatty, buttery	N.D.	221 ± 12^{a}	N.D.	N.D.	N.D.	N.D.	N.D.
884	1-Hexanol	MS/O	green	7 ± 2^{a}	399 ± 22^{c}	442 ± 12^{d}	74 ± 5^{b}	N.D.	N.D.	N.D.
994	1-Octen-3-ol	MS/O/RI/STD	mushroom	N.D.	62 ± 3^{b}	77 ± 9^{c}	104 ± 10^{d}	160 ± 7^{e}	66 ± 6^{b}	53 ± 10^{a}
1083	4-Thujanol	MS/O	green, minty	N.D.	26 ± 8^{b}	26 ± 3^{b}	20 ± 1^{a}	N.D.	N.D.	N.D.
1186	1-Nonanol	MS/O/STD	citrus, dusty	N.D.	64 ± 7^{b}	76 ± 4^{c}	17 ± 2^{a}	N.D.	N.D.	N.D.
1193	L-Terpinen-4-ol	MS	caramel, sweet	N.D.	42 ± 11^{c}	24 ± 2^{b}	19 ± 5^{a}	N.D.	N.D.	N.D.
1232	γ-Terpineol	MS/O	flower, peanut shell	N.D.	240 ± 64^{c}	110 ± 8^{ab}	126 ± 6^{b}	84 ± 6^{a}	N.D.	N.D.
	Total Alcohols			**7 ± 2^{a}**	**1,054 ± 108 ^{g}**	**953 ± 111^{f}**	**465 ± 29^{e}**	**243 ± 8^{d}**	**91 ± 7^{c}**	**53 ± 10^{b}**
<700	Acetic acid ethenyl ester	MS/O/STD	fatty	91 ± 11^{a}	N.D.	N.D.	N.D.	N.D.	170 ± 25^{b}	215 ± 26^{c}
757	2-Butyl acetate	MS/O/STD	fatty, fruit	19 ± 4^{a}	21 ± 5^{a}	23 ± 3^{a}	19 ± 3^{a}	20 ± 2^{a}	25 ± 2^{a}	20 ± 8^{a}
1143	Methyl nicotinate	MS	sulfur	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	60 ± 13^{a}
1271	Linalyl acetate	MS	milk, nutty, fatty	N.D.	929 ± 105^{d}	706 ± 34^{c}	648 ± 14^{b}	457 ± 24^{a}	N.D.	N.D.
	Total Esters			**110 ± 10^{a}**	**950 ± 108 ^{g}**	**729 ± 33^{f}**	**667 ± 14^{e}**	**476 ± 28^{d}**	**194 ± 18^{b}**	**295 ± 18^{c}**
<700	3-Methyl-2-butanone	MS/O	peanut shell, woody	21 ± 2^{a}	42 ± 11^{a}	N.D.	25 ± 3^{a}	50 ± 4^{a}	N.D.	469 ± 26^{b}
<700	3-Penten-2-one	MS/O	peanut, musty	N.D.	4 ± 1^{a}	N.D.	N.D.	N.D.	3 ± 0^{a}	N.D.
<700	2-Butanone	MS/O/RI	yogurt	N.D.	33 ± 4^{d}	26 ± 2^{c}	29 ± 3 ^{cd}	20 ± 2^{b}	14 ± 1^{a}	26 ± 2^{c}
707	Acetoin	MS/O/STD	milk	5 ± 2^{a}	N.D.	35 ± 4^{c}	68 ± 4^{e}	132 ± 6^{f}	48 ± 3^{d}	24 ± 5^{b}
714	2,3-Pentanedione	MS/O/RI/STD	butter	N.D.	N.D.	N.D.	14 ± 1^{a}	64 ± 4^{b}	53 ± 3^{ab}	263 ± 20^{c}
782	Acetylacetone	MS	rancidity	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	9 ± 2^{a}
788	Cyclopentanone	MS/O/RI/STD	minty	N.D.	N.D.	N.D.	N.D.	N.D.	8 ± 1^{a}	7 ± 2^{a}
878	1-(Acetyloxy)-2-propanone	MS/STD	rancidity	N.D.	N.D.	N.D.	N.D.	225 ± 10^{c}	45 ± 7^{a}	181 ± 21^{b}
893	4-Cyclopentene-1,3-dione	MS	earthy	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	65 ± 14^{a}
895	2-Heptanone	MS/O/RI/STD	nutty, fruit	N.D.	N.D.	N.D.	N.D.	N.D.	11 ± 1^{a}	21 ± 4^{b}
992	2-Octanone	MS/O/RI/STD	fruit, apple	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	17 ± 4^{a}
1075	Acetophenone	MS/O/RI/STD	sesame oil	N.D.	N.D.	N.D.	N.D.	166 ± 16^{b}	35 ± 3^{a}	37 ± 7^{a}
1161	(+)-2-Bornanone	MS/O	green	N.D.	38 ± 11^{a}	131 ± 3^{d}	60 ± 2^{c}	47 ± 3^{b}	N.D.	N.D.
1262	(-)-Carvone	MS/O	green, minty	N.D.	214 ± 24^{a}	24 ± 3^{a}	N.D.	N.D.	N.D.	N.D.
	Total Ketones			**26 ± 3^{a}**	**331 ± 43^{d}**	**216 ± 13^{c}**	**196 ± 13^{b}**	**703 ± 72^{e}**	**217 ± 29^{c}**	**1,021 ± 168^{f}**
<700	2-Methyl-propanal	MS/STD	rancidity	N.D.	N.D.	N.D.	N.D.	21 ± 5^{a}	13 ± 1^{a}	15 ± 6^{a}
<700	2-Methyl-butanal	MS/O/STD	green, peanut	N.D.	N.D.	N.D.	76 ± 5^{a}	244 ± 10^{b}	506 ± 33^{c}	634 ± 32^{d}
<700	3-Methyl-butanal	MS/O/STD	fermented, sour	N.D.	10 ± 2^{a}	12 ± 3^{a}	59 ± 6^{b}	104 ± 6^{c}	271 ± 22^{d}	261 ± 23^{d}
813	Hexanal	MS/O/RI/STD	green	25 ± 2^{b}	N.D.	11 ± 1^{a}	100 ± 3^{d}	126 ± 4^{e}	53 ± 4^{c}	25 ± 6^{b}
973	Benzaldehyde	MS/O/RI/STD	caramel, nutty	2 ± 1^{a}	13 ± 4^{b}	37 ± 5^{c}	71 ± 6^{e}	160 ± 6^{f}	45 ± 3^{d}	13 ± 1^{b}
1174	(E)-2-Nonenal	MS/O/STD	spicy, burnt	N.D.	12 ± 3^{b}	8 ± 2^{a}	31 ± 3^{c}	41 ± 4^{d}	N.D.	N.D.
1274	3-Methoxy-benzaldehyde	MS	fatty, milky, peanut	N.D.	N.D.	N.D.	67 ± 3^{b}	58 ± 6^{a}	N.D.	N.D.

