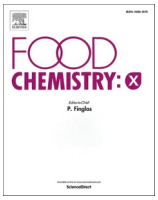
Food Chemistry: X 22 (2024) 101381

Contents lists available at ScienceDirect

Food Chemistry: X

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



Microwave pretreatment effects on the aroma precursors, sensory characteristics and flavor profiles of fragrant rapeseed oil

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ARTICLE INFO

Keywords:
Microwave pretreatment
Fragrant rapeseed oil
Lipid oxidation
Maillard reaction
Glucosinolate degradation

ABSTRACT

Microwave technology offers a rapid and uniform heating method. This study investigated how microwave pretreatment affects the aroma precursors and flavor of fragrant rapeseed oils (FROs). Microwave pretreatment led to decreased levels of polyunsaturated fatty acids, sugars, protein-bound amino acids, and glucosinolates. Using gas chromatography–mass spectrometry, we identified 66 volatile compounds in the oil samples. Among these, based on odor activity values (OAV $\geq$ 1), we found 9 aldehydes, 1 ketone, 6 pyrazines, 1 isothiocyanate, and 7 nitriles as the key aroma-active compounds, contributing fatty-like, nutty-like, and pungent-like odors, respectively. The electronic nose results highlighted W5S and W1W as primary sensors for determining the flavor profiles of FROs. Notably, aroma-active pyrazines exhibited strong negative correlations with sucrose, cysteine, lysine, and isoleucine. This research provides essential insights for enhancing the aroma of FROs.

1. Introduction

Rapeseed (*Brassica napus* L.) is a significant contributor to the global vegetable oil market and ranks third in importance after palm and soybean. It accounts for approximately 12% of total oil production worldwide (Zheng & Liu, 2022). Annual global rapeseed oil production reached 27.98 million metric tons between 2019 and 2020 (Zhang et al., 2021). Rapeseed oil is notable for its higher levels of oleic acid (C18:1) and linoleic acid (C18:2) than other fatty acids and thus offers superior nutritional value (Liang et al., 2023). Furthermore, rapeseed oil is a rich source of various active ingredients, including tocopherols, polyphenols, phytosterols, and carotenoids (Tan et al., 2022; Zhang et al., 2022).

Flavor is a critical sensory attribute that plays a significant role in determining consumers' acceptance of rapeseed oil (Zhang et al., 2021; Zhang, Cao, & Liu, 2020). Several factors can influence the flavor profiles of rapeseed oil, including rapeseed variety, processing methods, degumming methods, and storage conditions (Liang et al., 2023). Cold pressing and hot pressing are the two commonly used extraction methods for vegetable oils and differ mainly in terms of heating pretreatment. Fragrant rapeseed oil (FRO) is a type of hot-pressed rapeseed oil that accounts for >30% of the rapeseed oil consumption in China. Previous studies have shown that moderate heat pretreatment can enhance the oxidative stability of FRO (Shrestha & De Meulenaer, 2014; Tan et al., 2022). Furthermore, >100 aroma-active compounds have been identified in FROs (Zhang et al., 2021). Consequently, heat pretreatment can improve the nutritional and sensory qualities of FRO. Roasting, as a traditional pretreatment method, is widely employed in the processing of FRO. Typically, rapeseed plants are roasted at temperatures ranging from 150 to 180 °C for durations ranging from 10 to 60 min in industrial settings (Yu, Wang, Zhang, Liu, & Li, 2021; Zhang et al., 2021). However, rapeseed plants are sensitive to heat, and excessive heat can potentially degrade the quality of FRO. Heterocyclic amines and polycyclic aromatic hydrocarbons, which are known carcinogens, can form during the roasting of oilseeds at high temperatures (Zhang et al., 2020). Moreover, this method often requires a lengthy processing time and involves substantial energy consumption (Yin et al., 2022).

Recently, there has been growing interest in the development of microwave pretreatment due to its numerous advantages. These include reduced energy consumption, higher heating rates, and improved

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https://doi.org/10.1016/j.fochx.2024.101381
Received 28 November 2023; Received in revised form 14 February 2024; Accepted 9 April 2024
Available online 12 April 2024

product quality (Musa Ozcan & Uslu, 2023). Unlike roasting, microwave heating converts electromagnetic energy into thermal energy instead of transferring heat (Yin et al., 2022). Microwave energy can quickly penetrate oilseeds, allowing for the generation of desirable flavors and minimizing nutrient loss in pressed oils (He, Wu, & Yu, 2021; Jia et al., 2023). During heat processing, various complex chemical reactions occur within rapeseed plants, including lipid oxidation, the Maillard reaction, glucosinolate (GSL) degradation, and amino acid degradation (Yang et al., 2022). The oxidation of fatty acids, particularly unsaturated fatty acids, produces degradation products such as hexanal and (E,E)-2,4-heptadienal, which contribute to fatty and green odor notes (Kiralan & Ramadan, 2016). The Maillard reaction, on the other hand, results in the production of compounds such as 2,5-dimethylpyrazine and 3-ethyl-2,5-dimethylpyrazine, which contribute to nutty and roasted aromas (Wei et al., 2012; Zhou et al., 2019). Additionally, thermal degradation of GSLs can yield compounds such as 2,4-pentadienenitrile and 4-isothiocyanato-1-butene, which are associated with pungent and spicy flavor characteristics (Zhang et al., 2021).

Previous studies have focused mainly on the effect of microwave pretreatment on the flavor of FROs. However, there is insufficient literature available regarding the association between aroma precursors in different rapeseeds and the flavor profiles of extracted FROs during microwave heating.

The main objective of this research was to achieve the following:

1. Explore the effect of microwave pretreatment on the flavor profiles of FROs, considering both qualitative and quantitative aspects via gas chromatography–mass spectrometry (GC–MS) and an electronic nose (E–nose).
2. Identify the aroma-active compounds in the FROs through odor activity value (OAV $\geq$ 1) calculations.
3. Investigate the relationships between fatty acid compositions (FACs), amino acids, sugars, GSLs, and key volatile compounds under microwave treatment.

By obtaining a deeper understanding of the flavor profiles of FROs prepared using microwave heating and the relationships between these profiles and their precursor components, we aim to provide valuable insights and a research foundation for flavor-focused production of FROs.

2. Materials and methods

2.1. Materials

Currently, double-low (low erucic acid and low GSLs) rapeseed comprises approximately 90% of the commercial rapeseed industry and is generated through selective breeding and the use of high-quality rapeseed varieties (Xiao et al., 2022). However, China, a major rapeseed producer, still has a significant proportion of rapeseed plants with high levels of erucic acid and GSLs (Zhang et al., 2022). Consequently, this study utilized two types of high-generation inbred lines of seeds as experimental materials. One variety, sample 1 (LELG), exhibited low erucic acid (5.06%) and low GSLs (11.33 μmol/g), while sample 2 (HEHG) displayed high erucic acid (44.55%) and high GSLs (192.12 μmol/g). The seeds were sourced from the Hybrid Rape Research Center of Shaanxi Province, China.

