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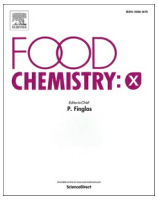

Comparison of microwave and hot-air roasting on microstructure of sesame seed, aroma-active, hazardous components, and sensory perception of sesame oil

Wen-ting Yin [a,b], Chen-jia Yang [a], Xin-yun He [a], Yu-hang Zhao [a], Hua-min Liu [a,b,*], Zhuo-qing Zhai [a], Xue-de Wang [a,b,*]

[a] School of Food Science and Technology, Henan University of Technology, 100 Lianhua Road, Zhengzhou 450001, China
[b] Institute of Special Oilseed Processing and Technology, 100 Lianhua Road, Zhengzhou 450001, China

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ABSTRACT

The unclear effects of microwaves, as a greener alternative to hot air, on sensory perception, aroma, and hazardous components of sesame oil were investigated. Microwaves (900 W, 6–10 min) created more seed porosity and cell destruction and facilitated more γ-tocopherol release in sesame oil (349.30–408.50 mg/kg) than 200 °C, 20 min hot air (304.90 mg/kg). Microwaves (6–10 min) generated more aromatic heterocyclics (42.40–125.12 mg/kg) and aldehydes (5.15–2.08 mg/kg) in sesame oil than hot air (25.59 mg/kg and 1.34 mg/kg). Microwaves (6 min) produced sesame oil with a stronger roasted sesame flavour, and weaker bitter and burnt flavour than hot air. Microwaves reduced harman (≤775.19 ng/g), norharman (≤1,069.99 ng/g), and benzo(a)pyrene (≤1.59 μg/kg) in sesame oil than hot air (1,319.85 ng/g, 1,168.40 ng/g, and 1.83 μg/kg). Appropriate microwave is a promising alternative to hot air in producing sesame oil with a better sensory profile, more bioactive, and less carcinogenic components.

Introduction

The ancient oil crop sesame (*Sesamum indicum* L.) is cultivated worldwide, especially abundant in Sudan, Myanmar, Tanzania, India, Nigeria, China, and Burkina Faso (Yin et al., 2021). The global output of sesame seeds in 2020 is approximately 6.8 million tons (FAO, 2022). Sesame seed has about 45 %-55 % fats, of which 80 % are unsaturated linoleic acid and oleic acid, making sesame oil a healthy edible oil that is associated with reduced risks of blood cholesterol and atherosclerosis diseases (Namiki, 2007). Sesame oil also contains abundant natural bioactive phytochemicals including tocopherols, phytosterol, and sesame lignans, which have shown strong anti-oxidative, anti-inflammatory, and anti-mutagenic functions (Langyan et al., 2022; Mohamed Ahmed et al., 2021; Qin et al., 2020).

Sesame seeds are traditionally pretreated by hot-air roasting (industrial mass production parametres: 150–250 °C, 20–60 min) before being extracted for sesame oil. The quality of sesame oil is determined by the seed roasting process, which is essential for flavour generation through lipid oxidation, caramelisation, Strecker degradation, and the Maillard reaction (Yin et al., 2021). Meanwhile, roasting sesame seeds at high temperatures for a long period by hot air can result in the thermal loss of nutrients such as tocopherols and proteins and may cause the synthesis of carcinogenic compounds including harman, norharman, and benzo(a)pyrene (Ji, Liu, Shi, Wang & Wang, 2019; Mohamed Ahmed et al., 2021; Shin et al., 2016).

Sesame seed contains 20–25 % proteins which are consisted of more than 30 % essential amino acids, making sesame seed an excellent plant protein resource (Liu, Yao, Yan & Wang, 2020). However, sesame proteins can be severely denatured when excessive thermal energy from the hot air is applied (Cai et al, 2021; Yang et al., 2021). As a result, the sesame meal, as the processing by-product of oil, is usually sold cheaply as fertilizer or animal feed, not as a food ingredient, which is a waste of plant protein resources (Yang et al., 2021). Not to mention that traditional hot-air processing demands high energy input and generates a large quantity of exhaust gas (Masalimov, Fayzrakhmanov, Yanbaev, Tagirova & Kiyamov, 2020). A greener, and softer technology of sesame seed pretreatment is therefore demanded to produce sesame oil, which should be more efficient, and cause less thermal destruction to sesame

* Corresponding authors at: School of Food Science and Technology, Henan University of Technology, 100 Lianhua Road, Zhengzhou 450001, China.
E-mail addresses: hmliu@haut.edu.cn (H.-m. Liu), 13903865584@126.com (X.-d. Wang).

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seeds.

Ultrasound-assisted extraction, as an emerging technology, can quickly destroy the cellular structure of the oilseeds through cavitation allowing efficient oil extraction (Chemat, Huma, & Khan, 2011). However, oilseeds should be ground and dispersed in solvents before ultrasonication (Koubaa et al., 2016), and the follow-up removal of solvents will cause a considerable loss of volatile flavour components in the oil. Another novel technique, pulsed electric fields (PEF), can also improve the oil yield of seeds (Navarro et al., 2022; Zeng, Han, & Zi, 2010), however, it may not be sufficient to generate enough flavour compounds for sesame oil due to its non-thermal nature.

Microwave radiation is more efficient in energy generation and less time-consuming than hot-air roasting due to its volumetric heating nature, making it a possible alternative to hot-air roasting for flavour generation in oilseed (Cai et al., 2021). A considerable amount of heat can be generated instantaneously inside the oilseeds as a result of the interactions of polar molecules with microwaves (Cai et al., 2021; Chandrasekaran, Ramanathan & Basak, 2013). The oil extraction rate of microwaved sesame seed has been shown to increase compared with the untreated raw sesame seeds (Abou Gharbia, Shehata & Shahidi, 2000; Mohamed Ahmed et al., 2020). It was hypothesised that microwaves may have a different impact on the physical structure of sesame seeds compared to the hot-air roasting, which may explain their different effects on the extraction efficiency and the quality of the sesame oil. Huang et al (2023) observed more cavities and cracks appeared on the surface of sesame seeds after microwaves while the surface of raw seeds was dense and smooth. However, little is known about the effect of microwaves on the inner cross-sectional and subcellular structure of sesame seeds which may provide a more insightful explanation of the influence of microwaves on oil extraction efficiency and quality of sesame oil.

Microwaves were reported to affect the contents of bioactive compounds including tocopherols and sesame lignans in sesame oils, but inconsistent results were obtained. Some showed that microwaves could improve tocopherols and sesame lignans in sesame oil while others reported the opposite results (Abou Gharbia et al., 2000; Mohamed Ahmed et al., 2020; Jia et al., 2019). The effect of microwave roasting on the contents of bioactive compounds in sesame oil may be dependent on tthe genotypes and origins of sesame and the parametres of the microwave. In addition, the heat generated by microwaves may also result in the generation of potentially carcinogenic HCAs and benzo(a)pyrene which are often detected in hot-air roasted sesame oil (Zhang et al., 2020). The effect of microwaves on the contents of carcinogenic compounds in sesame oil has not been reported but is worth investigating. The influence of microwaves on the aroma-active components of sesame oil is scarcely reported and not well understood. Jia et al. (2019) reported that volatile pyrazines in sesame oil increased with increasing microwave pretreatment time using HS-SPME and GC × GC-TOF/MS. HS-SPME is suitable for the extraction of low-molecular and highly volatile compounds but is insufficient for extracting the majority of diverse volatile components especially the low-volatility ones in sesame oil, while solvent-assisted flavour evaporation (SAFE) can extract more aroma-active compounds from sesame oil with a higher extraction efficiency and better repeatability (Yin et al., 2021; Yin et al., 2022). SAFE has been proven to be sufficient in extracting natural volatile compounds from sesame oil, sunflower oil, and olive oil at a low extraction temperature, and the aroma recombination models of SAFE extracts show a good similarity to the natural aroma profiles of these oils (Neugebauer, Granvogl, & Schieberle, 2020; Yin et al., 2021; Yin et al., 2022a). As far as the authors are aware, no report has been found on the identification of aroma-active compounds in microwave-pretreated sesame oil using SAFE and GC-O-MS analysis, which could help identify more aroma-active compounds in sesame oil and understand better the effect of microwave pretreatment on the characteristic aroma-active components of sesame oil in comparison to hot-air roasting. In addition, the effects of microwave and hot-air roasting on the sensory perception of sesame oil are scarcely compared.

It was hypothesised that appropriate microwave pretreatment of sesame seeds might have advantages in oil extraction efficiency, flavour generation, bioactive compounds maintenance, and control of hazardous components in sesame oil due to its volumetric heating mechanism which could not only reduce heating temperature and time but also have special impacts on the microstructure of sesame seeds, compared with hot-air roasting. Therefore, this study systematically investigated the influence of microwave pretreatment on the surface, cross-sectional microstructure, and subcellular structure of sesame seeds and the aroma-active composition, sensory perception, contents of tocopherols, lignans, HCAs, and benzo(a)pyrene of sesame oil, in comparison to the hot-air roasting.

2. Material and methods

2.1. Chemicals

The purity of all chemicals used was at least 95 %. Dichloromethane, anhydrous sodium sulfate, sodium hydroxide, sodium chloride, glacial acetic acid, chloroform, boron trifluoride, potassium iodide, anhydrous ethanol, isopropanol, hexane, D-glucose, ammonium formate, acetonitrile, fatty acids were purchased from Sigma Aldrich (Sternheim, Germany). Glutaraldehyde, Sodium dihydrogen phosphate, disodium hydrogen phosphate, acetone, and methanol were purchased from Kemiou Chemical Reagent Co., Ltd (Tianjin, China). Spurr's resin, osmium tetroxide, lead citrate, and uranyl acetate were from SPI-Chem Inc. (Pennsylvania, USA). The mixed standard of $C_5 - C_{30}$ alkanes was bought from Agilent Technology Co., Ltd (California, USA). Aroma-active compounds, 4-nonanol, sesame lignans, and benzo[*a*]pyrene standards were from Macklin Biochemical Co., Ltd (Shanghai, China). The standards of α-, β-, δ-, and γ-tocopherols were provided by Sanqu Biotechnology Co., Ltd (Beijing, China). The standards of Norharman and Harman were from Santa Cruz Biotechnology Inc. (California, USA).

2.2. Pretreatment of sesame seeds and production of sesame oil

White sesame seeds (Zhuzhi No. 22) were harvested in 2021 from Zhumadian, China. Sesame seeds (250 g/batch) were microwaved at 2.45 GHz and 900 W for 2, 4, 6, 8, or 10 min while being stirred once a minute. The temperature of sesame seeds was determined by a TS550 infrared thermometer (Shenzhen Hongyuan Taisheng Electronic Technology Co., Ltd, China) and moisture content was measured immediately after being microwaved according to the procedure of ISO 665: 2020. The raw sesame seeds were also hot-air roasted (200 °C, 20 min) to prepare the control oil sample. According to previous studies, the sesame oil extracted from 200 °C, 20 min hot-air roasted sesame seeds in a lab scale (250 g/batch) was considered to have a moderate sesame oil flavour among the commercial products (Yin et al., 2020 & 2021).

The raw seeds (RS), microwaved (M2, M4, M6, M8, and M10), or hot-air roasted sesame seeds (R20) (1.5 kg/batch) were then pressed by a hydraulic machine (intermittent pressure at 40 MPa for 60 min). The crude oil sample was centrifuged at 4000 RPM for 10 min, purified by a 40 μm filter paper, briefly stored at −24 °C, and analysed within a month of production. The oil yields of sesame seeds were determined based on Ji et al. (2019). Each oil sample was prepared in 3 batches for triplicate analysis.