(*continued on next page*)

Table 1 (*continued*)

RI [A]	Aroma-active compounds	Identification Method [B]	Odour Description [C]	Relative concentration (μg/kg) in sesame oil sample						
				RP	RS	M3S	M6S	M9S	M6P	HP
	Total Aldehydes			**27 ± 2^{a}**	**34 ± 8^{b}**	**67 ± 8^{c}**	**405 ± 15^{d}**	**753 ± 50^{e}**	**889 ± 62^{f}**	**959 ± 49 ^{g}**
740	Pyrazine	MS/O/RI/STD	roasted, burnt, popcorn	N.D.	N.D.	2 ± 1^{a}	6 ± 1^{a}	95 ± 10^{b}	15 ± 2^{a}	146 ± 28^{c}
834	Methyl-pyrazine	MS/O/RI/STD	roasted	N.D.	N.D.	N.D.	216 ± 12^{a}	1,989 ± 44^{c}	395 ± 46^{b}	2,060 ± 132^{c}
923	2,5-Dimethyl-pyrazine	MS/O/RI/STD	nutty, salty	N.D.	N.D.	9 ± 2^{a}	838 ± 56^{b}	3,048 ± 30^{d}	872 ± 52^{b}	2,206 ± 128^{c}
928	Ethyl-pyrazine	MS/O/RI/STD	salty, roasted	N.D.	N.D.	N.D.	62 ± 5^{a}	387 ± 7^{b}	61 ± 2^{a}	384 ± 75^{b}
931	2,3-Dimethyl-pyrazine	MS/O/RI/STD	salty, roasted	N.D.	N.D.	N.D.	12 ± 2^{a}	268 ± 18^{d}	44 ± 5^{b}	205 ± 48^{c}
936	Ethenyl-pyrazine	MS/O	roasted peanut	N.D.	N.D.	N.D.	N.D.	N.D.	5 ± 1^{a}	48 ± 10^{b}
1014	2-Ethyl-5-methyl-pyrazine	MS/O/RI/STD	burnt, roasted, salty	N.D.	N.D.	N.D.	194 ± 23^{a}	820 ± 28^{c}	198 ± 12^{a}	378 ± 82^{b}
1014	2-Ethyl-6-methyl-pyrazine	MS/O/RI	burnt, roasted	N.D.	N.D.	N.D.	118 ± 12^{b}	411 ± 7^{d}	73 ± 5^{a}	253 ± 53^{c}
1015	Trimethyl-pyrazine	MS/O/RI/STD	burnt, nutty	N.D.	N.D.	N.D.	347 ± 25^{a}	1,213 ± 31^{c}	311 ± 23^{a}	655 ± 100^{b}
1029	Acetylpyrazine	MS/O/RI/STD	chocolate, popcorn	N.D.	N.D.	N.D.	N.D.	167 ± 10^{c}	20 ± 1^{a}	107 ± 19^{b}
1033	2-Ethenyl-6-methyl-pyrazine	MS/O	roasted	N.D.	N.D.	N.D.	2 ± 1^{a}	49 ± 13^{b}	7 ± 1^{a}	N.D.
1046	2-Methyl-5-(1-methylethyl)-pyrazine	MS/RI	roasted, peanuts	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	3 ± 1^{a}
1092	2,6-Diethyl-pyrazine	MS/O/RI	roasted	N.D.	N.D.	N.D.	N.D.	94 ± 18^{b}	19 ± 2^{a}	84 ± 16^{b}
1092	3-Ethyl-2,5-dimethyl-pyrazine	MS/O/RI	roasted	N.D.	N.D.	N.D.	342 ± 10^{a}	1,172 ± 33^{c}	344 ± 22^{a}	467 ± 46^{b}
1119	2-Methyl-6-(1-(E)-propenyl)-pyrazine	MS/RI	roasted	N.D.	N.D.	N.D.	108 ± 5^{b}	163 ± 21^{c}	54 ± 8^{a}	N.D.
1132	3-Methyl-2-pyrazinylmethanol	MS/RI	burnt, smoky	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	17 ± 5^{a}
1134	1-(5-Methyl-2-pyrazinyl)-1-ethanone	MS	roasted	N.D.	N.D.	N.D.	83 ± 2^{b}	N.D.	6 ± 1^{a}	N.D.
1135	1-(6-Methyl-2-pyrazinyl)-1-ethanone	MS	caramel, popcorn	N.D.	N.D.	N.D.	86 ± 5^{b}	133 ± 13^{d}	15 ± 5^{a}	103 ± 23^{c}
1159	5H-5-Methyl-6,7-dihydrocyclopentapyrazine	MS/O/RI/STD	popcorn	N.D.	N.D.	N.D.	N.D.	74 ± 5^{c}	9 ± 1^{a}	59 ± 12^{b}
1171	3,5-Diethyl-2-methyl-pyrazine	MS/O/RI	scallion, caramel	N.D.	N.D.	N.D.	25 ± 9^{a}	144 ± 7^{d}	34 ± 2^{b}	45 ± 10^{c}
1226	6,7-Dihydro-2,5-dimethyl-5H-cyclopentapyrazine	MS	burnt	N.D.	N.D.	N.D.	N.D.	46 ± 7^{c}	9 ± 1^{a}	28 ± 5^{b}