For the analysis, 2-octanol (purity >99%) and *n*-alkanes (C7–C30) of chromatographic grade were obtained from Sigma–Aldrich (Shanghai, China). Sucrose, glucose, fructose, raffinose, stachyose, galactose, and 5-hydroxymethylfurfural (HMF) standards (purity >98%) were also obtained from Sigma–Aldrich. The free amino acid mixed standard was obtained from Supelco (Bellefonte, PA, USA).

2.2. Sample preparation

Microwave heating was carried out based on a previously published method (He et al., 2021). In this process, rapeseeds (100 g) were placed in an 18.5 cm diameter Pyrex® glass Petri dish and roasted in a microwave oven (Midea, MG823LA3-NR, China) at 800 W for various durations (0, 2, 4, 6, and 8 min). Subsequently, the roasted seeds were allowed to cool naturally to room temperature. One portion was powdered, while the other part was pressed using a pressing machine. Finally, the free radicals and oxidation products (FROs) were collected through centrifugation at 2191 $\times g$ for 15 min and stored at −20 °C until further use.

2.3. Determination of chemical parameters

The peroxide value (PV), acid value (AV), *p*-anisidine value (*p*-AnV), conjugated dienes (K232), and conjugated trienes (K268) were measured according to the American Oil Chemists' Society (AOCS) official methods Cd 8b-90, Cd 3d-63, Cd 18–90, and Ch 5–91, respectively.

2.4. FAC analysis

The fatty acids were converted into their corresponding fatty acid methyl esters (FAMEs) using the method described by Zhang, Akhymetkan, Chen, Dong, et al. (2022). The analysis of FAC in FRO was performed using an Agilent gas chromatography (GC) system (6890 N, Agilent Technologies, USA) equipped with an autosampler (7683, Agilent Technologies, USA). The system was equipped with an HP-INNOWAX capillary column (30 m × 0.25 mm i.d., 0.25 μm f.t.) and a flame ionization detector. The chromatographic conditions used were the same as those previously described in our publication (Chen et al., 2023). The column temperature was set as follows: initial temperature of 60 °C was increased to 200 °C at a rate of 10 °C/min, increased to 240 °C at a rate of 8 °C/min, and held at this temperature for 15 min. The flow rate was 1.3 mL/min. The temperature of the detector and injector was set at 250 °C. Fatty acids were identified by comparison with a standard FAME mixture (C4-C24, Supelco) and quantified using C19:0 methyl ester as an internal standard.

2.5. Determination of sugars

The quantification of sugar contents was carried out using the method described by Sen and Gokmen (2022). Briefly, 1.0 g of sample was homogenized with 15 mL of distilled water and then centrifuged at 7000 $\times g$ for 15 min. The supernatants were filtered through 0.45 μm PTFE syringe filters and transferred to chromatography vials, which were subsequently sealed. The analysis of sugars was performed using a high-performance liquid chromatography (HPLC) system (Waters 2695, Waters, USA) equipped with a refractive index detector (Waters 2414, Waters, USA). Chromatographic separation was achieved using an Aminex HPX-87H anion-exchange column (Bio-Rad, Hercules, USA). Isocratic elution was performed with 2.5 mM sulfuric acid at a flow rate of 0.6 mL/min. The concentration of each sugar was determined using external calibration curves and is expressed as mg/100 g of raw material.

2.6. Analysis of protein-bound aids

The analysis of protein-bound amino acids was conducted according to the literature with some modifications (Spain & Funk, 2022). For acid hydrolysis, 50 mg of pulverized powder was weighed, placed in a glass tube and then treated with 5 mL of hydrochloric acid at a concentration of 6 M. The mixture was immersed in an oil bath at 110 °C for 24 h. The resulting mixture was filtered and concentrated by evaporation at 60 °C. After concentration, the residue was diluted with 10 mL of sodium

citrate buffer (pH 2.2). Finally, the amino acid composition was determined using an amino acid analyzer (Biochrom 30, Biochrom, UK).

2.7. Measurement of HMF

The HMF levels in rapeseeds were detected using the method established by Zhang et al. (2022). A precise amount of 2.0 g of powder sample was weighed into a 10 mL centrifuge tube. To this mixture, 4 mL of ethyl acetate was added, and the mixture was vortexed for 1 min and then centrifuged at 2540 ×*g* and 4 °C for 10 min. This extraction process was repeated three times. After centrifugation, the supernatants were combined and concentrated using a rotary evaporator. Then, 1 mL of deionized water was added to the extract, and the mixture was vigorously mixed and subjected to centrifugation to remove the upper layer. The remaining volume was filtered through a 0.45 μm filter. The filtered extract was injected into an Agilent 1260 HPLC (Santa Clara, CA, USA) system equipped with a UV detector and an autosampler. Chromatographic separations were performed on a C18 column (Waters BEH C18, 150 × 2.1 mm, 1.7 μm). The measurement was conducted at 284 nm, and quantification was performed using a calibration curve in the range of 0.1–200 mg/mL.

2.8. Quantification of GSLs

A 200 g portion of the defatted sample was extracted by boiling in 70% methanol (*v*/v) under reflux. The extract was applied to a DEAE-Sephadex A-25 column and eluted with 3 mL of distilled water. Subsequently, desulfation was carried out by adding 1 mL of purified sulfatase solution and incubating the column for 16 h at 35 °C. The desulfated GSLs were then washed with 5 mL of distilled water and freeze-dried. GSL determination was conducted according to our previously published method (Zhang, Chen, Zhao, Wang, & Yu, 2022). An Agilent 1100 series HPLC system equipped with a SinoChrom ODS-BP column (10 mm × 250 mm, 5 μm) was used for chromatographic analysis. The quantification of GSLs was performed using sinigrin as an internal standard, and the concentrations were adjusted according to the relative response factors.

2.9. Volatile component analysis

Volatile compound identification was performed using headspace-solid phase microextraction (HS-SPME) combined with GC–MS, as previously described (Zhang, Chen, Zhao, Chen, et al., 2022). Briefly, 4 g of the oil sample was placed in a 20 mL headspace vial along with 4 μL of 2-octanol (0.49 mg/mL) as an internal standard. The sample vials were equilibrated in an incubator at 50 °C for 30 min with agitation at 500 rpm. Subsequently, a 50/30 μm divinylbenzene/carboxen/polydimethylsiloxane (DVB/CAR/PDMS) fiber (Supelco, Bellefonte, PA, USA) measuring 1 cm in length was inserted into the headspace for 30 min. Afterward, the SPME fiber was thermally desorbed by placing it in a GC–MS injector set at 250 °C for 3 min. A The analysis was conducted using a Shimadzu QP2010 GC–MS system (Kyoto, Japan) and a DB–17MS column (60 m × 0.25 mm ID, 0.25 μm film thickness). Mass spectra were obtained in profile mode with a mass range of *m/z* 35 to m/z 500. The qualitative identification of volatile compounds was based on the retention indices of the reference standards and mass spectra matching using the NIST mass spectral library (NIST14, Gaithersburg, MD, USA). The quantification of volatile compounds was performed by comparing their peak areas to that of the internal standard compound. Compounds with ≤85% similarity to the NIST library were excluded.