2.3. Microstructure observation of sesame seeds

The surface and cross-sectional morphologies of sesame seeds were observed by a Quanta Feg 250 scanning electron microscope (SEM) (Thermal Fisher Scientific Inc., USA). Sesame seed was glued to the conductive tape, sprayed with a gold film, and observed under the acceleration voltage of 20.0 kV.

The subcellular structures of sesame seeds were observed by the

HT7800 transmission electron microscope (TEM) (Hitachi Co. Ltd., Japan). The sample preparation for TEM was referred to Xu et al. (2019). Sesame seeds were fixated with 2.5 % glutaraldehyde, buffered at pH 7.0 by 0.2 M phosphate buffer, and stored at −4 °C for 8 h. The solution was removed and the sample tissue was rinsed with the phosphate buffer (0.1 M, pH 7.0). The sample was then fixated with 1 % osmic acid solution for 2 h. The osmic acid solution was then removed and the samples were rinsed with the phosphate buffer (0.1 M, pH 7.0). The sample was dehydrated sequentially with increasing concentrations of ethanol solutions (from 30 %, 50 %, 70 %, 80 %, 90 %, 95 % to 100 %). The dehydrated sample tissue was then embedded in Spurr's resin at 70 °C overnight before being cut to 80 nm sections by the LEICA EM UC7 ultramicrotome (Leica Co. Ltd., Germany). Then, the ultrathin sections were stained with 1 % lead citrate solution and 5 % aqueous uranyl acetate before being examined by the TEM.

2.4. Analysis of colour indexes and lipid oxidation of sesame oil

The colour indexes, peroxide value (POV), and acid value (AV) of sesame seed oils were measured based on the procedures of ISO 27608: 2010, ISO 660: 2020, and ISO 3960: 2017, respectively. The oxidation stability of sesame oils, as indicated by the oxidation induction time (OIT, hours), was measured by a 743 Rancimat instrument (Metrohm China Ltd.), where a 5 g sesame oil sample was incubated at 110 °C, the conductivity was 0–500 μS/cm, and the airflow rate was 20 L/h (Yu et al., 2021).

2.5. Analysis of tocopherols in sesame oil

The tocopherols in sesame seed oil were measured by an E2695 HPLC-FLD (Waters Corporation, USA) and quantified according to the external calibration for the tocopherol standard compound (Yu et al., 2021). The Waters XBridege BEH Amide column (250 mm × 4.6 mm × 5 μm) was set to 40 °C. The mobile phase consisted of isopropanol and hexane (1:99, v:v) (1.0 mL/min). 10 μL sample was injected into HPLC. The emission wavelength was 330 nm and the excitation wavelength was 295 nm.

2.6. Analysis of lignans in sesame oil

The analysis of sesamol, sesamin, and sesamolin in sesame oil was conducted by the E2695 HPLC with a 2489 UV/Vis detector (Waters Corporation, USA) and quantified by the external calibration method according to the method of Shi, Liu, Jin, & Wang (2017). The lignans in sesame oil were extracted with 9 mL methanol (purity ≥ 99.9 %) and filtered by an organic filter membrane (0.22 μm) before sampling. The sample injection volume was 10 μL. The column used was the Waters Sun Fire C_{18} (250 mm × 4.6 mm × 5 μm) which was set to 30 °C. The mobile phase consisted of deionised water and methanol (30:70, V: V) (0.8 mL/min). The determination wavelength was 287 nm.

2.7. Volatile extraction of sesame oil by solvent-assisted flavour evaporation (SAFE)

SAFE was performed to extract volatile compounds from sesame oil based on Yin et al. (2022a). A total of 100 g sesame oil sample, 240 μL 4-nonanol (1 mg/ml) as the internal standard, and 225 mL dichloromethane as the solvent were mixed at 25 °C for 12 h for the initial solvent extraction. The volatile compounds from the mixture were then further extracted in the SAFE apparatus at 3 × 10^{-3} Pa, dried by anhydrous Na_2SO_4, stored at –24 °C overnight, and purified by a filter paper (40 μm). The supernatant was concentrated to 1 mL by a 50 cm Vigreux column and then a nitrogen stream of ≥ 99.999 % purity. The extract went through a 0.22 μm microfiltration membrane prior to GC analysis.

2.8. GC-O-MS analysis of aroma-active compounds

The SAFE extracts were measured by an Agilent 7890B GC coupled with a 5977 MS detector (Agilent, USA) and an ODP-3 olfactometry (Gerstel Inc., USA). The GC column applied was an HP-5MS capillary column (30 m × 0.25 mm × 0.25 μm) (Agilent). The flow rate of helium (purity ≥ 99.999 %, carrier gas) was set to 1.8 L/min. The GC oven temperature increased from 40 °C to 130 °C (5 min hold) at 3 °C/min and raised to 250 °C (5 min hold) at 10 °C/min. The effluent from the GC column was equally distributed to the ODP and the MS in the ratio of 1:1 (v: v). The compounds were analysed by MS in the electron impact mode at 70 eV in the full-scan mode (*m/z* 33——400, 2.0 scan/s). The GC injection port, the ion source, the quadrupole, and the ODP were set to 250 °C, 230 °C, 150 °C, and 280 °C, respectively. Three trained panelists (2 males and 1 female) identified any perceivable aroma-active compounds from the ODP-3 and recorded their odour description. Aroma-active compounds were verified by mass spectra (MS) according to the NIST 17 library, odour description (O), and retention indices (RI) compared with authentic standard references (STD). The retention index (RI) of an odourant was determined based on the RIs of C_5 − C_{30} *n*-alkanes which were obtained under the same GC–MS settings. Semi-quantification of aroma-active compounds was conducted based on the internal standard method (Yin et al., 2022b).

2.9. Sensory evaluation of sesame oil

Sensory characteristics of sesame oil samples were rated by a trained panel (5 females, 5 males) from the sensory panel of the Henan University of Technology based on descriptive quantitative analysis (QDA) according to the standard method of Chinese Cereals and Oils Association, T/CCOA 29:2020 Sensory Evaluation of Sesame Oil. Singed informed consent was collected from all participants prior to this study. Appropriate ethical protocols to ensure the rights and privacy of all volunteers were utilized during the execution of this research. The sesame oil (10 mL) was placed in a brown-tasting glass (75 mL), preconditioned, and tasted at 35 ± 1 °C. Panelists rated 9 sensory attributes of each sample including earthy, raw sesame seed, green, roasted sesame seed, nutty, caramel, burnt, smoky aromas, and bitter taste on a 10 cm continuous line scale where 0 represented "not detectable" while 10 cm represented "extremely strong". The panelists were asked to cleanse their palates using Granny Smith apples and water. Sensory data were obtained by Compusense Cloud (Compusense Inc., Canada) in individual booths of a sensory lab conforming to the requirements of ISO: 8589:2007.

2.10. Analysis of harman and norharman in sesame oil

Heterocyclic amines (HCAs) i.e. harman and norharman in sesame oil sample were extracted by the solid-phase extraction (SPE) (Merck KGaA, Germany) and measured by an E2695 HPLC (Waters, Massachusetts, USA) equipped with a 2475 FLD according to a published method (Zhang et al., 2020). The 250 mm x 4.6 mm x 5 μm Waters Sun Fire C_{18} column was set to 30 °C. The mobile phases (0.8 mL/min) consisted of acetonitrile (solvent A) and ammonium formate (10 mmol/L, pH = 6.8). HPLC gradient was as follows: 0–0.2 min, 0–10 % solvent A; 0.2–1 min, 10 % − 30 % solvent A; 1–10 min, 30 %-60 % solvent A; 10–15 min, 60 %-90 % solvent A. The injection sample volume was 10 μL. The excitation wavelength was 300 nm while the emission wavelength was 400 nm in the FLD. Harman and Norharman were quantified by building calibration curves for the external standards.

2.11. Analysis of benzo(a)pyrene in sesame oil

Benzo(a)pyrene in sesame oil samples was extracted by SPE and measured by the E2695 HPLC and 2475 FLD (Waters, Massachusetts, USA) according to the method of Ji, Liu, & Wang (2020). The Waters

PAH C_{18} reversed-phase column (250 mm × 4.6 mm × 5 μm) was set to 30 °C. The mobile phases (1.0 mL/min) consisted of acetonitrile (solvent A) and water. The HPLC gradient was as follows: 0–8 min, 60 % solvent A; 8–18 min, 60 %-100 % solvent A; 18–28 min, 100 % solvent A; 28–29 min, 100 %-60 % solvent A; 29–35 min, 60 % solvent A. The sample injection volume was 20 μL. Benzo(a)pyrene was quantified by building a calibration curve for the external standard.

2. 12 statistical analysis

Data were analysed by ANOVA with *Duncan's* comparison tests in SPSS 21.0 (SPSS Inc., USA). The significant level was $p \leq 0.05$ and indicated by small letters next to the data, where any two data that did not share any same small letter were significantly different. Sampling and analysis in all experiments were conducted in triplicates. Data are presented as mean ± standard deviation (SD).

3. Results and discussion

3.1. Surface, cross-sectional, and subcellular structures of sesame seeds

The surface morphologies (Fig. 1, left-column images) of raw sesame seeds and 2–6 min microwaved seeds were intact with small cylindrical protuberances. Apparent cracks and ruptures started to appear on the husks of sesame seed after being microwaved for 8 min. Large parts of the sesame husk started to flake off after being microwaved for 10 min. In comparison, larger parts of the sesame husk fell off the seed and only small pieces remained on the surface of the seed after being hot-air roasted for 20 min. The heat transfer within the seed during microwaves started from the inside and moisture was transferred from the interior of the sesame seed to the surface. In comparison, hot air heated the seed from the husk to the interior part, making the husk dry and easier to break due to dryness (Cai et al., 2021). The husk of sesame seed may have the function of preventing oxygen from permeating, thus protecting the nutrients and oils inside the cell structure (Goszkiewicz, Kołodziejczyk, & Ratajczyk, 2020; Yin et al., 2022b).

The cross-sectional morphology of a raw sesame seed (Fig. 1, middle-column image) had a few natural holes and a smooth tissue structure. With the increase of microwave time, greater porosity and more tissue destruction were observed inside the seeds. Tissue destruction in the hot-air roasted sesame seed was also observed, but less porosity was caused by hot-air roasting compared to 8–10 min microwaves. The rapid water migration from the inside of the sesame seed to the outside during microwaves may be responsible for the greater ruptures of cell structures and more seed porosity (Cai et al., 2021).

Subcellular microstructures of sesame seeds after different pretreatments were shown in the right-column images in Fig 1. Inside a raw sesame seed and the 2 min microwaved sesame seed, many oil bodies were closely packed, protein bodies were scattered in the cytoplasmic networks, and the cells were well-shaped with integrated cell walls and membranes. After ≥ 4 min microwave pretreatments, the cytoplasmic network started to break and the oil bodies aggregated to form larger oil droplets. Apparent breakage of cell walls was observed after ≥ 6 min microwave pretreatment. After 10 min microwave, coagulation of protein and deformation of the cellular structure were observed, which were possibly due to the lack of mechanical support from the integrated cell walls. In comparison, aggregation of oil bodies was also observed in the hot-air roasted sesame seed, but no apparent break of cell walls was observed. Other studies suggested that the break of cell walls, coagulation of protein bodies, and aggregation of oil bodies during heat treatment on oil seeds may reduce the affinity of oil for solid structures and result in an increased oil yield (Xu et al., 2019).