	Total Pyrazines			**N.D.**	**N.D.**	**11 ± 2^{a}**	**2,438 ± 77^{b}**	**10,274 ± 139^{d}**	**2,493 ± 142^{b}**	**7,297 ± 682^{c}**
769	Pyrrole	MS/O/RI/STD	caramel, bitter	N.D.	N.D.	N.D.	17 ± 4^{a}	145 ± 22^{b}	18 ± 2^{a}	289 ± 74^{c}
813	1-Ethyl-1H-pyrrole	MS/O/RI/STD	popcorn, roasted	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	25 ± 6^{a}
858	2-Methyl-1H-pyrrole	MS/RI	burnt, roasted	N.D.	N.D.	N.D.	5 ± 1^{a}	12 ± 4^{ab}	17 ± 8^{b}	102 ± 17^{c}
1092	1-(1H-Pyrrol-2-yl)-ethanone	MS/O/RI/STD	green	N.D.	N.D.	N.D.	N.D.	241 ± 26^{b}	N.D.	126 ± 10^{a}
1125	1,5-Dimethyl-2-pyrrolecarbonitrile	MS/O/RI	caramel, popcorn	N.D.	N.D.	N.D.	N.D.	61 ± 6^{c}	6 ± 0^{a}	50 ± 10^{b}
	Total Pyrroles			**N.D.**	**N.D.**	**N.D.**	**22 ± 6^{a}**	**460 ± 22^{b}**	**41 ± 11^{a}**	**603 ± 87^{c}**
703	2,5-Dimethyl-furan	MS/O/RI/STD	caramel	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	12 ± 4^{a}
719	2-Vinylfuran	MS/O/RI	roasted sesame	N.D.	N.D.	N.D.	N.D.	3 ± 1^{a}	N.D.	36 ± 16^{b}
846	3-Furaldehyde	MS	caramel, sweet	N.D.	N.D.	7 ± 1^{a}	59 ± 3^{b}	425 ± 35^{c}	14 ± 3^{a}	N.D.
853	2-(2-Propenyl)-furan	MS/O/RI	caramel, fruit	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	17 ± 3^{a}
878	2-Furanmethanol	MS/OSTD	roasted peanuts	N.D.	N.D.	N.D.	16 ± 2^{a}	268 ± 11^{c}	13 ± 2^{a}	201 ± 33^{b}
878	3-Furanmethanol	MS/RI/STD	caramel, salty	N.D.	N.D.	N.D.	N.D.	273 ± 5^{b}	13 ± 4^{a}	N.D.
926	1-(2-Furanyl)-ethanone	MS/O/RI/STD	salty, roasted	N.D.	N.D.	N.D.	85 ± 3^{a}	145 ± 9^{c}	N.D.	127 ± 24^{b}
931	2(5H)-Furanone	MS/O/RI/STD	caramel	N.D.	N.D.	N.D.	N.D.	44 ± 3^{a}	N.D.	N.D.
977	5-Methyl-2-furancarboxaldehyde	MS/O/RI/STD	sweet, nutty, caramel	N.D.	N.D.	N.D.	29 ± 2^{a}	345 ± 15^{c}	13 ± 2^{a}	279 ± 33^{b}
987	2,4-Dihydroxy-2,5-dimethyl-3(2H)-furan-3-one	MS/O/RI	barley, butter	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	171 ± 30^{a}
1007	2-Pentyl-furan	MS/O/RI/STD	roasted	7 ± 1^{a}	N.D.	N.D.	N.D.	N.D.	25 ± 3^{b}	30 ± 6^{c}
1109	Furaneol	MS/O/RI/STD	popcorn	N.D.	N.D.	N.D.	N.D.	N.D.	100 ± 8^{a}	213 ± 36^{b}
	Total Furans			**7 ± 1^{a}**	**N.D.**	**7 ± 1^{a}**	**188 ± 12^{b}**	**1,503 ± 129^{d}**	**178 ± 15^{b}**	**1,087 ± 153^{c}**
749	Thiazole	MS/O	roasted meat	N.D.	N.D.	N.D.	N.D.	24 ± 2^{b}	7 ± 1^{a}	54 ± 13^{c}
810	2-Methyl-thiazole	MS/O/RI/STD	onion, garlic	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	20 ± 3^{a}
819	4-Methylthiazole	MS/O/RI/STD	potato chips	N.D.	N.D.	N.D.	N.D.	38 ± 2^{b}	7 ± 1^{a}	55 ± 12^{c}
901	2,4-Dimethyl-thiazole	MS/O/RI/STD	salty, roasted, garlic	N.D.	N.D.	N.D.	N.D.	19 ± 2^{a}	N.D.	18 ± 3^{a}
921	2,5-Dimethyl-thiazole	MS/O	roasted, nutty	N.D.	N.D.	N.D.	N.D.	28 ± 2^{b}	N.D.	23 ± 2^{a}
936	4,5-Dimethyl-thiazole	MS/O/RI	popcorn, rice	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	5 ± 2^{a}
942	4,5-Dihydro-2-methyl-thiazole	MS/O/RI/STD	sulphury	N.D.	N.D.	N.D.	N.D.	44 ± 2^{c}	8 ± 1^{a}	31 ± 7^{b}