2.10. E–nose analysis

The volatile fingerprint of the FROs was analyzed using a portable *E*-nose system PEN3 (Win Muster Airsense Analytics, Inc., Schwerin, Germany), following the method of Xu, Yu, Liu, and Zhang (2016). The device is equipped with a sensor array unit consisting of ten metal oxide semiconductors (Table S1). A 1 g sample of FRO was placed in a 20 mL headspace sample bottle and immediately capped. The sample headspace was then pumped over the sensor surfaces at a flow rate of 400 mL/min. The detection time was 60 s, during which the sensor signals were recorded.

2.11. Sensory evaluation

Descriptive sensory analysis of FRO was conducted using a protocol based on our previous research (Zhang, Chen, Zhao, Chen, et al., 2022). The sensory evaluation panel included 10 individuals (5 males and 5 females) aged between 22 and 31 years, all of whom had undergone sensory evaluation training. An aliquot of 20 g of each sample was placed in a 50 mL glass vial labeled with a random three-digit number and sealed with a plastic lid. The following odor descriptors were evaluated for the oil sample: nutty-like, burnt-like, pickled-like, pungent-like, green-like, and fatty-like. The intensity of each aroma attribute was scored on a 10-point scale (0 = very low intensity, 10 = strong intensity). These experiments were conducted on rapeseed oils obtained from raw and microwaved seeds in accordance with national regulations (China); thus, no further ethical permission was needed.

2.12. Statistical analysis

The experiments were conducted three times on different occasions. Comparisons were performed using one-way ANOVA followed by Duncan's multiple range test ($p < 0.05$) in SPSS version 11.5. Principal component analysis (PCA) was also conducted using Minitab (version 19.0). The effects of rapeseed variety and time on sensory evaluation were also analyzed by two-way ANOVA using SPSS software.

3. Results and discussion

3.1. Changes in chemical parameters during microwave heating

PV was utilized to identify the primary lipid oxidation products of oils, particularly hydroperoxides (Zhang et al., 2023). As depicted in Fig. S1a, the PV of all the samples exhibited a general trend of initial increase followed by a decrease. This trend could be primarily attributed to the instability of hydroperoxides, leading to their further decomposition into low-molecular-weight molecules such as aldehydes, ketones, and acids (Chen et al., 2023).

The AV serves as a crucial parameter for assessing oil deterioration, as it quantifies the content of free fatty acids (FFAs). FFAs are generated primarily through triglyceride hydrolysis and partly through hydroperoxide degradation (Ahmad Tarmizi, Niranjan, & Gordon, 2013). In Fig. S1b, changes in the AV in FROs during microwave heating are illustrated. With prolonged treatment time, the AV of the FROs gradually increased. After microwave treatment, the AVs of the FROs obtained from the LELG and HEHG were 1.10 and 0.92 mg/g, respectively.

p-AnV signifies the production of aldehydes, predominantly 2-alkenals and 2,4-alkadienals, which arise from hydroperoxide decomposition (Zhao, Zhang, Wang, & Devahastin, 2021). As shown in Fig. S1c, the initial *p*-AnVs in the FROs decreased and then significantly increased with prolonged usage time. By the end of the treatment, the *p*-AnVs of oils obtained from LELG and HEHG increased to 6.24 and 23.88 units, respectively. This notable difference can be attributed to the susceptibility of oils rich in polyunsaturated fatty acids (PUFAs) to degradation during heating (Zhang et al., 2022).

K232 represents the degree of primary oxidation product formation, while K268 quantifies the levels of carbonyl compounds, particularly ketones (Li et al., 2021). The effects of microwave pretreatment on K232 and K268 in the FROs are depicted in Fig. S1d-e. The levels of K232 and K268 in the oils of both sample groups increased with prolonged microwave time. These findings further indicate that microwave heating

Table 1
Changes in FAC of FROs during microwave heating.

Rapeseed varieties	Fatty acid composition (g/100 g)	Time (min)				
		0	2	4	6	8
LELG	C16:0	4.04 ± 0.02c	4.02 ± 0.01c	4.02 ± 0.01c	4.17 ± 0.02b	4.59 ± 0.01a
	C18:0	2.23 ± 0.12c	2.43 ± 0.03bc	2.47 ± 0.10bc	2.66 ± 0.06b	3.82 ± 0.04a
	C18:1	60.16 ± 4.13a	61.95 ± 1.22a	62.41 ± 3.51a	63.13 ± 2.17a	64.96 ± 1.34a
	C18:2	17.00 ± 1.31a	16.58 ± 0.58a	16.2 ± 0.61a	15.59 ± 1.10a	14.31 ± 0.93a
	C18:3	10.28 ± 1.21a	9.81 ± 1.13a	9.74 ± 0.46a	7.58 ± 1.01ab	6.07 ± 0.72b
	C22:1	5.06 ± 0.23a	4.86 ± 0.16a	4.73 ± 0.08a	4.61 ± 0.11a	4.53 ± 0.07a
HEHG	C16:0	2.91 ± 0.03d	2.98 ± 0.06 cd	3.06 ± 0.01c	3.29 ± 0.05b	3.74 ± 0.02a
	C18:0	1.22 ± 0.13b	1.31 ± 0.27b	1.54 ± 0.10b	2.37 ± 0.06a	2.58 ± 0.08a
	C18:1	23.01 ± 1.50c	24.66 ± 1.63c	28.97 ± 1.31bc	34.27 ± 2.15ab	38.82 ± 1.44a
	C18:2	9.55 ± 0.29a	9.23 ± 0.46ab	8.37 ± 0.22b	7.22 ± 0.14c	5.13 ± 0.05d
	C18:3	16.45 ± 2.02a	16.04 ± 1.72a	13.27 ± 0.37ab	11.25 ± 1.11ab	10.05 ± 1.24b
	C22:1	44.55 ± 1.81a	44.07 ± 1.20a	43.23 ± 1.33a	40.16 ± 2.47a	39.21 ± 1.52a

Results are presented as means ± SD. Values with different superscript letters superscript in the same row are significantly different between groups ($p < 0.05$). *Abbreviations:* FRO, fragrant rapeseed oil; FAC, fatty acid composition.

can facilitate the accumulation of lipid oxidation products.