3.2. Impact of microwaves on temperature, moisture, and oil yield of sesame seed

The temperature of sesame seeds rose continuously with the extension of microwave time ($p < 0.05$) **(**Fig. 2**A)**. When the microwave time was ≤ 4 min, the temperature of sesame seeds (25 °C-175 °C) was lower than the temperature of hot-air roasting (200 °C). The temperature of sesame seed reached 215 °C, 245 °C, and 270 °C, respectively, after 6, 8, and 10 min microwaves. The moisture in raw sesame seeds was initially 4.73 %, quickly dropped to 1.32 % after 4 min microwaves, and reduced slowly to 0.22 % after 10 min microwaves which was not significantly different from the moisture of seed (0.21 %) after 20 min hot-air roasting.

The oil yield of raw sesame seeds was 23.84 %, which was improved gradually to 28.48 %-34.74 % with increasing microwave time from 2 min to 8 min ($p < 0.05$), but a further increase of microwave time to 10 min did not increase the oil yield further (34.36 %) (Fig. 2**B**). In comparison, hot-air roasting (200 ℃, 20 min) increased the oil yield to 32.62 % which was lower than the oil yields after 8–10 min microwaves. The improvement of the oil yields of sesame seeds after 8–10 min microwaves may be explained by the increased seed porosity and greater disruption of cell structures as described in section 3.1. Microwaves have been reported to efficiently increase the porosity of sunflower seeds (Yin et al., 2022b) and may break the covenant bonds between the solid cellular matrix and the oil molecules, thus facilitating the release of oil and improving oil yield (Hu et al., 2018).

3.3. Impact of microwaves on colour indexes and lipid oxidation of sesame oil

The red and yellow indexes of sesame oils did not change when sesame seeds were microwaved for ≤ 4 min and increased dramatically with the extension of microwave time from 6 min to 10 min ($p < 0.05$, Fig. 2**C & 2D**). The red indexes of the sesame oils extracted from 8 to 10 min microwaved sesame seeds were higher than the 20 min hot-air roasted sesame oil ($p < 0.05$). The darkened oil colour may indicate the accumulation of polymerised pigments, i.e. melanoidins, at the late stage of the Maillard reaction during heat treatments (Mohamed Ahmed et al., 2021). It seems that microwave pretreatment was more efficient for the generation of pigments in a shorter process time compared with hot-air roasting.

The peroxide value (POV) indicates the levels of peroxides and hydroperoxides as the primary lipid oxidation products (Hou et al., 2021). Compared with the cold-pressed sesame oil, the POVs of sesame oils extracted from the 2–6 min microwaved sesame seeds decreased slightly ($p < 0.05$) (Fig. 2**E**). This was likely because the small number of peroxides and hydroperoxides which were naturally present in the cold-pressed sesame oil quickly decomposed into aldehydes and ketones at the initial stage of microwave pretreatment (Mohamed Ahmed et al.,2021). After 8–10 min microwave pretreatment, the POVs of sesame oils increased dramatically, which may be because more peroxides and hydroperoxides were produced through lipid oxidation (Jing, Guo, Wang, Zhang & Yu, 2020). The POV of sesame oil extracted from 20 min hot-air roasted seeds was 0.83 mmol/kg, which was not statistically different from the sesame oil extracted from 2 to 6 min microwaved seeds (0.87–0.89 mmol/kg), but lower than the sesame oils extracted from 8 to 10 min microwaved seeds (3.42–3.49 mmol/kg) ($p < 0.05$). This may be due to the higher temperatures (245 °C and 270 °C) induced by 8–10 min microwaves which facilitated the thermal oxidation of fatty acids to form more peroxides and hydroxides.

The acid value (AV) indicates the level of free fatty acids in sesame oil (Yin et al., 2022b). In comparison to the sesame oil extracted from raw seeds, the AVs of sesame oils extracted from 2 to 4 min microwaved seeds decreased slightly ($p < 0.05$) (Fig. 2**F**). This may be due to the formation of aldehydes and ketones as the results of oxidation of free fatty acids that were naturally present in sesame oil (Qin et al., 2020). With the extension of microwave time from 6 min to 10 min, the AVs of sesame oils steadily increased, which was possibly due to the accumulation of free fatty acids as more triglyceride started to degrade (Ji et al., 2019). The AV of sesame oil extracted from 20 min hot-air roasted seeds

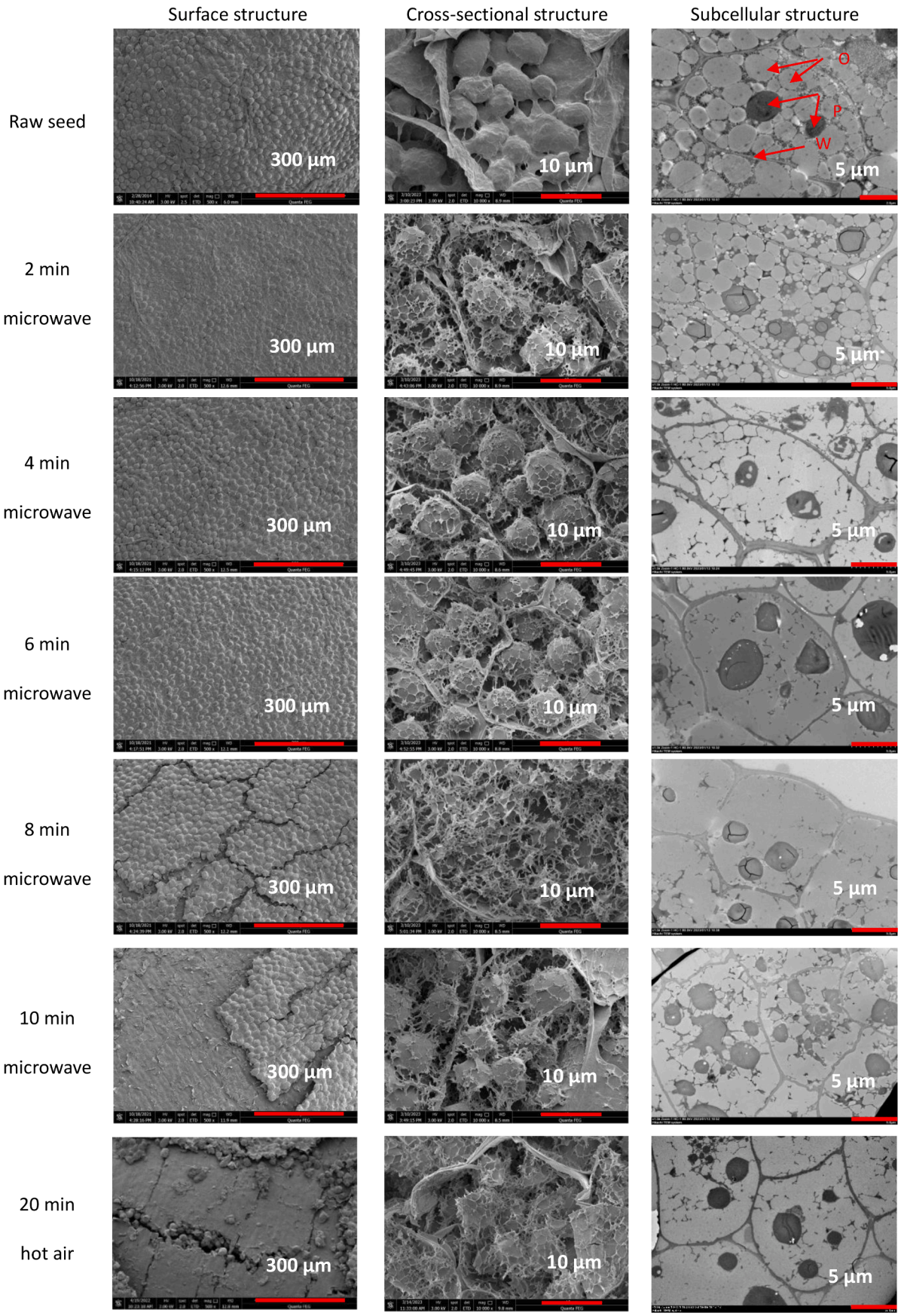

Fig. 1. Observation of the surface, cross-sectional and subcellular structures of sesame seeds after different seed pretreatments.

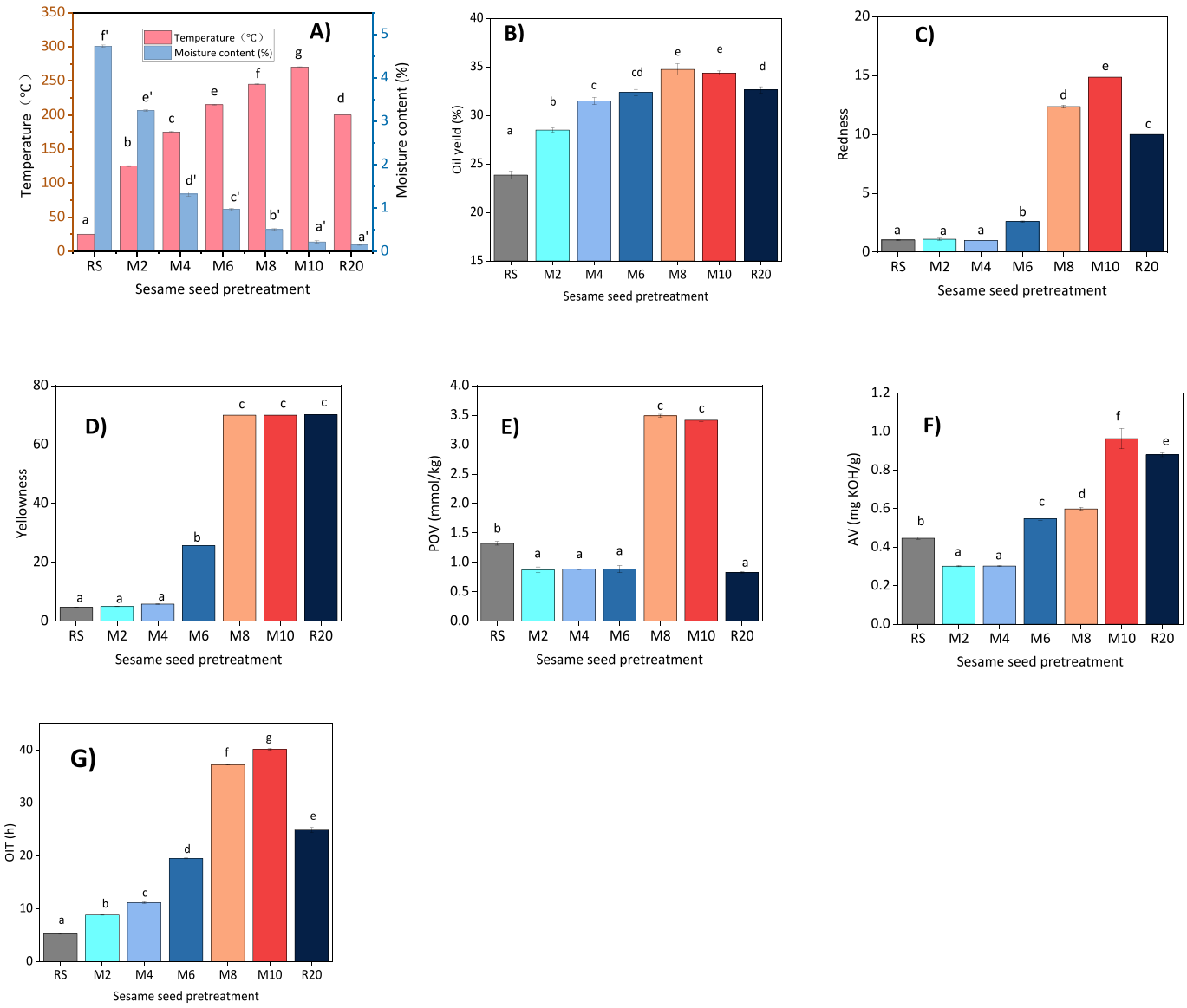

Fig. 2. Effect of different seed pretreatments on the physiochemical properties of sesame seed and sesame oil.