(*continued on next page*)

Table 1 (*continued*)

RI [A]	Aroma-active compounds	Identification Method [B]	Odour Description [C]	Relative concentration (μg/kg) in sesame oil sample						
				RP	RS	M3S	M6S	M9S	M6P	HP
960	2,4-Dimethyl-2-thiazoline	MS/O/RI	spicy	N.D.	N.D.	N.D.	N.D.	277 ± 8^{c}	82 ± 5^{a}	139 ± 26^{b}
1032	2-Acetylthiazole	MS/O/RI/STD	roasted meat	N.D.	N.D.	N.D.	N.D.	21 ± 1^{a}	N.D.	N.D.
1298	4-Methyl-5-thiazoleethanol	MS/O	nutty	N.D.	N.D.	N.D.	N.D.	91 ± 7^{b}	N.D.	44 ± 11^{a}
	Total Thiazolines			**N.D.**	**N.D.**	**N.D.**	**N.D.**	**541 ± 56^{c}**	**104 ± 5^{a}**	**390 ± 52^{b}**
764	3-Methyl-thiophene	MS/O/RI	musty, sulfur, burnt	N.D.	N.D.	N.D.	N.D.	11 ± 1^{a}	N.D.	31 ± 4^{b}
963	Dihydro-3-(2H)-thiophenone	MS/O/RI	popcorn, sweet	N.D.	N.D.	N.D.	20 ± 2^{a}	90 ± 5^{c}	18 ± 1^{a}	40 ± 5^{b}
1153	1-(4-Hydroxy-3-thienyl)-ethanone	MS	meat, roasted	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	9 ± 2^{a}
	Total Thiophenones			**N.D.**	**N.D.**	**N.D.**	**20 ± 2^{a}**	**101 ± 5^{d}**	**18 ± 1^{a}**	**85 ± 17^{c}**
1128	Maltol	MS/RI	smoky	N.D.	N.D.	N.D.	78 ± 10^{a}	230 ± 23^{b}	N.D.	81 ± 7^{a}
1331	2-Methoxy-4-vinylphenol	MS/O/RI/STD	smoky	N.D.	N.D.	88 ± 8^{a}	467 ± 31^{d}	951 ± 28^{e}	394 ± 30^{c}	227 ± 49^{b}
1353	1,3-Benzodioxol-5-ol	MS/O/RI/STD	burnt, roasted	N.D.	N.D.	N.D.	N.D.	297 ± 7^{a}	N.D.	354 ± 27^{b}
	Total Phenols			**N.D.**	**N.D.**	**88 ± 8^{a}**	**546 ± 37^{c}**	**1,477 ± 112^{e}**	**394 ± 30^{b}**	**664 ± 48^{d}**
<700	Acetic acid	MS/O/STD	sour	N.D.	383 ± 42^{b}	N.D.	N.D.	120 ± 8^{a}	N.D.	N.D.
755	Pyridine	MS/O/RI/STD	sour, pesticide	N.D.	N.D.	N.D.	N.D.	27 ± 2^{a}	10 ± 2^{a}	64 ± 6^{b}
757	Dimethyl disulfide	MS	onion, roasted	N.D.	7 ± 1^{a}	N.D.	N.D.	27 ± 6^{a}	8 ± 1^{a}	87 ± 14^{b}
878	1,3-Dimethyl-benzene	MS/O/RI/STD	bitter, tablet	30 ± 10^{a}	145 ± 15^{c}	129 ± 32^{bc}	108 ± 30^{b}	143 ± 21^{bc}	32 ± 1^{a}	38 ± 10^{a}

Note: Sample oil **RP**: raw seed - screw pressing; sample oil **RS**: raw seed - subcritical extraction; sample oil **M3S**, **M6S**, and **M9S**: microwaved seed (3, 6, and 9 min) - subcritical extraction; sample oil **M6P**: 6 min microwaved seed - screw pressing; sample oil **HP**: hot-air roasted seed (180 °C, 20 min) - screw pressing.
For each row, values without any same superscript letters are significantly different ($p < 0.05$). "N.D." indicates not detected.

[A] RI: Retention index on HP-5MS capillaries.

[B] MS, RI, O, and STD represent that the compounds were identified by mass spectra, retention index, olfactometry, and standard chemical, respectively.

[C] Odour description was obtained by GC-O analysis.

subcritical-extracted M6S oil (2.14 ng/g) contained less PAH4 than the screw-pressed M6P oil (3.21 ng/g) ($p < 0.05$). PAHs are probably highly soluble in subcritical propane and can be removed from sesame oil during solvent removal (Ji, Jiang, Zhang, Hou, & Sun, 2022).

The sum concentration of PAH4 in the HP oil (3.97 ng/g) was the highest among all oils, suggesting that traditional hot-air roasting is more efficient in generating PAH4 than microwave pretreatment. This is consistent with previously published studies showing that appropriate microwave pretreatment on oilseeds could reduce the level of PAH4 in oils compared with hot-air roasting (Yin, Shi, Li, Wang, et al., 2022; Yin et al., 2023).

HCAs in oils

Norharman and Harman are the two most commonly found HCAs in sesame oil which are "Group 2B" carcinogens (Zhang et al., 2020). No HCA was detected in the RS and RP oils (Fig. 3**F & G**). The concentrations of Norhaman and Harman in the subcritical-extracted sesame oils increased from 3.33 ng/g to 45.86 ng/g with the increasing microwave time from 3 min to 9 min ($p < 0.05$). Norhaman and Harman are generated through the pyrolysis of proteins and amino acids during microwave treatment and accumulated upon prolonged heating (Zhang et al., 2020). The subcritical-extracted M6S oil had significantly lower HCAs (9.20 ng/g) than the screw-pressed M6P oil (60.69 ng/g) ($p < 0.05$). The excessive heat generated inside the screw expeller during screw pressing may have generated more HCAs in sesame oil compared with the low-temperature subcritical extraction. In addition, the HP oil (390.50 ng/g) had the highest content of HCAs among all sesame oils ($p < 0.05$), suggesting that traditional hot-air roasting may generate more HCAs than microwaves.

Aroma-active compounds in differently processed sesame oils

Using GC-O-MS, a total of 102 aroma-active compounds were identified in sesame oils (Table 1). The sum concentrations of aroma-active terpenes, alcohols, esters, ketones, aldehydes, phenols, S-, O-, and *N*-heterocyclic compounds in different sesame oils were normalised and visually compared in a heatmap (Fig. 4). The blue to red blocks indicate the relative contents of aroma-active compounds from low to high.

Aroma-active terpenes, alcohols, and esters

Most terpenes, alcohols, and esters are naturally occurring substances in oilseeds and are unstable upon heating (Yin et al., 2021; Yin, Shi, Li, Ma, et al., 2022). In general, the concentrations of aroma-active terpenes, alcohols, and esters were significantly higher in the subcritical-extracted oils (RS, M3S, M6S, and M9S) than in the screw-pressed oils (RP, M6P, and HP) ($p < 0.05$) (Fig. 4). The closed environment and low processing temperature in the subcritical extraction equipment may reduce the loss of many natural volatile compounds in sesame oils through evaporation, oxidation, and decomposition. With the increasing microwave time, aroma-active esters, alcohols, and terpenes in the subcritical-extracted sesame oils continuously decreased ($p < 0.05$). It may be due to the evaporation and decomposition of these natural concurring volatiles in sesame seeds during microwave roasting. This was consistent with previous studies reporting microwaves reduce the concentrations of aroma-active esters, alcohols, and terpenes in sunflower oils (Yin, Maradza, Xu, Ma, et al., 2022) and sesame oils (Yin et al., 2023).