3.2. Effect of microwave pretreatment on the FACs

Table 1 displays the FAC results of the unroasted and microwave roasted FROs. Noticeable differences were observed in the FAC of the analyzed oils. The primary fatty acids in the raw LELG oil sample were oleic (60.16%), linoleic (17.00%), and linolenic (10.28%) acids. In contrast, the dominant fatty acids in the raw HEHG oil sample were oleic (23.01%), linoleic (9.55%), linolenic (16.45%), and erucic (44.55%) acids. Additionally, palmitic and stearic acids were present in small amounts in all the samples. The FAC experienced slight changes with increasing microwave time. Linoleic and linolenic acids decreased slightly, while the contents of palmitic, stearic, and oleic acids increased in FROs after microwave roasting at 800 W for 8 min. Previous research has also noted microwave-induced alterations in the FAC of Nigella seed oil, specifically a slight decrease in PUFA levels and an increase in saturated fatty acid concentration (Suri, Singh, & Kaur, 2022). PUFAs are sensitive to high temperatures and are easily oxidized, which may account for the changes in FAC observed in the oil samples following microwave treatment (Zhang, Chen, Zhang, Sagymbek, et al., 2022).

3.3. Effect of microwave pretreatment on the sugars

Changes in sugar levels were monitored in microwave-roasted rapeseeds (Table 2). Two predominant sugars, sucrose (53.81 mg/100 g in LELG, 39.42 mg/100 g in HEHG), stachyose (19.43 mg/100 g in LELG, 12.24 mg/100 g in HEHG), and raffinose (5.04 mg/100 g in LELG, 3.46 mg/100 g in HEHG), were identified among the rapeseed varieties. Furthermore, their levels in LELG were significantly higher than those in HEHG ($p < 0.05$). Trace amounts of fructose, glucose, and galactose were also detected in the samples. The concentrations of sucrose, stachyose, and raffinose tended to decrease rapidly with increasing microwave time. >30 % of these sugars were lost during the microwave heating process. Since these sugars act as direct precursors of HMF in nuts and seeds, their abundance is expected to decrease with extended roasting time (Sen & Gokmen, 2022).

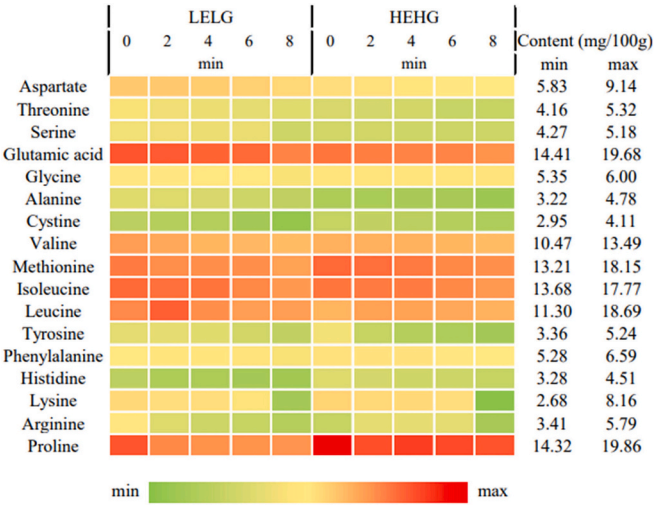

Fig. 1. Changes in contents of protein-bound amino acids in two rapeseeds during microwave heating.

Table 2
Changes in contents of sugars in two rapeseeds during microwave heating.

Rapeseed varieties	Time (min)	Glucose	Fructose	Sucrose	Raffinose	Stachyose	Galactose
LELG	0	0.35 ± 0.03Ab	0.45 ± 0.05Bc	53.81 ± 0.02Aa	5.04 ± 0.03Aa	19.43 ± 0.16Aa	0.02 ± 0.01Ac
	2	0.33 ± 0.02Ab	0.34 ± 0.03Cd	47.39 ± 0.04Bb	3.38 ± 0.01Bbc	16.64 ± 0.01Bb	–
	4	0.32 ± 0.03Ab	0.31 ± 0.00Cd	46.45 ± 0.02Cc	3.26 ± 0.06Bc	16.30 ± 0.03Cc	–
	6	0.18 ± 0.01Bc	0.47 ± 0.01Bc	36.41 ± 0.13Dg	3.05 ± 0.03Cd	15.28 ± 0.02Dd	–
	8	0.36 ± 0.04Ab	0.60 ± 0.03Ab	23.53 ± 0.11Eh	2.88 ± 0.01De	13.60 ± 0.02Ee	–
HEHG	0	0.56 ± 0.03Aa	0.33 ± 0.02Cd	39.42 ± 0.04Ad	3.46 ± 0.03Ab	12.24 ± 0.02Af	0.37 ± 0.01Aa
	2	0.33 ± 0.01Bb	0.11 ± 0.01De	39.41 ± 0.04Ad	3.35 ± 0.10Abc	11.40 ± 0.06Bg	0.23 ± 0.02Bb
	4	0.27 ± 0.02Bb	0.08 ± 0.01De	39.08 ± 0.13Be	2.81 ± 0.02Bef	11.20 ± 0.03Cg	–
	6	0.33 ± 0.01Bb	0.51 ± 0.04Bc	37.26 ± 0.08Cf	2.69 ± 0.04Bf	9.70 ± 0.08Dh	–
	8	0.57 ± 0.05Aa	1.02 ± 0.02Aa	14.39 ± 0.06Di	1.21 ± 0.02Cg	5.23 ± 0.05Ei	–

Results are presented as means ± SD. Column values in the same rapeseed variety with the different capital letters are significantly different ($p < 0.05$); column values in the different rapeseed varieties with different lowercased letters are significantly different ($p < 0.05$); "-", non-detectable.

Table 3
Changes in contents of GSLs in two rapeseeds during microwave heating.