(0.88 mg KOH/g) was slightly lower than the 10 min microwaved oil (0.96 mg KOH/g) but higher than the ≤ 8 min microwaved oils (≤0.60 mg KOH/g) ($p < 0.05$). The AVs and POVs of all sesame oil samples were within the limits of ISO 660:2020 (4 mg KOH/g) and ISO 3960:2017 (7.5 mmol/kg), respectively.

The oxidation induction time (OIT) of sesame oils increased upon increasing microwave time ($p < 0.05$) (Fig. 2**G**), indicating that microwave pretreatment improved the oxidation stability of sesame oil. The 20 min hot-air roasting also increased the OIT of sesame oil greater than the 0–6 min microwaves, but not as much as the 8–10 min microwaves ($p < 0.05$). Many of the Maillard reaction products (MRPs) are highly antioxidative and could improve the stability of oil (Liu et al., 2020). Microwaves may be more efficient in generating antioxidative MRPs than hot-air roasting, thus improving the oxidation stability of sesame oil in a shorter processing time.

3.4. *Impact of microwaves on the concentration of γ-tocopherol in sesame oils*

Only one tocopherol, i.e. γ-tocopherol was detected in the sesame oils. The concentration of γ-tocopherol in the cold-pressed sesame oil was 381.51 mg/kg (Fig. 3**A**). Microwaving sesame seeds for 2–6 min increased the γ-tocopherol concentration in sesame oils (399.81–408.50 mg/kg) ($p < 0.05$). The destruction of cell structure and increased seed porosity by microwaves (Fig. 1) may break the bonds between tocopherol molecules and other molecules such as proteins and phospholipids, facilitating the release of more tocopherol molecules to the oil phase during the mechanical pressing process (Mohamed Ahmed et al., 2020). However, when the microwave time reached 8–10 min, the concentrations of γ-tocopherol in the sesame oils reduced slightly to 349.30–353.69 mg/kg, which were 7.29 %-8.44 % less than that in the cold-pressed sesame oil ($p < 0.05$). This was likely because of the pyrolysis and oxidation of γ-tocopherol upon accumulated heat by prolonged microwaves (Ji et al., 2019). In comparison, the 20 min hot-air roasting reduced the γ-tocopherol concentration in sesame oil to 304.91 mg/kg, which was a 20.07 % loss of γ-tocopherol compared with the cold-pressed sesame oil. Microwave pretreatments, even with a short-term higher processing temperature (215–270 °C), maintained more γ-tocopherol in sesame oil compared with hot-air roasting (200 °C, 20 min). This was partly due to higher remaining sesame seed husks after microwaves than the hot-air roasting (Fig.1). The seed husk may have the function of inhibiting oxygen from entering the sesame seed

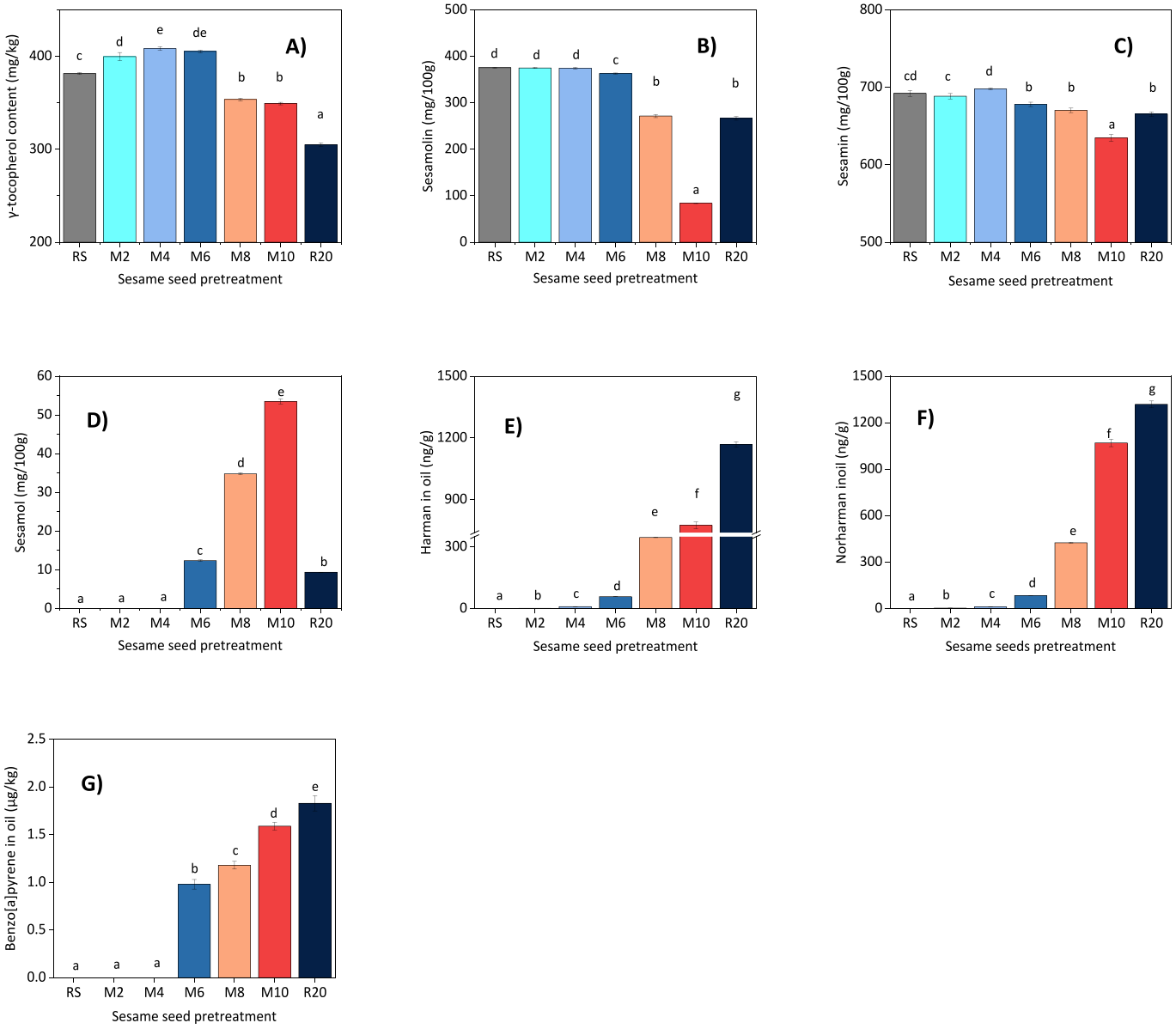

Fig. 3. Effect of different seed pretreatments on the concentrations of bioactive and hazardous compounds in sesame oils.

and hence protecting γ-tocopherol from oxidation (Hu et al., 2018).

3.5. Effect of microwaves on concentrations of lignans in sesame oil

Two sesame lignans were naturally present in the cold-pressed sesame oil, i.e. sesamolin (375.64 mg/100 g) and sesamin (692.25 mg/100 g) (Fig. 3**B & C**). The concentration of sesamolin in sesame oil reduced slowly to 363.25 mg/100 g when sesame seeds were microwaved for 6 min and dropped quickly from 271.71 mg/100 g to 83.84 mg/100 g after 8–10 min microwave pretreatment ($p < 0.05$). The amount of sesamin in sesame oil decreased slightly to 634.81 mg/100 g after 10 min microwave pretreatment ($p < 0.05$). Sesamin is a tetrahydrofuran lignan that is believed more heat-stable than sesamolin (Rosalina & Weerapreeyakul, 2021). Meanwhile, sesamol appeared in the sesame oil after 6 min microwave pretreatment (12.41 mg/100 g), and increased to 53.50 mg/100 g after 10 min microwave pretreatment ($p < 0.05$) (Fig. 3**D**). In comparison, after 20 min hot-air roasting, the concentrations of sesamolin and sesamin were reduced to 266.58 mg/100 g and 665.47 mg/100 g, respectively, which were not significantly different to the concentration of sesamolin and sesamin in sesame oil after 8 min microwave pretreatment (271.71 mg/100 g and 670.36 mg/100 g, respectively). However, the 10 min microwave pretreatment reduced the concentrations of sesamolin and sesamin to a greater extent than hot-air roasting ($p < 0.05$). The generation of sesamol in sesame oil was less by hot-air roasting (9.38 mg/100 g) compared to 6–10 min microwave pretreatment (12.41–53.50 mg/100 g). Sesamolin and sesamin can be thermally decomposed to sesamol which has a stronger antioxidant activity and is believed to contribute to the improved stability of sesame oil after seed roasting pretreatment (Rosalina & Weerapreeyakul, 2021; Yu et al., 2021).

3.6. Impact of microwaves on the aroma-active components of sesame oil

Only 5 aroma-active compounds were detected in the sesame oil extracted from raw seeds, including 2-pentyl-furan, hexanal, nonanal, 4-(1-methylethyl)-benzaldehyde, and (3*E*)-4,8-dimethylnona-1,3,7-triene (Table 1). After microwave pretreatments (900 W, 2–10 min), 98 aroma-active compounds were characterised in sesame oils, including 19 pyrazines, 13 pyrroles, 6 pyridine, 4 volatile phenols, 14 furans, 18 sulphur-containing compounds (8 thiazoles, 5 thiophenes, 4 thiols, and dimethyl