Aroma-active aldehydes and ketones

Aroma-active aldehydes and ketones detected in sesame oils are mainly derived from lipid oxidation (Zhang et al., 2021). The concentrations of aroma-active aldehydes and ketones in the subcritical-extracted RS oil were 34 μg/kg and 331 μg/kg, respectively, higher than in the screw-pressed RP oil (27 μg/kg and 26 μg/kg, respectively) ($p < 0.05$). An increased degree of lipid auto-oxidation may have occurred in the RS oil due to the high activities of lipase and

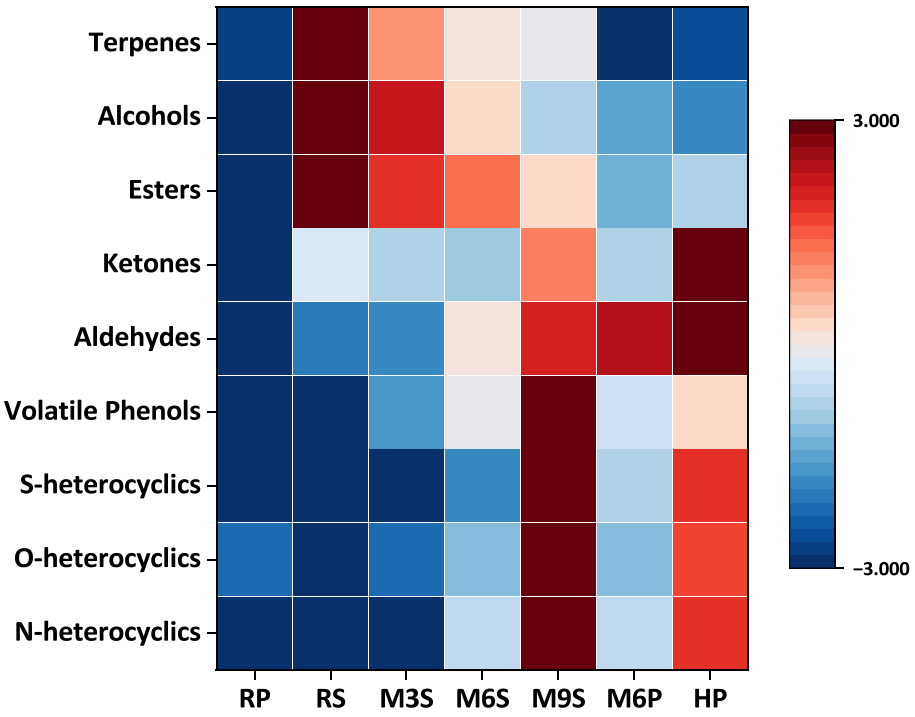


Fig. 4. A heatmap analysis visualised by Z-score normalization for the sums of different aroma-active compounds in sesame oil samples.

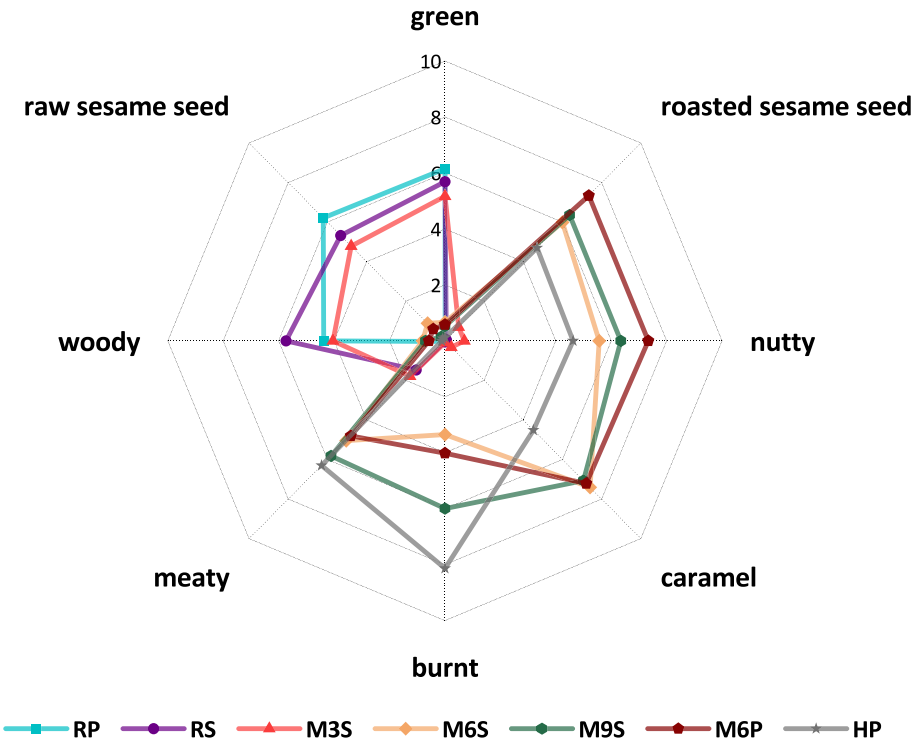


Fig. 5. The aroma profiles of sesame oil samples produced through different processes.

lipoxygenase under low-temperature subcritical extraction (Tian & Hua, 2021). The concentrations of aroma-active aldehydes and ketones in subcritical-extracted sesame oils increased with the increasing microwave time ($p < 0.05$), reflecting the increased degree of lipid oxidation upon the thermal effect of microwaves. More aroma-active aldehydes and ketones were identified in the screw-pressed M6P oil (889 μg/kg and 217 μg/kg) than in the subcritical-extracted M6S (405 μg/kg and 196 μg/kg). It was probably due to the high processing temperature inside the screw chamber that caused more lipid oxidation than subcritical extraction. The HP oil had the highest amounts of aroma-active aldehydes (959 μg/kg) and ketones (1,021 μg/kg) among all sesame oils ($p < 0.05$), indicating that that the hot-air roasting may cause a greater degree of lipid oxidation than microwaves.

Aroma-active heterocyclic compounds

Heterocyclic compounds are produced through the Maillard reaction, Strecker degradation, and caramelisation during oilseed roasting (Zhang, Cao, & Liu, 2020). No aroma-active heterocyclic compound was detected in the RS oil, while only one heterocyclic compound, i.e. 2-pentyl-furan, was detected in the RP oil (7 μg/kg). 2-Pentyl-furan is a typical lipid oxidation product in sesame oil (Yin et al., 2021). The heat generated through extrusion and friction inside the screw expeller may facilitate lipid oxidation to form 2-pentyl-furan.