Rapeseed varieties	Time (min)	Progoitrin	Epi-progoitrin	Glucoraphanin	Gluconapoleiferin	Gluconapin	4-Hydroxyglucobrassicin	Glucobrassicanapin	Glucotropaeolin	Glucobrassicin	Gluconasturtiin	4-Methoxyglucobrassicin
LELG	0	2.20 ± 0.02Ae	–	–	–	3.06 ± 0.08Ae	4.28 ± 0.13Ab	0.39 ± 0.01Af	0.53 ± 0.03Ae	0.36 ± 0.04Aa	0.96 ± 0.06Ab	0.24 ± 0.03Acd
	2	2.22 ± 0.01Ae	–	–	–	2.59 ± 0.04Be	3.98 ± 0.11Ac	0.40 ± 0.06Af	0.50 ± 0.01Aed	0.32 ± 0.02ABab	0.77 ± 0.02Bc	0.19 ± 0.01Ade
	4	2.12 ± 0.03Be	–	–	–	2.46 ± 0.02Be	3.24 ± 0.02Be	0.23 ± 0.03Bf	0.46 ± 0.06Aede	0.24 ± 0.01Bc	0.35 ± 0.05Cd	0.15 ± 0.05Ae
	6	1.22 ± 0.00Ce	–	–	–	1.87 ± 0.05Ce	1.40 ± 0.13Cf	–	0.41 ± 0.02Ade	0.15 ± 0.02Cd	–	–
	8	–	–	–	–	0.89 ± 0.01De	0.05 ± 0.01Dg	–	0.07 ± 0.01Bf	–	–	–
HEHG	0	96.89 ± 2.21Aa	2.57 ± 0.10Aa	2.76 ± 0.03Aa	1.73 ± 0.05Aa	70.25 ± 1.31Aa	4.62 ± 0.02Aa	8.70 ± 0.04Aa	1.67 ± 0.02Aa	0.26 ± 0.05Abc	1.23 ± 0.02Aa	0.29 ± 0.02Cc
	2	90.14 ± 1.45Bb	2.49 ± 0.13Aa	2.74 ± 0.05Aa	1.64 ± 0.01Aa	67.37 ± 0.97Ab	3.78 ± 0.06Bcd	8.19 ± 0.07Bb	1.19 ± 0.04Cc	–	0.74 ± 0.04Cc	1.26 ± 0.04Aa
	4	89.90 ± 1.37Bb	2.45 ± 0.06Aa	2.24 ± 0.01Bb	1.64 ± 0.03Aa	67.25 ± 1.02Ab	3.63 ± 0.04Cd	7.90 ± 0.09Bc	1.40 ± 0.01Bb	–	1.03 ± 0.02Bb	1.19 ± 0.01Aa
	6	71.42 ± 2.06Cc	2.02 ± 0.08Bb	2.06 ± 0.01Cc	1.33 ± 0.02Bb	54.11 ± 1.17Bc	1.18 ± 0.02Df	6.49 ± 0.13Cd	0.80 ± 0.06Dd	–	0.34 ± 0.01Dd	0.71 ± 0.03Bb
	8	17.65 ± 1.38Dd	0.45 ± 0.17Cc	0.57 ± 0.03Dd	–	19.05 ± 0.07Cd	0.23 ± 0.01Eg	2.23 ± 0.03De	0.36 ± 0.02Ed	–	0.45 ± 0.06Dd	0.15 ± 0.02De

Results are presented as means ± SD. Column values in the same rapeseed variety with the different capital letters are significantly different ($p < 0.05$); column values in the different rapeseed varieties with different lowercased letters are significantly different ($p < 0.05$); "-", non-detectable. *Abbreviations:* GSL, glucosinolate.

3.4. Effect of microwave pretreatment on protein-bound amino acids

Amino acids serve as the fundamental building blocks of proteins and play a crucial role in the generation of aroma compounds (Lian, Cheng, & Sun, 2023). The variations in protein-bound amino acid concentrations resulting from microwave heating can be observed in Fig. 1. The predominant amino acids found in rapeseeds were proline, glutamic acid, methionine, isoleucine, leucine, and valine, with concentrations ranging from 11.30 to 27.08 mg/100 g. The progression of the Maillard reaction can lead to a reduction in protein-bound amino acid content (Sen & Gokmen, 2022). The greatest losses in proline content were observed at 800 W within 8 min, corresponding to 5.27 and 7.22 mg/100 ×g for LELG and HEHG, respectively. Glutamic acid, methionine, isoleucine, and lysine also exhibited rapid decreases during microwave heating. It is well known that the concentrations of these heavy metals significantly decrease during roasting (Zhang, Chen, Zhao, Chen, et al., 2022).

3.5. Changes in HMF concentration during microwave heating

HMF is primarily generated in foods as a result of thermal processes such as caramelization (through the decomposition of hexose sugars) or the Maillard reaction (Berk, Hamzalioglu, & Gokmen, 2019). Fig. S2 shows the formation of HMF in rapeseeds subjected to microwave heating at 800 W for different durations. No HMF was detected in the raw seeds. However, its levels rapidly increased upon microwave heating. The highest HMF formation was observed in LELG (52.18 mg/kg) after 8 min of heating. In the HEHG, only 11.50 mg/kg HMF was formed due to the lower amount of hexose sugars. Similarly, Suri, Singh, Kaur, Yadav, and Singh (2020) observed an increase in HMF levels in wheat germ with the intensification of the roasting process.

3.6. Effect of microwave pretreatment on the GSLs

GSLs are sulfur-containing secondary metabolites found mainly in the Cruciferae family. In the analysis of the two varieties of rapeseed, six aliphatic GSLs (progoitrin, epi-progoitrin, glucoraphanin, gluconapoleiferin, gluconapin, and glucobrassicanapin), three indole GSLs (4-hydroxyglucobrassicin, glucobrassicin, and 4-methoxyglucobrassicin), and two aromatic GSLs (glucotropaeolin and gluconasturtiin) were identified (Table 3). Among them, progoitrin (2.20–96.89 μmol/g), gluconapin (3.06–70.25 μmol/g), and 4-hydroxyglucobrassicin (4.28–4.62 μmol/g) were the most abundant GSLs in all the samples. These compounds are recognized as characteristic GSLs of rapeseeds, and their degradation products contribute to their pungent taste and aroma (Zhang, Chen, Zhao, Wang, & Yu, 2022). The total GSL content in the rapeseeds decreased by 78.46–91.60% after microwave processing. Previous studies have revealed that GSL degradation can facilitate the formation of nitriles and isothiocyanates in FROs under microwave treatment (Zhou et al., 2018).

3.7. Analysis of aroma-active compounds in FROs

Sixty-six volatile compounds were identified in the FROs using GC–MS analysis, as summarized in Table 4. These compounds included 14 aldehydes, 3 ketones, 12 alcohols, 7 acids, 3 alkanes, 3 furans, 11 pyrazines, 11 nitriles, and 2 isothiocyanates.

During the roasting process, the thermal oxidation and decomposition of unsaturated lipids result in the formation of oxygen-containing compounds. As indicated in Table 4, aldehydes were found to be the major pyrolysis compounds in FROs, contributing to the development of fatty, green, nutty, or rancid odors. Specifically, the oil samples analyzed contained 9 aroma-active aldehydes. For example, (E,E)-2,4-heptadienal was identified as a peroxidation product of linolenic acid, while hexanal and heptanal were produced through the degradation of linoleic acid (Yin et al., 2021). Octanal and nonanal were previously identified as

Table 4
Changes in concentrations and OAVs of volatile compounds in FROs during microwave heating.