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

No.	Aroma-active compounds	RI [A]	Identification Method [B]	Odour Description [c]	Relative concentration (mg/kg) in oil after different pretreatment						
					RS	M2	M4	M6	M8	M10	R20
1	Methyl-pyrazine	820	MS/O/RI	popcorn, roasted	ND	ND	1.35 ± 0.08^{a}	10.89 ± 0.46^{c}	ND	ND	4.74 ± 0.15^{b}
2	2,5-Dimethyl-pyrazine	918	MS/O/RI/STD	roasted, nutty	ND	ND	3.26 ± 0.13^{a}	11.24 ± 0.44^{c}	35.94 ± 0.01^{d}	38.21 ± 0.01^{e}	3.72 ± 0.25^{b}
3	Ethyl-pyrazine	921	MS/O/RI/STD	roasted, nutty	ND	ND	0.32 ± 0.07^{a}	1.65 ± 0.08^{c}	2.61 ± 0.01^{d}	ND	0.38 ± 0.02^{b}
4	2,3-Dimethyl-pyrazine	925	MS/O/STD	roasted	ND	ND	0.19 ± 0.01^{a}	1.03 ± 0.11^{c}	3.76 ± 0.19^{e}	3.05 ± 0.03^{d}	0.70 ± 0.01^{b}
5	2-Ethylene piperazine	926	MS/O	spicy, roasted	ND	ND	ND	ND	ND	ND	0.25 ± 0.01^{a}
6	Ethenyl-pyrazine	943	MS/O	roasted peanut	ND	ND	ND	0.13 ± 0.01^{a}	1.27 ± 0.05^{c}	0.87 ± 0.03^{b}	ND
7	2-Isopropyl-pyrazine	969	MS/O/RI	mushroom	ND	ND	ND	ND	0.33 ± 0.01^{b}	0.30 ± 0.01^{a}	ND
8	2-Ethyl-5-methylpyrazine	989	MS/O/RI/STD	savory, roasted	ND	ND	ND	ND	ND	ND	0.26 ± 0.01^{a}
9	Trimethyl-pyrazine	1001	MS/O/RI/STD	roasted, nutty	ND	ND	1.13 ± 0.02^{a}	4.55 ± 0.18^{b}	15.02 ± 0.61^{d}	7.57 ± 0.01^{c}	ND
10	2-Ethyl-6-methylpyrazine	1008	MS/O/RI	nutty	ND	ND	ND	1.27 ± 0.09^{a}	15.88 ± 0.29^{c}	11.13 ± 0.14^{b}	ND
11	2-Ethenyl-6-methylpyrazine	1012	MS/O/RI	roasted, nutty	ND	ND	0.02 ± 0.01^{a}	0.15 ± 0.01^{b}	0.95 ± 0.03^{c}	0.91 ± 0.02^{c}	ND
12	2-ethenyl-6-methyl-Pyrazine	1018	MS/O	fried peanut	ND	ND	ND	ND	ND	ND	0.20 ± 0.02^{a}
13	2-Ethenyl-5-methylpyrazine	1025	MS/O/RI	roasted, nutty	ND	ND	ND	ND	ND	0.91 ± 0.03^{a}	ND
14	Acetyl-pyrazine	1029	MS/O/RI/STD	roasted, nutty	ND	ND	0.09 ± 0.01^{a}	ND	ND	ND	0.42 ± 0.01^{b}
15	1-(6-Methyl-2-pyrazinyl) ethanone	1029	MS	roasted sesame	ND	ND	ND	ND	ND	ND	0.42 ± 0.01^{a}
16	2,3-Diethyl-5-methylpyrazine	1049	MS/O/STD	roasted	ND	ND	ND	ND	ND	ND	0.04 ± 0.01^{a}
17	2-Methyl-5- (1-methylethyl)-pyrazine	1051	MS	nutty	ND	ND	ND	0.02 ± 0.01^{a}	ND	0.15 ± 0.00^{c}	0.06 ± 0.01^{b}
18	Isopropenyl-pyrazine	1066	MS	burnt	ND	ND	ND	0.07 ± 0.01^{a}	0.67 ± 0.01^{c}	ND	0.15 ± 0.01^{b}
19	3-Ethyl-2,5-dimethylpyrazine	1078	MS/O/RI	roasted, nutty	ND	ND	1.05 ± 0.04^{a}	3.04 ± 0.13^{c}	ND	ND	1.23 ± 0.03^{b}
20	2,6-Diethylpyrazine	1092	MS/O/RI	roasted	ND	ND	ND	ND	ND	ND	0.15 ± 0.01^{a}
21	2,3-Dimethyl-5-ethylpyrazine	1094	MS/O/RI/STD	roasted, nutty	ND	ND	0.09 ± 0.01^{a}	0.31 ± 0.02^{b}	ND	ND	ND
22	2-Methyl-6- (1-propenyl)-(E)-pyrazine	1097	MS	roasted, nutty	ND	ND	0.17 ± 0.01^{a}	0.39 ± 0.02^{b}	ND	ND	ND
23	5H-5-Methyl-6,7-dihydrocyclopentapyrazine	1146	MS/O/RI/STD	roasted, nutty	ND	ND	ND	ND	0.91 ± 0.00^{b}	ND	0.23 ± 0.01^{a}
24	3,5-Diethyl-2-methylpyrazine	1166	MS/O/RI	roasted, nutty	ND	ND	0.10 ± 0.01^{a}	ND	ND	ND	ND
25	2-Isoamyl-6-methylpyrazine	1247	MS/O	roasted peanut	ND	ND	ND	0.07 ± 0.01^{a}	ND	ND	ND
	Total pyrazines				**ND**	**ND**	**7.77 ± 0.13^{a}**	**34.81 ± 0.37^{c}**	**77.34 ± 0.33^{e}**	**63.10 ± 0.26^{d}**	**12.95 ± 0.12^{b}**
26	Pyrrole	750	MS/O/STD	medicinal, burnt	ND	ND	ND	ND	ND	ND	0.92 ± 0.10^{a}
27	2-Ethyl-1H-pyrrole	809	MS/O/RI	roasted, nutty	ND	ND	ND	0.06 ± 0.00^{a}	ND	ND	ND
28	1-Ethyl-1H-pyrrole	822	MS/STD	rubber	ND	ND	ND	ND	ND	0.85 ± 0.01^{a}	ND
29	2,3-Dimethyl-1H-pyrrole	841	MS/O/RI	baked peanuts	ND	ND	ND	ND	0.34 ± 0.02^{b}	0.45 ± 0.02^{c}	0.06 ± 0.01^{a}
30	2-Methylpyrrole (2-methyl-1H-pyrrole)	844	MS/RI	sweetness	ND	ND	ND	ND	ND	ND	0.32 ± 0.02^{a}
31	3-Methyl-1H-pyrrole	846	MS/O	roasted	ND	ND	ND	0.27 ± 0.01^{a}	0.44 ± 0.01^{b}	ND	ND
32	2-Methyl-1H-pyrrole	863	MS	roasted sesame	ND	ND	ND	ND	2.48 ± 0.05^{b}	2.13 ± 0.06^{a}	ND
33	2,5-Dimethyl-1H-pyrrole	873	MS/O/RI/STD	roasted	ND	ND	ND	ND	0.44 ± 0.04^{b}	0.75 ± 0.03^{c}	0.05 ± 0.01^{a}
34	2,4-Dimethyl-pyrrole	953	MS/RI	bitter, spicy	ND	ND	ND	ND	ND	ND	0.10 ± 0.00^{a}
35	1-Butyl-1H-pyrrole	957	MS	nutty	ND	ND	ND	ND	0.04 ± 0.00^{a}	0.12 ± 0.00^{b}	ND

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Table 1 (*continued*)

No.	Aroma-active compounds	RI [A]	Identification Method [B]	Odour Description [c]	Relative concentration (mg/kg) in oil after different pretreatment						
					RS	M2	M4	M6	M8	M10	R20
36	1H-Pyrrole-2-carboxaldehyde	1025	MS/O/RI	coffee	ND	ND	ND	0.28 ± 0.00^{a}	ND	ND	ND
37	2-Formyl-4,5-dimethyl-pyrrole	1056	MS	burnt	ND	ND	ND	ND	ND	0.11 ± 0.00^{a}	ND
38	1- (1H-pyrrol-2-yl)-ethanone	1074	MS/O/RI/STD	green	ND	ND	ND	0.28 ± 0.01^{a}	10.12 ± 0.22^{c}	14.19 ± 0.33^{d}	1.53 ± 0.07^{b}
39	1,5-Dimethyl-2-pyrrolecarbonitrile	1104	MS/O	caramel	ND	ND	0.02 ± 0.01^{a}	0.15 ± 0.01^{b}	2.27 ± 0.07^{d}	ND	0.54 ± 0.01^{c}
40	1-Methyl-1H-pyrrole-2-carboxaldehyde	1149	MS/O/RI/STD	bitter aroma	ND	ND	ND	0.31 ± 0.01^{a}	1.48 ± 0.04^{b}	ND	ND
41	1- (2-Furanylmethyl)- 1H-pyrrole	1194	MS/O/RI/STD	nutty	ND	ND	0.01 ± 0.00^{a}	0.06 ± 0.01^{b}	0.76 ± 0.01^{d}	1.34 ± 0.05^{e}	0.18 ± 0.01^{c}
	Total pyrroles				**ND**	**ND**	**0.03 ± 0.01^{a}**	**1.40 ± 0.01^{b}**	**18.37 ± 0.23^{d}**	**19.94 ± 0.35^{e}**	**3.70 ± 0.20^{c}**
42	Pyridine	735	MS/O/RI/STD	mushroom	ND	ND	ND	ND	3.14 ± 0.11^{b}	ND	1.29 ± 0.17^{a}
43	2-Methyl-pyridine	820	MS/O	medicinal	ND	ND	ND	ND	ND	0.58 ± 0.00^{a}	ND
44	3-Methyl-pyridine	868	MS	salty	ND	ND	ND	ND	0.66 ± 0.02^{b}	ND	0.18 ± 0.01^{a}
45	2,5-Dimethyl-pyridine	940	MS	smoky	ND	ND	ND	ND	ND	0.43 ± 0.01^{a}	ND
46	1-(4-Pyridinyl)- ethanone	1005	MS	peanut	ND	ND	ND	ND	ND	ND	0.03 ± 0.01^{a}
47	1-(3-Pyridinyl)- ethanone	1006	MS	roasted peanut	ND	ND	ND	ND	ND	ND	0.06 ± 0.01^{a}
48	1- (2-Pyridinyl)-ethanone	1046	MS/O/RI/STD	rubber, burnt	ND	ND	ND	ND	0.25 ± 0.01^{b}	0.34 ± 0.02^{c}	0.23 ± 0.01^{a}
49	5-Methyl-2-pyridinamine	1140	MS	smoky	ND	ND	ND	ND	ND	0.29 ± 0.00^{a}	ND
	Total pyridines				**ND**	**ND**	**ND**	**ND**	**4.05 ± 0.11^{b}**	**1.63 ± 0.05^{a}**	**1.79 ± 0.16^{a}**
50	Thiofuran	< 700	MS	garlic	ND	ND	ND	ND	ND	ND	0.18 ± 0.01^{a}
51	2,5-Dimethyl-furan	703	MS/O	caramel	ND	ND	ND	ND	0.46 ± 0.01^{b}	1.31 ± 0.02^{c}	0.18 ± 0.01^{a}
52	2-Vinylfuran	707	MS/O	caramel, nutty	ND	ND	ND	0.02 ± 0.01^{a}	0.16 ± 0.01^{c}	0.23 ± 0.02^{d}	0.13 ± 0.01^{b}
53	Furfural	828	MS/O	salty, nutty	ND	ND	0.04 ± 0.01^{a}	0.25 ± 0.01^{b}	0.82 ± 0.06^{c}	ND	ND
54	2- (2-Propenyl)-furan	859	MS/O/RI	nutty	ND	ND	ND	ND	ND	0.10 ± 0.01^{a}	0.12 ± 0.01^{b}
55	2-Furanmethanol	864	MS/O/RI/STD	nutty	ND	ND	ND	0.38 ± 0.03^{a}	ND	ND	0.96 ± 0.01^{b}
56	2-Pentanoylfuran	902	MS/STD	spicy, nutty	ND	ND	ND	ND	ND	ND	0.31 ± 0.00^{a}
57	1- (2-Furanyl)-ethanone	912	MS/O/RI/STD	sweet	ND	ND	ND	0.37 ± 0.02^{a}	1.48 ± 0.02^{c}	1.70 ± 0.10^{d}	0.51 ± 0.01^{b}
58	1- (2-Furanyl)- 1-pentanone	920	MS/RI/STD	roasted	ND	ND	0.07 ± 0.01^{a}	ND	ND	ND	ND
59	2-Furfurylthiol	928	MS/O/RI	caramel, sulphury	ND	ND	ND	ND	8.72 ± 0.38^{c}	4.88 ± 0.00^{b}	1.59 ± 0.06^{a}
60	2(5H)-Furanone	937	MS/O/RI/STD	leather	ND	ND	ND	ND	0.38 ± 0.01^{a}	0.52 ± 0.01^{b}	ND
61	2-*n*-Butylfuran	956	MS	peanut	ND	ND	ND	ND	ND	ND	0.04 ± 0.01^{a}
62	5-Methyl-2-furancarboxaldehyde	972	MS/STD	pesticide	ND	ND	0.04 ± 0.01^{a}	ND	3.24 ± 0.13^{c}	ND	0.51 ± 0.03^{b}
63	2,4-Dihydroxy-2,5-dimethyl-3 (2H)-furan-3-one	986	MS/O/RI	caramel	ND	ND	0.02 ± 0.01^{a}	0.12 ± 0.01^{b}	0.21 ± 0.00^{c}	ND	ND
64	2-Acetyl-5-methylfuran	1049	MS/O/RI	nutty	ND	ND	ND	ND	0.44 ± 0.01^{b}	0.51 ± 0.01^{c}	0.10 ± 0.01^{a}
65	2-Pentyl-furan	1098	MS/O/STD	caramel	0.02 ± 0.01^{a}	0.02 ± 0.01^{a}	0.09 ± 0.01^{b}	0.10 ± 0.01^{c}	0.20 ± 0.01^{e}	0.24 ± 0.01^{f}	0.14 ± 0.01^{d}
66	Furaneol	1100	MS/O/RI/STD	nutty	ND	ND	ND	0.88 ± 0.00^{d}	0.16 ± 0.01^{b}	0.02 ± 0.01^{a}	0.25 ± 0.01^{c}
	Total furans				**0.02 ± 0.01^{a}**	**0.02 ± 0.00^{a}**	**0.26 ± 0.01^{b}**	**2.12 ± 0.06^{c}**	**16.27 ± 0.54^{f}**	**9.51 ± 0.15^{e}**	**5.02 ± 0.06^{d}**
67	Isothiazole	708	MS/O/RI	sulphury, roasted	ND	ND	ND	0.02 ± 0.01^{a}	0.05 ± 0.01^{b}	ND	ND
68	2-Methyl-thiazole	809	MS/O/STD	sulphury, onion	ND	ND	ND	ND	0.56 ± 0.03^{c}	0.41 ± 0.02^{b}	0.28 ± 0.01^{a}
69	4-Methyl-thiazole	854	MS/O/RI/STD	sulphury, roasted	ND	0.01 ± 0.00^{a}	0.02 ± 0.01^{b}	0.30 ± 0.02^{c}	1.74 ± 0.11^{e}	0.71 ± 0.02^{d}	ND