The *N*-heterocyclics detected in sesame oils (Table 1) included pyrazines, pyrroles, and pyridines which are products generated through the Maillard reaction between amino compounds and reducing sugars (Yin et al., 2021). The O-heterocyclics (furans) detected in sesame oils are produced through sugar caramelisation or lipid peroxidation (Liu, Wang, Tamogami, Chen, & Zhang, 2020). The S-heterocyclics detected in sesame oils included thiazoles and thiophenes which are products of the Maillard reaction between sulphur-containing amino compounds and reducing sugars, or products of Strecker degradation of sulphury amino acids (Jia, Zhou, Wang, Liu, Huang, & Huang, 2019). The concentrations of aroma-active N-, O-, and S-heterocyclics increased with the increasing microwave time in the subcritical-extracted sesame oils ($p < 0.05$) (Fig. 4). There was no significant difference in aroma-active N- and O-heterocyclics between the screw-pressed M6P oil and the subcritical-extracted M6S oil, both of which were extracted from 6 min microwaved sesame seeds. However, the sum concentration of aroma-active S-heterocyclics in the M6P oil (122 μg/kg) was higher than that in the M6S oil (20 μg/kg) ($p < 0.05$). Part of the highly versatile S-heterocyclics might have been evaporated during the subcritical solvent removal process (Liu et al., 2020). In addition, the sum concentration of aroma-active N-, O- or S-heterocyclics in the M9S was significantly higher than that in the HP oil ($p < 0.05$), suggesting that microwave roasting could generate more aroma-active heterocyclics in a shorter process time than conventional hot-air roasting. This is consistence with a previous comparison of the flavour generation efficiency between microwaves and hot air (Yin et al., 2023).

Aroma-active phenolic compounds

Aroma-active phenolics were absent from the RS and RP oils which were extracted from raw sesame seeds (Table 1). The sum concentration of aroma-active phenolics increased dramatically with the increasing microwave time in the subcritical-extracted sesame oils ($p < 0.05$). The production of volatile phenolics such as 2-methoxy-4-vinylphenol is caused by the thermal depolymerisation of lignins in sesame seed husks upon roasting (He et al., 2022). The concentration of the aroma-active phenolics in M6P oil (394 μg/kg) was significantly lower than in the M6S oil (546 μg/kg), suggesting that the subcritical extraction of the aromatic phenolics was more efficient than the screw pressing. In addition, the M9S oil contained the highest concentration of aroma-active phenolics (1,477 μg/kg), which was more than twice that in the HP oil (664 μg/kg) ($p < 0.05$). This suggests that microwaves efficiently generated more aroma-active phenolics in a shorter process time than conventional hot-air roasting.

Sensory perception of differently processed sesame oils

The aroma perceptions of all sesame oil samples are shown in Fig. 5. There was a significant sample effect ($p < 0.05$) on all aroma attributes and no significant interaction effect between samples and panelists, indicating that the panel was performed adequately. The aroma perceptions of RP, RS, and M3S oils were dominated by the woody, raw sesame seed and green notes, while the M6S, M6P, M9S, and HP oils were mainly characterised by the perceived aromas of roasted sesame seed, nutty, caramel, burnt, and meaty. With the increase of microwave time from 6 min to 9 min, the perceived intensities of roasted sesame seed, nutty, and burnt aromas increased in the subcritical-extracted oils (M6S and M9S) ($p < 0.05$). This is due to the accumulation of volatile products from the Maillard reaction and caramelisation volatile during heating (Yin et al., 2021). Compared with the M6P oil, the perceived intensities of roasted sesame seed, nutty, and burnt aromas slightly decreased in the M6S oil ($p < 0.05$), while the caramel and meaty aromas were not significantly different between the two oils.

The hot-air pretreated sesame oil (HP) had a stronger perceived burnt aroma while weaker perceived roasted sesame seed, nutty and caramel aromas compared with the microwave-pretreated sesame oils (M3S, M6S, M9S, and M6P) ($p < 0.05$). The sensory attributes of caramel, nutty, and roasted sesame seed aromas are generally preferred by consumers, while the strong burnt aroma is considered an undesired attribute in sesame oil (Yin et al., 2020). It indicates that microwave pretreatment may have the advantage of producing sesame oil with a more desired sensory profile than traditional hot-air roasting.

Conclusion

The novel processing method to produce fragrant sesame oil using microwave pretreatment followed by subcritical propane extraction has several advantages over the conventional combination of hot-air roasting and screw pressing. Microwaves can generate flavour faster in sesame oil with a better sensory profile compared to conventional hot-air roasting. Subcritical propane low-temperature extraction improves the oil yield and lipid oxidation stability and has higher extraction rates for tocopherols, sesame lignans, aroma-active terpenes, alcohols, and esters while reducing the carcinogenic PAHs and HCAs in sesame oil. The combination of microwaves and subcritical extraction is promising for producing fragrant sesame oil with superior qualities of nutrition, flavour, and safety while improving the utilisation value of sesame protein. The emerging subcritical low-temperature extraction, as a green, economical, and eco-friendly technique with low energy consumption and carbon emission, has a great potential to be applied from a broader perspective in the agro-food, flavour, and fragrance industries to efficiently extract lipids, proteins, bioactive components, essential oils, flavour, and fragrance et al. Future studies could focus on the scale-up application of microwaves and subcritical extraction in a variety of foods and by-products.

CRediT authorship contribution statement

Fan Zhang: Data curation, Formal analysis, Investigation, Writing – original draft. **Xue-de Wang:** Funding acquisition, Methodology, Supervision. **Ke Li:** Data curation, Investigation. **Wen-ting Yin:** Conceptualization, Funding acquisition, Supervision, Writing – review & editing. **Hua-min Liu:** Methodology, Validation. **Xin-liang Zhu:** Methodology, Validation. **Peng Hu:** Methodology, Project administration.

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

The data that has been used is confidential.

Acknowledgments

This work was funded by the China Agriculture Research System of MOF and MARA [grant number CARS-14, 2022-2025], the National Natural Science Foundation of China [grant number 32202210, 2023-2025], the Joint Fund of Science and Technology Research and Development Plan of Henan Province [grant number 232103810071, 2023-2024], and the Cultivation Programme for Young Backbone Teachers in

Henan University of Technology.

Informed Consent Statement.

All participants were clearly informed about the details of this study and their consent was obtained prior to the study. Appropriate protocols for protecting the rights and privacy of all participants were utilised during the execution of the study.