Compounds	[a]Odor threshold (mg/kg)	RI	Variety	[b]Concentration (mg/kg)					[c]OAV				
				0 min	2 min	4 min	6 min	8 min	0 min	2 min	4 min	6 min	8 min
Aldehydes													
2-Methylbutanal	0.11	647	LELG	0.02 ± 0.00	0.02 ± 0.01	0.03 ± 0.00	0.26 ± 0.03	0.46 ± 0.06	< 1	< 1	< 1	2.32	4.19
			HEHG	0.02 ± 0.00	0.02 ± 0.01	0.02 ± 0.01	0.76 ± 0.08	1.01 ± 0.06	< 1	< 1	< 1	6.87	9.14
Pentanal	0.24	670	LELG	0.04 ± 0.01	0.06 ± 0.01	0.06 ± 0.01	0.11 ± 0.01	0.19 ± 0.03	< 1	< 1	< 1	< 1	< 1
			HEHG	–	–	–	–	–	< 1	< 1	< 1	< 1	< 1
Hexanal	0.073	771	LELG	0.12 ± 0.03	0.26 ± 0.06	0.27 ± 0.03	0.35 ± 0.06	1.26 ± 0.11	1.64	3.56	3.74	4.79	17.26
			HEHG	0.08 ± 0.02	0.09 ± 0.02	0.04 ± 0.00	0.08 ± 0.02	–	1.07	1.17	< 1	1.08	< 1
2-Ethylhexanal	0.041	968	LELG	0.02 ± 0.01	–	–	–	–	< 1	< 1	< 1	< 1	< 1
			HEHG	–	0.02 ± 0.00	–	–	–	< 1	< 1	< 1	< 1	< 1
Furfural	0.7	801	LELG	–	–	–	–	2.51 ± 0.17	< 1	< 1	< 1	< 1	3.59
			HEHG	–	–	–	3.18 ± 0.01	42.25 ± 0.18	< 1	< 1	< 1	4.55	60.36
Heptanal	0.05	879	LELG	0.07 ± 0.01	0.05 ± 0.00	0.08 ± 0.01	0.07 ± 0.01	0.08 ± 0.01	1.40	1.00	1.40	1.60	2.00
			HEHG	0.04 ± 0.01	0.04 ± 0.01	0.02 ± 0.00	0.03 ± 0.00	0.04 ± 0.01	< 1	< 1	< 1	< 1	< 1
Octanal	0.0007	983	LELG	0.08 ± 0.03	0.07 ± 0.01	0.11 ± 0.03	0.17 ± 0.04	0.28 ± 0.04	114.29	100.00	157.14	242.86	400.00
			HEHG	0.01 ± 0.00	0.04 ± 0.00	0.08 ± 0.02	0.11 ± 0.02	0.17 ± 0.03	14.29	57.14	114.29	157.14	242.86
(*E*,*E*)-2,4-Heptadienal	0.05	945	LELG	–	0.02 ± 0.00	0.06 ± 0.01	0.28 ± 0.02	0.91 ± 0.08	< 1	< 1	1.20	5.60	18.20
			HEHG	–	0.02 ± 0.00	0.08 ± 0.01	0.47 ± 0.05	1.16 ± 0.05	< 1	< 1	1.60	9.40	23.20
Benzeneacetaldehyde	0.022	1051	LELG	0.25 ± 0.06	0.28 ± 0.03	0.34 ± 0.04	0.98 ± 0.11	0.84 ± 0.02	11.32	12.73	15.63	44.49	38.13
			HEHG	0.13 ± 0.02	0.11 ± 0.03	0.18 ± 0.03	0.32 ± 0.01	1.28 ± 0.03	5.87	4.83	8.03	14.55	58.18
(*E*)-2-Octenal	0.12	1039	LELG	0.02 ± 0.00	–	0.01 ± 0.00	–	–	< 1	< 1	< 1	< 1	< 1
			HEHG	–	–	–	–	–	< 1	< 1	< 1	< 1	< 1
Nonanal	0.15	1087	LELG	0.19 ± 0.05	0.22 ± 0.04	0.25 ± 0.06	0.36 ± 0.02	1.49 ± 0.07	1.28	1.45	1.65	2.39	9.93
			HEHG	0.12 ± 0.01	0.12 ± 0.02	0.17 ± 0.03	1.87 ± 0.10	3.46 ± 0.14	< 1	< 1	1.13	12.47	23.07
Decanal	0.65	1189	LELG	0.01 ± 0.00	0.13 ± 0.02	0.02 ± 0.00	–	–	< 1	< 1	< 1	< 1	< 1
			HEHG	–	–	–	–	–	< 1	< 1	< 1	< 1	< 1
5-Methyl-2-furancarboxaldehyde	0.26	938	LELG	–	–	–	–	0.72 ± 0.23	< 1	< 1	< 1	< 1	2.77
			HEHG	–	–	–	–	18.99 ± 0.37	< 1	< 1	< 1	< 1	73.03
(*Z*)-2-Heptenal	0.25	932	LELG	0.06 ± 0.02	–	–	–	–	< 1	< 1	< 1	< 1	< 1
			HEHG	0.03 ± 0.00	0.10 ± 0.02	0.07 ± 0.02	–	–	< 1	< 1	< 1	< 1	< 1
Ketones													
2-Heptanone	3	868	LELG	–	–	–	–	0.09 ± 0.03	< 1	< 1	< 1	< 1	< 1
			HEHG	–	–	–	–	–	< 1	< 1	< 1	< 1	< 1
2,3-Pentanedione	0.003	768	LELG	–	–	–	0.31 ± 0.03	0.56 ± 0.08	< 1	< 1	< 1	102.09	185.09
			HEHG	–	–	–	–	0.60 ± 0.06	< 1	< 1	< 1	< 1	201.21
1-(2-Furanyl)-ethanone	10	884	LELG	–	–	–	0.03 ± 0.00	0.08 ± 0.01	< 1	< 1	< 1	< 1	< 1
			HEHG	–	–	–	0.38 ± 0.03	4.77 ± 0.12	< 1	< 1	< 1	< 1	< 1
Nitrile													
Methallyl cyanide	1	718	LELG	–	–	1.23 ± 0.11	12.70 ± 0.6	18.21 ± 0.31	< 1	< 1	1.23	12.70	18.21
			HEHG	2.20 ± 0.03	3.85 ± 0.17	8.81 ± 0.13	1.38 ± 0.05	0.13 ± 0.02	2.20	3.85	8.81	1.38	< 1

(*continued on next page*)

Table 4 (*continued*)