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Table 1 (*continued*)

No.	Aroma-active compounds	RI [A]	Identification Method [B]	Odour Description [c]	Relative concentration (mg/kg) in oil after different pretreatment						
					RS	M2	M4	M6	M8	M10	R20
70	2,4-Dimethyl-thiazole	879	MS/O/RI/STD	sulphury, garlic	ND	ND	ND	0.11 ± 0.01^{a}	0.98 ± 0.08^{c}	1.04 ± 0.02^{c}	0.19 ± 0.01^{b}
71	2,5-Dimethyl- thiazole	921	MS	spicy, grilled meat	ND	ND	ND	ND	ND	ND	0.28 ± 0.00^{a}
72	4,5-Dimethyl-thiazole	947	MS/O	sulphury	ND	ND	ND	0.03 ± 0.01^{a}	0.28 ± 0.01^{b}	0.28 ± 0.01^{b}	0.06 ± 0.01^{a}
73	4,5-Dihydro-2-methyl-thiazole	950	MS/O/RI/STD	sulphury, roasted	ND	ND	0.03 ± 0.01^{a}	0.34 ± 0.02^{c}	1.49 ± 0.08^{e}	0.53 ± 0.04^{d}	0.19 ± 0.01^{b}
74	2,4-Dimethyl-2-thiazoline	954	MS/O/RI	nutty, sulphury	ND	ND	0.27 ± 0.01^{a}	1.91 ± 0.08^{c}	3.26 ± 0.02^{d}	0.72 ± 0.01^{b}	0.75 ± 0.01^{b}
75	Trimethyl-isothiazole	978	MS/O/RI	sulphury, roasted	ND	ND	ND	ND	ND	0.07 ± 0.00^{b}	0.05 ± 0.01^{a}
	Total thiazoles				**ND**	**0.01 ± 0.00^{a}**	**0.32 ± 0.01^{b}**	**2.71 ± 0.10^{d}**	**8.36 ± 0.10^{f}**	**3.76 ± 0.10^{e}**	**1.80 ± 0.03^{c}**
76	3-Methyl-thiophene	749	MS/O/RI	roasted, nutty	ND	ND	0.02 ± 0.01^{a}	0.10 ± 0.01^{b}	0.55 ± 0.03^{d}	0.56 ± 0.03^{d}	0.21 ± 0.01^{c}
77	2,5-Dihydrothiophene	758	MS/O	garlic	ND	ND	ND	ND	ND	ND	0.03 ± 0.01^{a}
78	3,4-Dimethyl-thiophene	879	MS/O/RI	roasted	ND	ND	ND	ND	ND	0.06 ± 0.01^{a}	ND
79	2,3-Dimethyl-thiophene	884	MS/O/RI	roasted, nutty	ND	ND	ND	ND	0.07 ± 0.01^{b}	ND	0.02 ± 0.01^{a}
80	Dihydro-3- (2H)-thiophenone	947	MS/O/RI	sulphury, mushroom	ND	ND	ND	0.50 ± 0.02^{a}	ND	ND	ND
81	Dihydro-2-methyl-3(2H)-thiophenone	995	MS/O/RI/STD	sulphury, popcorn	ND	ND	ND	0.07 ± 0.01^{a}	ND	0.12 ± 0.01^{b}	0.07 ± 0.02^{a}
	Total thiophenes				**ND**	**ND**	**0.02 ± 0.01^{a}**	**0.67 ± 0.01^{c}**	**0.62 ± 0.03^{c}**	**0.74 ± 0.01^{d}**	**0.33 ± 0.02^{b}**
82	2-Methyl-1-propanethiol	< 700	MS/O/RI	sulphury	ND	ND	ND	0.39 ± 0.01^{a}	ND	ND	ND
83	2-Methyl-1-butanethiol	756	MS/O	roasted	ND	ND	ND	0.04 ± 0.01^{a}	0.11 ± 0.01^{c}	0.07 ± 0.01^{b}	ND
84	3-Mercapto-propanoic acid-methyl ester	928	MS/O/STD	sulphury, rice	ND	ND	0.01 ± 0.00^{a}	0.05 ± 0.01^{b}	ND	ND	ND
85	Phenethyl mercaptan	1173	MS/O	wood	ND	ND	0.03 ± 0.01^{a}	0.21 ± 0.01^{b}	ND	0.26 ± 0.02^{c}	ND
	Total thiols				**ND**	**ND**	**0.04 ± 0.01^{a}**	**0.69 ± 0.03^{d}**	**0.11 ± 0.01^{b}**	**0.33 ± 0.01^{c}**	**ND**
86	P-cresol	839	MS	smoky	ND	ND	ND	ND	0.26 ± 0.01^{a}	0.34 ± 0.01^{b}	ND
87	Methyl-maltol	1028	MS	flowery	ND	ND	ND	ND	ND	ND	0.34 ± 0.01^{a}
88	2-Methoxy-phenol	1090	MS/O/RI	smoky	ND	ND	0.08 ± 0.01^{a}	0.72 ± 0.07^{b}	0.80 ± 0.05^{b}	1.26 ± 0.03^{c}	3.19 ± 0.18^{d}
89	Mequinol	1119	MS	smoky	ND	ND	ND	ND	3.75 ± 0.06^{a}	4.33 ± 0.04^{b}	ND
90	2-Methoxy-4-vinylphenol	1329	MS/O/RI/STD	smoky	ND	ND	ND	ND	ND	1.25 ± 0.00^{b}	0.85 ± 0.01^{a}
91	Sesamol	1335	MS	burnt, salty aroma	ND	ND	ND	ND	ND	ND	2.36 ± 0.19^{a}
	Total phenols				**ND**	**ND**	**0.08 ± 0.01^{a}**	**0.72 ± 0.07^{b}**	**4.81 ± 0.10^{c}**	**7.18 ± 0.06^{e}**	**6.74 ± 0.11^{d}**
92	Isobutyraldehyde	< 700	MS/O	grilled meat	ND	ND	ND	ND	ND	ND	0.01 ± 0.00^{a}
93	3-Methyl-butanal	< 700	MS	fermented, sour	ND	ND	0.76 ± 0.02^{b}	0.97 ± 0.09^{c}	1.19 ± 0.02^{d}	0.56 ± 0.03^{a}	ND
94	2-Methyl-butanal	< 700	MS/O/STD	fruity	ND	ND	1.65 ± 0.06^{b}	2.74 ± 0.15^{c}	ND	ND	0.53 ± 0.01^{a}
95	Hexanal	903	MS/O/RI/STD	green, fruity	0.07 ± 0.01^{a}	0.06 ± 0.01^{a}	0.17 ± 0.01^{e}	0.14 ± 0.01^{d}	0.11 ± 0.01^{bc}	0.12 ± 0.01 ^{cd}	0.09 ± 0.01^{b}
96	(E)-2-heptenal	964	MS/O/RI/STD	green	ND	ND	0.04 ± 0.01^{a}	ND	ND	ND	ND
97	Benzaldehyde	966	MS/O/RI/STD	bitter, almond	ND	ND	0.10 ± 0.01^{a}	0.16 ± 0.01^{b}	0.83 ± 0.01^{c}	1.30 ± 0.06^{d}	ND
98	Benzeneacetaldehyde	1053	MS/O/STD	flowery	ND	ND	2.61 ± 0.10^{c}	0.83 ± 0.01^{b}	ND	0.10 ± 0.01^{a}	0.07 ± 0.02^{a}
99	2-Phenylpropionaldehyde	1100	MS/O	green, sweet	ND	ND	0.08 ± 0.01^{a}	0.18 ± 0.01^{b}	ND	ND	ND
100	Nonanal	1113	MS/O/RI/STD	green	0.03 ± 0.01^{b}	0.02 ± 0.01^{a}	0.08 ± 0.01^{c}	0.13 ± 0.01^{d}	ND	ND	ND
101	(*E,E*)-2,4-Decadienal	1292	MS/O/RI/STD	harshness	ND	ND	ND	ND	ND	ND	0.64 ± 0.01^{a}
102	Piperonal	1351	MS/O	wood, flower	ND	ND	ND	ND	0.43 ± 0.01^{a}	ND	ND

(*continued on next page*)

Table 1 (*continued*)