References

Ahmed, I. A. M., Uslu, N., Ozcan, M. M., Juhaimi, F. A. L., Ghafoor, K., Babiker, E. E., et al. (2021). Effect of conventional oven roasting treatment on the physicochemical quality attributes of sesame seeds obtained from different locations. *Food Chemistry, 338*, Article 128109. https://doi.org/10.1016/j.foodchem.2020.128109

Corso, M. P., Fagundes-Klen, M. R., Silva, E. A., Cardozo, L., Santos, J. N., Freitas, L. S., et al. (2010). Extraction of sesame seed (*Sesamun indicum* L.) oil using compressed propane and supercritical carbon dioxide. *The Journal of Supercritical Fluids, 52*(1), 56–61. https://doi.org/10.1016/j.supflu.2009.11.012

Food and Agriculture Organization (2020). Crops and livestock products. Retrieved from https://www.fao.org/faostat/zh/#data/QCL. Accessed August 23, 2023.

Gao, P., Ding, Y. P., Chen, Z., Zhou, Z., Zhong, W., Hu, C., et al. (2022). Characteristics and antioxidant activity of walnut oil using various pretreatment and processing technologies. *Foods, 11*(12), 1698. https://doi.org/10.3390/foods11121698

Gharby, S. (2022). Refining vegetable oils: Chemical and physical refining. *The Scientific World Journal, 2022*, 6627013. https://doi.org/10.1155/2022/6627013

Guo, T. T., Wan, C. Y., Huang, F. H., & Wei, C. L. (2021). Evaluation of quality properties and antioxidant activities of tiger nut (Cyperus esculentus L.) oil produced by mechanical expression or/with critical fluid extraction. *LWT-Food Science and Technology, 141*, Article 110915. https://doi.org/10.1016/j.lwt.2021.110915

He, M. K., He, Y. L., Li, Z. Q., Zhao, L. N., Zhang, S. Q., Liu, H. M., et al. (2022). Structural characterization of lignin and lignin-carbohydrate complex (LCC) of sesame hull. *International Journal of Biological Macromolecules, 209(PartA)*, 258–267. https://doi.org/10.1016/j.ijbiomac.2022.04.009

Hrnčič, M. K., Cör, D., Verboten, M. T., & Knez, Ž. (2018). Application of supercritical and subcritical fluids in food processing. *Food Quality and Safety, 2*(2), 59–67. https://doi.org/10.1093/fqsafe/fyy008

Hu, H., Liu, H. Z., Shi, A. M., Liu, L., Fauconnier, M. L., & Wang, Q. (2018). The effect of microwave pretreatment on micronutrient contents, oxidative stability and flavor quality of peanut oil. *Molecules, 24*(1), 62. https://doi.org/10.3390/molecules24010062

Huang, H. W., Hsu, C. P., Yang, B. B., & Wang, C. Y. (2013). Advances in the extraction of natural ingredients by high pressure extraction technology. *Trends in Food Science & Technology, 33*(1), 54–62. https://doi.org/10.1016/j.tifs.2013.07.001

Ji, J. M., Jiang, M. M., Zhang, Y. X., Hou, J., & Sun, S. D. (2022). Polycyclic aromatic hydrocarbons contamination in edible oils: A review. *Food Reviews International, 39*(9), 6977–7003. https://doi.org/10.1080/87559129.2022.2131816

Ji, J. M., Liu, Y. L., Shi, L. K., Wang, N. N., & Wang, X. D. (2019). Effect of roasting treatment on the chemical composition of sesame oil. *LWT-Food Science and Technology, 101*, 191–200. https://doi.org/10.1016/j.lwt.2018.11.008

Jia, X., Zhou, Q., Wang, J. Q., Liu, C. S., Huang, F. H., & Huang, Y. (2019). Identification of key aroma-active compounds in sesame oil from microwaved seeds using E-nose and HS-SPME-GCxGC-TOF/MS. *Journal of Food Biochemistry, 43*(10), Article e12786. https://doi.org/10.1111/jfbc.12786

Jin, L., Guo, Q., Zhang, M., Xu, Y. T., Liu, H. M., Ma, Y. X., et al. (2022). Effects of non-lipid components in roasted sesame seed on physicochemical properties of sesame paste. *LWT- Food Science and Technology, 165*, Article 113745. https://doi.org/10.1016/j.lwt.2022.113745

Lavenburg, V. M., Rosentrater, K. A., & Jung, S. (2021). Extraction methods of oils and phytochemicals from seeds and their environmental and economic impacts. *Processes, 9*(10), 1839. https://doi.org/10.3390/pr9101839

Li, F., Muhmood, A., Tavakoli, S., Park, S., Kong, L., Zhu, H., et al. (2023). Subcritical low temperature extraction of bioactive ingredients from foods and food by-products and its applications in the agro-food industry. *Critical Reviews in Food Science and Nutrition*. https://doi.org/10.1080/10408398.2023.2198009

Liu, H. M., Yao, Y. G., Ma, Y. X., & Wang, X. D. (2020). Ultrasound-assisted desolventizing of fragrant oil from red pepper seed by subcritical propane extraction. *Ultrasonics Sonochemistry, 63*, Article 104943. https://doi.org/10.1016/j.ultsonch.2019.104943

Liu, X. F., Wang, S., Tamogami, S., Chen, J. Y., & Zhang, H. (2020). Volatile profile and flavor characteristics of ten edible oils. *Analytical Letters, 54*(9), 1423–1438. https://doi.org/10.1080/00032719.2020.1803896

Martínez, M. L., Bordón, M. G., Lallana, R. L., Ribotta, P. D., & Maestri, D. M. (2017). Optimization of sesame oil extraction by screw-pressing at low temperature. *Food and Bioprocess Technology, 10*(6), 1113–1121. https://doi.org/10.1007/s11947-017-1885-4

Nimet, G., Da Silva, E. A., Palú, F., Dariva, C., Freitas, L. D., Neto, A. M., et al. (2011). Extraction of sunflower (Heliantus annuus L.) oil with supercritical CO_2 and subcritical propane: Experimental and modeling. *Chemical Engineering Journal, 168*(1), 262–268. https://doi.org/10.1016/j.cej.2010.12.088

Potočnik, T., & Košir, I. J. (2016). Influence of roasting temperature of pumpkin seed on PAH and aroma formation. *European Journal of Lipid Science and Technology, 119*(3), 1500593. https://doi.org/10.1002/ejlt.201500593