Compounds	[a]Odor threshold (mg/kg)	RI	Variety	[b]Concentration (mg/kg)					[c]OAV				
				0 min	2 min	4 min	6 min	8 min	0 min	2 min	4 min	6 min	8 min
			LELG	–	–	–	0.10 ± 0.02	0.09 ± 0.03	–	–	–	–	–
2,4-Pentadienenitrile	–	734	HEHG	0.05 ± 0.01	0.06 ± 0.01	0.19 ± 0.02	11.16 ± 0.23	15.96 ± 0.16	–	–	–	–	–
5-(Methylthio)-pentanenitrile	0.05	1130	LELG	–	–	–	–	–	< 1	< 1	< 1	< 1	< 1
			HEHG	0.05 ± 0.01	0.06 ± 0.02	0.23 ± 0.04	4.21 ± 0.10	6.74 ± 0.21	< 1	1.20	4.60	84.20	134.80
Benzenepropanenitrile	0.5	1201	LELG	0.21 ± 0.02	0.23 ± 0.02	0.22 ± 0.01	1.94 ± 0.16	0.72 ± 0.13	< 1	< 1	< 1	3.88	1.43
			HEHG	–	0.80 ± 0.02	0.39 ± 0.06	13.18 ± 0.09	17.98 ± 0.08	< 1	1.60	< 1	26.35	35.96
6-(Methylthio) hexanenitrile	–	1277	LELG	0.17 ± 0.03	0.22 ± 0.06	0.25 ± 0.04	0.98 ± 0.04	0.13 ± 0.01	–	–	–	–	–
			HEHG	0.04 ± 0.01	0.46 ± 0.05	0.22 ± 0.01	8.73 ± 0.12	13.25 ± 0.13	–	–	–	–	–
5-Cyano-1-pentene	10	823	LELG	–	0.10 ± 0.01	0.05 ± 0.00	1.20 ± 0.23	1.64 ± 0.06	< 1	< 1	< 1	< 1	< 1
			HEHG	0.54 ± 0.06	0.53 ± 0.02	1.27 ± 0.08	24.51 ± 0.47	31.69 ± 0.35	< 1	< 1	< 1	2.45	3.17
Heptanonitrile	–	947	LELG	–	–	–	0.07 ± 0.01	–	–	–	–	–	–
			HEHG	–	–	–	–	–	–	–	–	–	–
2-Methyl-butanenitrile	0.1	684	LELG	–	–	–	–	0.43 ± 0.08	< 1	< 1	< 1	< 1	4.26
			HEHG	–	–	–	–	–	< 1	< 1	< 1	< 1	< 1
3-Methylcrotononitrile	–	725	LELG	–	–	–	–	2.04 ± 0.04	–	–	–	–	–
			HEHG	–	–	–	148.45 ± 2.90	157.72 ± 3.81	–	–	–	–	–
5-Methyl-hexanenitrile	–	914	LELG	–	–	–	–	–	–	–	–	–	–
			HEHG	–	–	–	–	2.23 ± 0.02	–	–	–	–	–
2-Pentenenitrile	–	743	LELG	–	–	–	–	0.07 ± 0.02	–	–	–	–	–
			HEHG	–	–	0.01 ± 0.00	3.00 ± 0.06	4.41 ± 0.05	–	–	–	–	–
Isothiocyanates													
4-Isothiocyanato-1-butene	0.07	953	LELG	0.05 ± 0.00	0.20 ± 0.02	0.28 ± 0.03	0.41 ± 0.03	0.65 ± 0.10	< 1	2.83	4.00	5.86	9.29
			HEHG	1.83 ± 0.05	4.50 ± 0.06	3.74 ± 0.05	31.18 ± 1.38	23.44 ± 2.51	26.08	64.23	53.47	445.50	334.88
2-Isothiocyanato-butane	0.05	905	LELG	–	–	0.01 ± 0.00	0.03 ± 0.00	0.15 ± 0.03	< 1	< 1	< 1	< 1	3.02
			HEHG	–	–	–	–	–	< 1	< 1	< 1	< 1	< 1
Pyrazine													
Methyl pyrazine	0.25	797	LELG	0.01 ± 0.00	0.09 ± 0.03	0.01 ± 0.00	1.06 ± 0.05	6.31 ± 0.16	< 1	< 1	< 1	4.26	25.24
			HEHG	–	–	–	2.74 ± 0.06	4.00 ± 0.10	< 1	< 1	< 1	10.98	15.98
2,5-Dimethylpyrazine	0.02	888	LELG	0.14 ± 0.01	0.20 ± 0.02	2.70 ± 0.14	10.49 ± 0.37	16.27 ± 0.29	7.01	10.00	135.00	524.50	813.50
			HEHG	0.03 ± 0.02	0.06 ± 0.02	0.11 ± 0.01	8.91 ± 0.11	9.29 ± 0.26	1.34	3.00	5.50	445.66	464.40
Ethylpyrazine	0.089	892	LELG	–	–	–	–	1.01 ± 0.04	< 1	< 1	< 1	< 1	11.35
			HEHG	–	–	–	–	–	< 1	< 1	< 1	< 1	< 1
2,3-Dimethylpyrazine	0.4	896	LELG	–	–	–	0.06 ± 0.02	1.89 ± 0.08	< 1	< 1	< 1	< 1	4.72
			HEHG	–	–	–	0.16 ± 0.03	1.17 ± 0.01	< 1	< 1	< 1	< 1	2.92
2-Ethyl-6-methylpyrazine	0.026	977	LELG	–	–	–	0.42 ± 0.01	1.03 ± 0.03	< 1	< 1	< 1	16.01	39.62
			HEHG	–	–	–	0.32 ± 0.05	0.68 ± 0.03	< 1	< 1	< 1	12.46	26.15
Trimethylpyrazine	0.29	982	LELG	–	–	–	1.98 ± 0.05	3.49 ± 0.10	< 1	< 1	< 1	6.81	12.03
			HEHG	–	–	–	1.65 ± 0.01	2.82 ± 0.02	< 1	< 1	< 1	5.69	9.72
2-Ethyl-5-methylpyrazine	1	977	LELG	–	–	–	–	–	< 1	< 1	< 1	< 1	< 1
			HEHG	–	–	–	0.41 ± 0.06	1.17 ± 0.01	< 1	< 1	< 1	< 1	1.17
3-Ethyl-2,5-dimethylpyrazine	0.079	1062	LELG	–	–	0.03 ± 0.00	2.21 ± 0.08	3.53 ± 0.17	< 1	< 1	< 1	28.02	44.66

(*continued on next page*)

Table 4 (*continued*)