No.	Aroma-active compounds	RI [A]	Identification Method [B]	Odour Description [c]	Relative concentration (mg/kg) in oil after different pretreatment						
					RS	M2	M4	M6	M8	M10	R20
103	4-(1-Methylethyl)-benzaldehyde	1370	MS	spicy	0.02 ± 0.01^{b}	0.01 ± 0.00^{a}	0.01 ± 0.00^{a}	ND	ND	ND	ND
	Total aldehydes				**0.12 ± 0.01^{b}**	**0.09 ± 0.01^{a}**	**5.50 ± 0.09^{g}**	**5.15 ± 0.09^{f}**	**2.56 ± 0.06^{e}**	**2.08 ± 0.04^{d}**	**1.34 ± 0.13^{c}**
104	2,3-Pentanedione	< 700	MS/O/STD	caramel, butter	ND	ND	ND	0.39 ± 0.03^{b}	ND	ND	0.17 ± 0.01^{a}
105	2-Pentanone	< 700	MS/O	roasted	ND	ND	ND	ND	ND	0.51 ± 0.02^{b}	0.16 ± 0.01^{a}
106	2,3-Butanedione	< 700	MS	sour	ND	ND	ND	ND	ND	2.59 ± 0.22^{a}	ND
107	1-Cyclopropyl-ethanone	< 700	MS	medicinal	ND	ND	ND	ND	ND	0.56 ± 0.01^{a}	ND
108	3-Methyl-2-butanone	< 700	MS	peanut	ND	ND	ND	ND	ND	ND	2.33 ± 0.02^{a}
109	2-Butanone	< 700	MS	roasted	ND	ND	ND	ND	ND	ND	0.11 ± 0.01^{a}
110	3-Pentanone	702	MS/O	nutty, green	ND	ND	ND	ND	0.41 ± 0.02^{a}	ND	ND
111	3-Hexanone	766	MS	smoky	ND	ND	ND	ND	0.09 ± 0.01^{a}	0.09 ± 0.01^{a}	ND
112	Acetylacetone	782	MS/RI/STD	sesame oil, paste	ND	ND	ND	ND	ND	ND	0.03 ± 0.01^{a}
113	Acetophenone	1080	MS/O/STD	plastic, fruity	ND	ND	0.12 ± 0.01^{a}	ND	1.66 ± 0.01^{c}	ND	0.21 ± 0.02^{b}
	Total ketones				**ND**	**ND**	**0.12 ± 0.01^{a}**	**0.39 ± 0.03^{b}**	**2.16 ± 0.05^{c}**	**3.75 ± 0.06^{e}**	**3.01 ± 0.03^{d}**
114	1-Pentanol	875	MS/O/STD	fruity	ND	0.02 ± 0.01^{a}	0.15 ± 0.01^{b}	ND	ND	ND	ND
115	β-Vinyl-phenylethanol	943	MS	stinky	ND	ND	ND	ND	ND	ND	0.01 ± 0.00^{a}
116	1-Hexanol	974	MS/O	green, flower	ND	0.07 ± 0.01^{a}	ND	ND	ND	ND	ND
117	1-Octen-3-ol	990	MS/O/RI/STD	mushroom	ND	ND	0.13 ± 0.01^{a}	ND	ND	0.58 ± 0.01^{b}	ND
	Total Alcohols				**ND**	**0.09 ± 0.01^{b}**	**0.28 ± 0.01^{c}**	**ND**	**ND**	**0.58 ± 0.01^{d}**	**0.01 ± 0.00^{a}**
118	Acetic acid ethenyl ester	< 700	MS/O/STD	sweet	ND	ND	ND	0.77 ± 0.05^{a}	4.14 ± 0.02^{c}	2.73 ± 0.04^{b}	ND
119	(*E*)-2-octene	807	MS	smoky	ND	ND	ND	ND	ND	0.14 ± 0.01^{a}	ND
120	1,3-Dimethyl-benzene	874	MS/O/STD	medicinal	ND	0.06 ± 0.01^{a}	0.11 ± 0.01^{cd}	0.07 ± 0.01^{b}	0.27 ± 0.00^{e}	0.12 ± 0.01^{d}	0.10 ± 0.01^{c}
121	Dimethyl trisulfide	973	MS/O/RI/STD	sulphury	ND	ND	0.01 ± 0.00^{a}	0.09 ± 0.01^{b}	ND	ND	ND
122	(3*E*)-4,8-dimethylnona-1,3,7-triene	1232	MS	raw peanuts	0.02 ± 0.01^{b}	0.02 ± 0.01^{a}	ND	ND	ND	ND	ND
123	(*E*)-4-octene	799	MS	garlic, onion	ND	ND	ND	ND	ND	ND	0.02 ± 0.01^{a}

Note: For each row, values without any same superscript letter are significantly different ($p < 0.05$). "ND" indicates not detected.

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

[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.

trisulfide), 10 aldehydes, 7 ketones, 3 alcohols, 2 alkenes, 1 benzene, and 1 ester. Among these 98 compounds, 19 were newly found aroma-active compounds in sesame oil including 2-methyl-5-(1-methylethyl)-pyrazine, isopropenyl-pyrazine, 1-butyl-1H-pyrrole, 2-methyl-1H-pyrrole, 2-formyl-4,5-dimethyl-pyrrole, 1-ethyl-1H-pyrrole, 3-methyl-pyridine, 5-methyl-2-pyridinamine, 2,5-dimethyl-pyridine, 1-(2-furanyl)-1-pentanone, 5-methyl-2-furancarboxaldehyde, p-cresol, mequinol, 4-(1-methylethyl)-benzaldehyde, 3-hexanone, 2,3-butanedione, 1-cyclopropyl-ethanone, (*3E*)-4,8-dimethylnona-1,3,7-triene, and (E)-2-octene. In comparison, 70 aroma-active compounds were detected in the sesame oil extracted from hot-air roasted sesame seeds.

3.6.1. Aroma-active heterocyclics

Aroma-active heterocyclics including pyrazines, pyridines, pyrroles, furans, thiazoles, and thiophenes except 2-pentyl-furan were identified in the microwaved or hot-air roasted sesame oils but were absent from the cold-pressed sesame oil (Fig. 4, Table 1). Most heterocyclics are produced through the Maillard reaction, while 2-pentyl-furan was probably a product of lipid oxidation (Yin et al., 2021). The total concentration of aroma-active heterocyclic compounds initially rose from 0.02 to 125.12 mg/kg with increasing microwave time from 0 to 8 min and then decreased to 99.01 mg/kg with the extension of microwave time to 10 min ($p < 0.05$). This was probably because many heterocyclic compounds were generated and accumulated through the Maillard reaction, but they may be further polymerised to higher-molecular compounds at the late stage of the Maillard reaction when the heat was accumulated from prolonged microwaves (Zhou, Geng, Deng, Huang & Wang, 2019). The concentration of heterocyclic compounds in hot-air roasted sesame oil (R20) was 25.59 mg/kg, which was higher than the 4 min microwaved sesame oil (8.44 mg/kg) but lower than the 6–10 min microwaved sesame oils (42.40–125.12 mg/kg) ($p < 0.05$). These results suggest that appropriate microwave pretreatment may be more time efficient in generating heterocyclic aroma-active compounds in sesame oil than hot-air roasting, but excessive microwave might further reduce

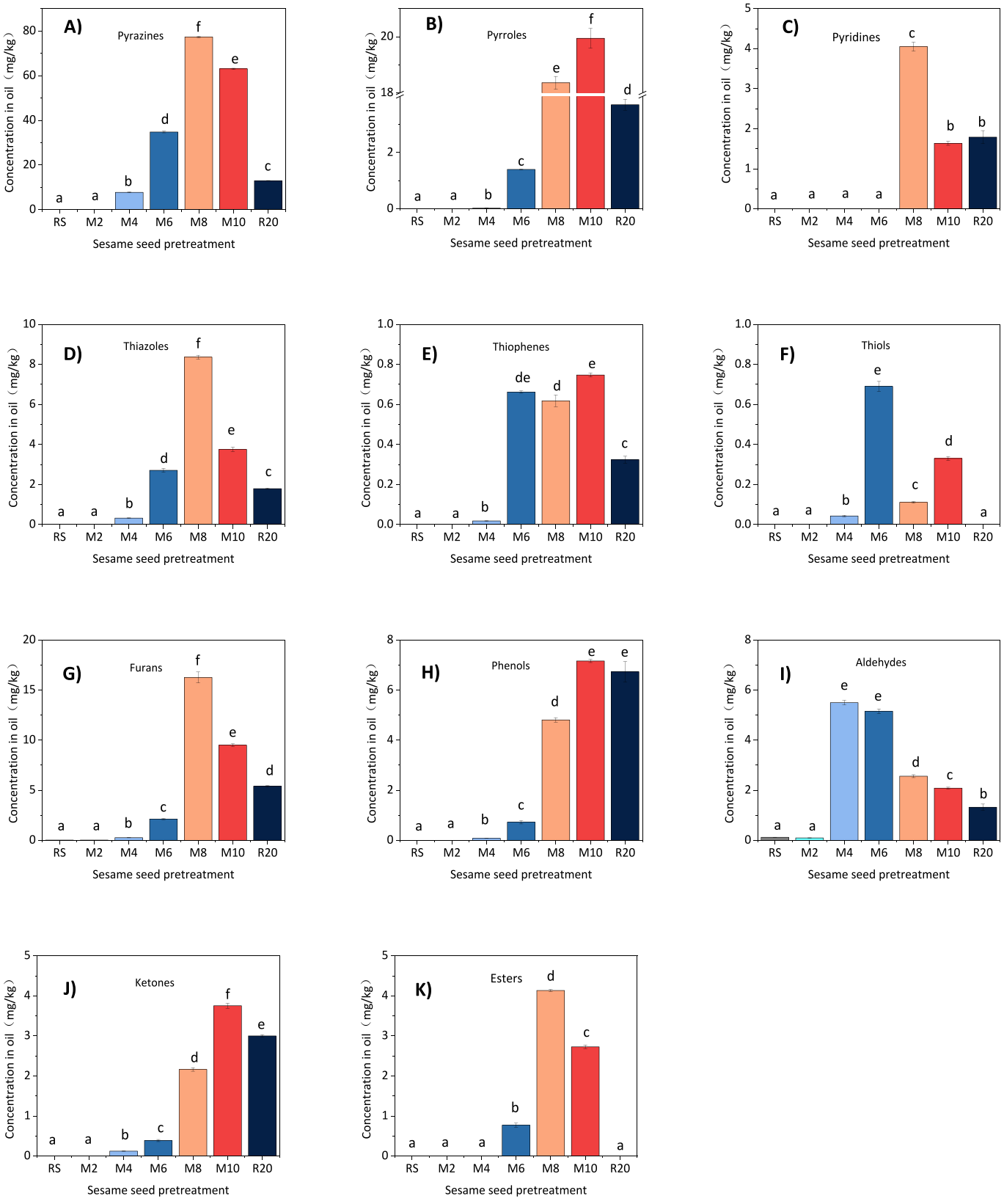

Fig. 4. Effect of different seed pretreatments on the sum of aroma-active compounds in different categaries.

the concentrations of heterocyclics in sesame oil.

The nitrogen-containing pyrroles, pyrazines, and pyridines are the products of the Maillard reaction generated between nitrogen-containing amino acids and sugars (Yin et al., 2021), which were characterised mainly by the roasted and nutty aromas (Table 1). Pyrazines were the richest odourants in sesame oils which were generated after 4 min microwave pretreatment, increased from 7.77 mg/kg to 77.34 mg/kg until 8 min, and dropped down to 63.10 mg/kg after 10

min microwave ($p < 0.05$) (Fig. 4**A**). Pyrazines have been identified as key odourants in roasted sesame oil (Yin et al., 2021). Tan et al. (2022) also reported that pyrazines in rapeseed initially increased upon moderate roasting, but reduced upon excessive roasting. The concentration of aroma-active pyrazines in hot-air roasted sesame oil (R20) was 12.95 mg/kg, which was higher than the 4 min microwaved sesame oil (M4, 7.77 mg/kg) but lower than the 6–10 min microwaved sesame oils (M6-M10, 34.81–77.34 mg/kg) ($p < 0.05$). A similar trend was observed in the concentration of pyrroles between different sesame oil samples (Fig. 4**B**). Aroma-active pyridines were only detected in the 8–10 min microwaved sesame oils (M8 and M10) and hot-air roasted sesame oil (R20), and the M8 sesame oil has the highest pyridines concentration ($p < 0.05$) (Fig. 4**C**).