Qin, Z., Han, Y. F., Wang, N. N., Liu, H. M., Zheng, Y. Z., & Wang, X. D. (2020). Improvement of the oxidative stability of cold-pressed sesame oil using products from the Maillard reaction of sesame enzymatically hydrolyzed protein and reducing sugars. *Journal of the Science of Food and Agriculture, 100*(4), 1524–1531. https://doi.org/10.1002/jsfa.10160

Shi, L. K., Zheng, L., Zhang, Y. R., Liu, R. J., Chang, M., Huang, J. H., et al. (2018). Evaluation and comparison of lipid composition, oxidation stability, and antioxidant capacity of sesame oil: An industrial-scale study based on oil extraction method. *European Journal of Lipid Science and Technology, 120*(10), 1800158. https://doi.org/10.1002/ejlt.201800158

Tian, Q. Y., & Hua, Y. F. (2021). Oxidation reactions in model systems simulating the processing of soybeans into soymilk: Role of lipase and lipoxygenase in volatile flavors formation. *International Journal of Food Properties, 24*(1), 19–202. https://doi.org/10.1080/10942912.2021.1873363

Wang, S., Zhang, Z. S., Zhang, T. F., & Wang, X. D. (2020). Extraction and characterization of flaxseed oil obtained with subcritical n-butane. *Journal of Oleo Science, 69*(9), 1011–1020. https://doi.org/10.5650/jos.ess20051

Wang, W. Y., Yan, Y. Y., Liu, H. M., Qi, K., Zhu, X. L., Wang, X. D., et al. (2021). Subcritical low temperature extraction technology and its application in extracting seed oils. *Journal of Food Process Engineering, 44*(10), Article e13805. https://doi.org/10.1111/jfpe.13805

Wei, P. P., Zhao, F. L., Wang, Z., Wang, Q. B., Chai, X. Y., Hou, G. G., et al. (2022). Sesame (*Sesamum indicum* L.): A comprehensive review of nutritional value, phytochemical composition, health benefits, development of food, and industrial applications. *Nutrients, 14*(19), 4079. https://doi.org/10.3390/nu14194079

Xu, Y. D., Zhou, Y. P., & Chen, J. (2017). Near-infrared spectroscopy combined with multivariate calibration to predict the yield of sesame oil produced by traditional aqueous extraction process. *Journal of Food Quality, 2017*, 2515476. https://doi.org/10.1155/2017/2515476

Yilmaz, N., Kodama, Y., & Numata, K. (2020). Revealing the architecture of the cell wall in living plant cells by bioimaging and enzymatic degradation. *Biomacromolecules, 21*(1), 95–103. https://doi.org/10.1021/acs.biomac.9b00979

Yin, W. T., Maradza, W., Xu, Y. F., Ma, X. T., Shi, R., Zhao, R. Y., et al. (2022). Comparison of key aroma-active composition and aroma perception of cold-pressed and roasted peanut oils. *International Journal of Food Science & Technology, 57*(5), 2968–2979. https://doi.org/10.1111/ijfs.15615

Yin, W. T., Ma, X. T., Li, S. J., Wang, X. D., Liu, H. M., & Shi, R. (2021). Comparison of key aroma-active compounds between roasted and cold-pressed sesame oils. *Food Research International, 150(Part*, A), 110794. https://doi.org/10.1016/j.foodres.2021.110794

Yin, W. T., Maradza, W., Ma, X. T., Yang, X., Lu, A., Shi, R., et al. (2020). Consumer acceptability and sensory profiling of sesame oils obtained from different processes. *Grain & Oil Science and Technology, 3*(2), 39–48. https://doi.org/10.1016/j.gaost.2020.04.001

Yin, W. T., Shi, R., Li, S. J., Ma, X. T., Wang, X. D., & Wang, A. N. (2022). Changes in key aroma-active compounds and sensory characteristics of sunflower oils induced by seed roasting. *Journal of Food Science, 87*(2). https://doi.org/10.1111/1750-3841.16044

Yin, W. T., Shi, R., Li, K., Wang, X. D., Wang, A. N., Zhao, Y. H., et al. (2022). Effect of microwave pretreatment of sunflower kernels on the aroma-active composition, sensory quality, lipid oxidation, tocopherols, heterocyclic amines and polycyclic aromatic hydrocarbons of sunflower oil. *LWT-Food Science and Technology, 170*, Article 114077. https://doi.org/10.1016/j.lwt.2022.114077

Yin, W. T., Yang, C. J., He, X. Y., Zhao, Y. H., Liu, H. M., Zhai, Z. Q., et al. (2023). Comparison of microwave and hot-air roasting on microstructure of sesame seed, aroma-active, hazardous components, and sensory perception of sesame oil. *Food Chemistry: X, 20*(2023), 101045. https://doi.org/10.1016/j.fochx.2023.101045

Zanqui, A. B., Da Silva, C., Ressutte, J., De Morais, D., Santos, J. M., Eberlin, M., et al. (2020). Brazil nut oil extraction using subcritical n-propane: Advantages and chemical composition. *Journal of the Brazilian Chemical Society, 31*(3), 603–612. https://doi.org/10.21577/0103-5053.20190225

Zhang, C. X., Xi, J., Zhao, T. P., Ma, Y. X., & Wang, X. D. (2020). β-Carbolines norharman and harman in vegetable oils in China. *Food Additives & Contaminants: Part B, 13*(3), 193–199. https://doi.org/10.1080/19393210.2020.1759701

Zhang, R. Y., Liu, H. M., Ma, Y. X., & Wang, X. D. (2019a). Characterization of fragrant oil extracted from pepper seed during subcritical propane extraction. *LWT-Food Science and Technology, 110*, 110–116. https://doi.org/10.1016/j.lwt.2019.04.072

Zhang, R. Y., Liu, H. M., Ma, Y. X., & Wang, X. D. (2019b). Effects of roasting on composition of chili seed and storage stability of chili seed oil. *Food Science and Biotechnology, 28*(5), 1475–1486. https://doi.org/10.1007/s10068-019-00578-9

Zhang, W. C., Cao, X., & Liu, S. Q. (2020). Aroma modulation of vegetable oils-A review. *Critical Reviews in Food Science and Nutrition, 60*(9), 1538–1551. https://doi.org/10.1080/10408398.2019.1579703

Zhang, Y., Li, X. L., Lu, X. Z., Sun, H., & Wang, F. J. (2021). Effect of oilseed roasting on the quality, flavor and safety of oil: A comprehensive review. *Food Research International, 150(Part*, A), 110791. https://doi.org/10.1016/j.foodres.2021.110791