The sulphury aroma-active compounds including thiophenes, thiazoles, and thiols (Fig. 4**D, E, F**, & Table 1) were the Maillard reaction products generated between the sulphury amino acids and reducing sugars, or the Strecker degradation products of sulphury amino acids (Jia et al., 2019), which are mainly characterised by the sulphury, meaty and onion-like aromas (Table 1). The sulphury aroma compounds appeared in sesame oil after ≥ 4 min microwave pretreatment. The total concentration of sulphury aroma compounds increased to the peak of 9.09 mg/kg in sesame oil after 8 min microwaves and dropped to 4.83 mg/kg after 10 min microwaves. The sum concentration of sulphury aroma compounds in the 20 min hot-air roasted sesame oil (R20) was 2.13 mg/kg, which was higher than the 4 min microwaved sesame oil (0.38 mg/kg) but lower than the 6–10 min microwaved sesame oils (4.07–9.09 mg/kg). Although sulphury aroma compounds are in trace amounts, they are very important to the overall flavour of sesame oil due to low odour thresholds (Yin et al., 2021).

The oxygen-containing furans are derived from lipid peroxidation or sugar caramelisation reactions (Liu et al., 2020), which were mainly described by the nutty, sweet, and caramel-like aromas (Table 1). Only one furan i.e. 2-pentyl-furan was present in the cold-pressed and 2 min microwaved sesame oils. The sum of aroma-active furans in sesame oils initially increased from 0.02 to 16.27 mg/kg with increasing microwave time until 8 min and dropped to 9.51 mg/kg after 10 min microwaves (Fig 4**G**). The sum of aroma-active furans in the 20 min hot-air roasted sesame oil was 5.02 mg/kg, which was higher than that in the 6 min microwaved sesame oil (M6) but lower than the 8–10 min microwaved sesame oils (M8-M10) ($p < 0.05$).

3.6.2. *Aroma-active phenolic compounds*

No aroma-active phenolic compound (smoky aroma) was present in the cold-pressed sesame oil (RS) until more than 4 min microwave pretreatment. Aromatic phenolic compounds may be produced through the thermal degradation of carboxylic acid phenols or lignins present in sesame seeds after roasting (Manley, Vallon, & Erickson, 2010; Yin et al., 2021). The total concentration of aroma-active phenolic compounds increased continuously from 0.08 mg/kg to 7.18 mg/kg with the extension of microwave time ($p < 0.05$) (Fig. 4**H**). The sum of aroma-active phenolic compounds in the 20 min hot-air roasted sesame oil (R20, 6.74 mg/kg) was not statistically different from that in the 10 min microwaved sesame oil (M10).

3.6.3. *Aroma-active aldehydes, ketones and esters*

The sum of aroma-active aldehydes in the sesame oils increased dramatically from 0.09 to 5.50 mg/kg with the initial increase of microwave time from 0 to 4 min (Fig. 4**I**), and then gradually decreased to 2.08 mg/kg after 10 min microwaves. The sum of aroma-active aldehydes in the 20 min hot-air roasted sesame oil was 1.34 mg/kg, lower than that in the 4–10 min microwaved sesame oils ($p < 0.05$). Most aldehydes in sesame oil are believed as the products of lipid oxidation or the Maillard reaction. Specifically, hexanal and nonanal (green and fruity aromas) were reported as the oxidation products of linoleic acid and oleic acid, respectively (Jia et al., 2019). 3-Methyl-butanal, 2-methyl-butanal (fermented and fruity aromas), and benzaldehyde (almond aroma) were believed the products of Strecker degradation of amino acids (Jia et al., 2019; Yin et al., 2021).

The total amount of aroma-active ketones in sesame oils increased continuously from 0 to 3.75 mg/kg with the extension of microwave time (Fig. 4**J**). Specifically, acetophenone was detected in sesame oil after 4 min microwave, which was reported as a product of Strecker degradation of phenylalanine (Zhou et al., 2019). 2,3-Butanedione was believed a Maillard reaction product (Yin et al., 2021). 2-Pentanone and 3-pentanone are the products of lipid oxidation (Jia et al., 2019). The sum of ketones in hot-air roasted sesame oil was higher than that in the 8 min microwaved sesame oil but lower than the 10 min microwaved sesame oil ($p < 0.05$). Esters are generated through esterification of alcohols with free fatty acids (Yin et al., 2022). Only one aroma-active ester, i.e. acetic acid ethenyl ester (sweet aroma), was detected in sesame oils extracted from 6 to 10 min microwaved seeds which was not detected in the hot-air roasted sesame oil.

3.7. *Impact of microwaves on the sensory perception of sesame oil*

The sensory profiles of all sesame oil samples are shown in Fig. 5. There was a significant sample effect on all sensory attributes ($p < 0.05$), and no significant interaction effect between sample and panellist, suggesting that the performance of the panel was adequate. The sensory profile of the cold-pressed sesame oil was characterised by strong raw sesame seed, earthy, and green aromas. The perceived intensities of these 3 sensory attributes were reduced in the 2 min microwaved sesame oil ($p < 0.05$) and disappeared in the 4–10 min microwaved sesame oils. With the increase of microwave time from 4 to 8 min, the perceived intensities of the roasted sesame seed, nutty, caramel-like, burnt, smoky aromas, and bitter taste gradually increased ($p < 0.05$). This was generally consistent with the changes in the concentrations of aroma-active pyrazines (roasted and nutty aroma), pyridines (nutty aroma), furans (caramel-like aroma), and phenolic compounds (smoky aroma) in sesame oils (Fig. 4). The sensory profile of the sesame oil extracted from 6 min microwaved sesame seeds was characterised by stronger perceived intensities of the roasted sesame seed, nutty, and caramel aromas while weaker burnt, smoky aromas, and bitter taste than the sesame oil extracted from the 20 min hot-air roasted sesame seeds ($p < 0.05$). After 8 min microwave pretreatment, the perceived roasted sesame seed, nutty, caramel, burnt and smoky aroma, and the bitter taste became intense in sesame oil. The sensory profile of 10 min microwaved sesame oil was dominated by a strong bitter taste, and strong burnt and smoky aromas which may not be desired by consumers, while the

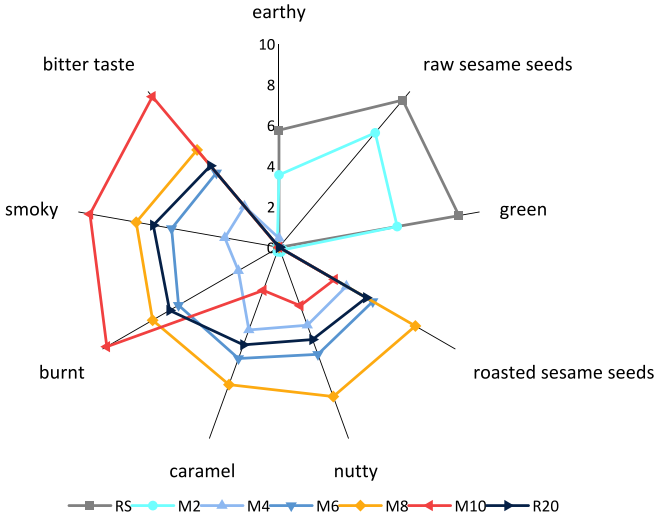

Fig. 5. Effect of different seed pretreatments on the sensory characterisation of sesame oils.

desirable caramel, nutty, and roasted sesame seed aromas reduced dramatically to mildly perceived intensities (Yin et al., 2020). The smoky, burnt aromas and bitter taste may be excessively accumulated at the late stage of the Maillard reaction upon excessive seed roasting (Yin et al., 2021). It suggests that the optimal microwave pretreatment was at 900 W for 6 min which produced the sesame oil with a better sensory profile than the hot-air roasting, while excessive microwave pretreatment (8–10 min, 900 W) compromised the sensory quality of sesame oil.

3.8. Impact of microwaves on harman, norharman, and benzo(a)pyrene of sesame oil

Heterocyclic amines (HCA) especially harman and norharman are recognised as the "Group 2B" carcinogens by the IARC of WHO and they are believed to be the Maillard reaction products (Ji et al., 2019). Harman and norharman were not detected in the cold-pressed sesame oil (Fig. 3**E & 3F**), increased with increasing microwave time, and reached 775.19 ng/g and 1069.99 ng/g, respectively, after 10 min microwave pretreatment. In comparison, the concentrations of harman and norharman in the sesame oil after hot-air roasting were 1319.85 ng/g and 1168.40 ng/g, respectively, which were higher than all microwave pretreatments ($p < 0.05$).

Benzo(a)pyrene is a well-known "Group 1" carcinogen that is often detected in oils. It may be transferred to oil through the environment or formed via pyrolysis and polymerization of lipids during the thermal process (Ji et al., 2019; Ji, Liu & Wang, 2020). Benzo(a)pyrene was detected in the sesame oils after 6–10 min microwaves (0.98–1.59 μg/kg), which was lower than the benzo(a)pyrene in hot-air roasted sesame oil (1.83 μg/kg) ($p < 0.05$). Benzo(a)pyrene in all sesame oils was within the limit of EC No 835: 2011 (2 ng/g).

4. Conclusion

Appropriate application of microwave had clear advantages over traditional hot-air roasting and our hypothesis was confirmed. The oil extraction rate of sesame seed was significantly improved by microwaves probably due to the increased degree of seed porosity and cell structure disruption. Meanwhile, microwaves showed higehr maintaining γ-tocopherol in sesame oil than hot air. Microwaves accelerated the generation of many important thermal-reaction products including aroma-active heterocyclics, aldehydes, ketones, and phenolic compounds, and facilitated the transformation of sesamin and sesamolin to sesamol in sesame oil. The optimal microwave settings suggested by the current study were at 900 W for 6 min because it generated intense flavour with the most preferred sensory quality while maintaining high bioactive compounds and low carcinogenic components. However, excessive microwave pretreatment (900 W, 10 min) caused a further decrease in aroma-active heterocyclics and γ-tocopherol and increased the perception of undesirable sensory attributes including a strong bitter taste and a burnt flavour. The future application of microwaves in sesame oil production, as a greener alternative to hot air, is therefore very promising. Further scale-up study is necessary to investigate the application and feasibility of microwaves in large-scale production in the real sesame oil industry.

CRediT authorship contribution statement

Wen-ting Yin: Conceptualization, Funding acquisition, Supervision, Writing – original draft, Writing – review & editing. **Chen-jia Yang:** Project administration, Writing – review & editing. **Xin-yun He:** Data curation. **Yu-hang Zhao:** Formal analysis, Investigation, Methodology, Project administration, Writing – original draft. **Hua-min Liu:** Funding acquisition, Supervision. **Zhuo-qing Zhai:** Formal analysis. **Xue-de Wang:** Funding acquisition, Supervision.

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.

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