Compounds	[a]Odor threshold (mg/kg)	RI	Variety	[b]Concentration (mg/kg)					[c]OAV				
				0 min	2 min	4 min	6 min	8 min	0 min	2 min	4 min	6 min	8 min
			HEHG	–	–	–	–	2.10 ± 0.05	< 1	< 1	< 1	< 1	26.58
2-Ethyl-3,5-dimethylpyrazine	0.02	1098	LELG	–	–	–	–	0.27 ± 0.03	< 1	< 1	< 1	< 1	13.50
			HEHG	–	–	–	–	–	< 1	< 1	< 1	< 1	< 1
(*Z*)-2-Methyl-6-(1-propenyl)-pyrazine	0.009	1081	LELG	–	–	–	0.16 ± 0.02	0.34 ± 0.04	< 1	< 1	< 1	17.97	37.38
			HEHG	–	–	–	–	–	< 1	< 1	< 1	< 1	< 1
3,5-Diethyl-2-methylpyrazine	0.014	1142	LELG	–	–	–	–	0.23 ± 0.01	< 1	< 1	< 1	< 1	16.40
			HEHG	–	–	–	–	–	< 1	< 1	< 1	< 1	< 1
Alcohols													
3-Methyl-1-butanol	0.1	716	LELG	–	–	0.03 ± 0.01	–	–	< 1	< 1	< 1	< 1	< 1
			HEHG	0.04 ± 0.01	0.13 ± 0.02	0.18 ± 0.02	–	–	< 1	1.31	1.80	< 1	< 1
1-Methoxy-2-propanol	4	659	LELG	–	–	–	–	–	< 1	< 1	< 1	< 1	< 1
			HEHG	0.02 ± 0.00	–	–	–	–	< 1	< 1	< 1	< 1	< 1
1-Pentanol	1.5	746	LELG	0.17 ± 0.05	0.19 ± 0.03	0.24 ± 0.03	0.30 ± 0.01	0.32 ± 0.04	< 1	< 1	< 1	< 1	< 1
			HEHG	–	–	–	–	–	< 1	< 1	< 1	< 1	< 1
2,3-Butanediol	668	767	LELG	–	–	–	–	–	< 1	< 1	< 1	< 1	< 1
			HEHG	0.02 ± 0.00	0.03 ± 0.00	0.02 ± 0.01	–	–	< 1	< 1	< 1	< 1	< 1
2-(Methylthio)-ethanol	0.12	813	LELG	–	–	–	–	–	< 1	< 1	< 1	< 1	< 1
			HEHG	0.01 ± 0.00	0.12 ± 0.01	0.04 ± 0.01	–	–	< 1	1.00	< 1	< 1	< 1
2-Furanmethanol	15	835	LELG	–	–	–	0.12 ± 0.01	0.54 ± 0.02	< 1	< 1	< 1	< 1	< 1
			HEHG	–	–	–	1.20 ± 0.02	3.43 ± 0.04	< 1	< 1	< 1	< 1	< 1
1-Hexanol	10	854	LELG	0.19 ± 0.05	–	0.35 ± 0.06	0.21 ± 0.03	–	< 1	< 1	< 1	< 1	< 1
			HEHG	0.04 ± 0.01	–	–	–	–	< 1	< 1	< 1	< 1	< 1
4-Methyl-1-pentanol	0.82	824	LELG	–	2.91 ± 0.13	–	–	0.30 ± 0.01	< 1	3.54	< 1	< 1	< 1
			HEHG	–	0.11 ± 0.03	0.03 ± 0.00	–	–	< 1	< 1	< 1	< 1	< 1
1-Heptanol	20	958	LELG	0.01 ± 0.00	0.02 ± 0.00	0.02 ± 0.00	0.04 ± 0.01	0.07 ± 0.00	< 1	< 1	< 1	< 1	< 1
			HEHG	0.01 ± 0.00	0.06 ± 0.01	0.13 ± 0.03	0.44 ± 0.06	0.71 ± 0.03	< 1	< 1	< 1	< 1	< 1
1-Octanol	0.027	1060	LELG	0.05 ± 0.02	0.08 ± 0.01	0.13 ± 0.01	0.20 ± 0.01	0.39 ± 0.02	1.85	2.96	4.81	7.41	14.56
			HEHG	–	–	–	–	–	< 1	< 1	< 1	< 1	< 1
Phenylethyl alcohol	1.2	1094	LELG	–	0.25 ± 0.03	0.06 ± 0.01	–	–	< 1	< 1	< 1	< 1	< 1
			HEHG	0.11 ± 0.02	0.20 ± 0.02	0.11 ± 0.02	–	–	< 1	< 1	< 1	< 1	< 1
1,2-Butanediol	70	798	LELG	–	–	–	0.06 ± 0.00	0.11 ± 0.03	< 1	< 1	< 1	< 1	< 1
			HEHG	–	–	–	–	–	< 1	< 1	< 1	< 1	< 1
Acids													
Propanoic acid	0.72	687	LELG	0.06 ± 0.00	0.64 ± 0.09	0.14 ± 0.01	–	–	< 1	< 1	< 1	< 1	< 1
			HEHG	0.03 ± 0.01	–	–	–	–	< 1	< 1	< 1	< 1	< 1
Butanoic acid	0.135	777	LELG	0.01 ± 0.00	0.32 ± 0.03	0.02 ± 0.00	–	–	< 1	2.37	< 1	< 1	< 1
			HEHG	0.01 ± 0.00	–	–	–	–	< 1	< 1	< 1	< 1	< 1
3-Methyl-butanoic acid	0.022	837	LELG	0.02 ± 0.00	0.25 ± 0.01	–	0.03 ± 0.01	–	1.13	11.54	< 1	1.23	< 1
			HEHG	0.14 ± 0.02	0.09 ± 0.02	0.03 ± 0.01	–	–	6.51	4.25	1.43	< 1	< 1
2-Methyl-butanoic acid	0.11	845	LELG	0.02 ± 0.00	0.1 ± 0.02	0.01 ± 0.00	0.03 ± 0.00	–	< 1	< 1	< 1	< 1	< 1
			HEHG	0.04 ± 0.02	0.05 ± 0.01	0.02 ± 0.00	–	–	< 1	< 1	< 1	< 1	< 1
Pentanoic acid	0.6	876	LELG	0.02 ± 0.00	0.11 ± 0.01	–	–	–	< 1	< 1	< 1	< 1	< 1

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Table 4 (*continued*)

Compounds	[a]Odor threshold (mg/kg)	RI	Variety	[b]Concentration (mg/kg)					[c]OAV				
				0 min	2 min	4 min	6 min	8 min	0 min	2 min	4 min	6 min	8 min
			HEHG	0.01 ± 0.00	–	–	–	–	< 1	< 1	< 1	< 1	< 1
			LELG	0.05 ± 0.01	0.46 ± 0.03	–	–	–	< 1	< 1	< 1	< 1	< 1
Hexanoic acid	0.46	972	HEHG	0.03 ± 0.00	–	–	0.16 ± 0.03	–	< 1	< 1	< 1	< 1	< 1
			LELG	–	–	–	–	–	< 1	< 1	< 1	< 1	< 1
Nonanoic acid	0.05	1253	HEHG	–	–	–	0.45 ± 0.05	1.07 ± 0.02	< 1	< 1	< 1	9.08	21.38
Alkane													
			LELG	0.07 ± 0.02	0.80 ± 0.03	0.13 ± 0.03	0.11 ± 0.04	0.20 ± 0.03	< 1	< 1	< 1	< 1	< 1
Cyclohexane	160	657	HEHG	0.05 ± 0.00	0.09 ± 0.02	0.09 ± 0.01	0.16 ± 0.03	0.78 ± 0.02	< 1	< 1	< 1	< 1	< 1
			LELG	–	0.23 ± 0.01	0.03 ± 0.01	–	–	–	–	–	–	–
2,4-Dimethylheptane	–	796	HEHG	–	0.03 ± 0.00	–	–	–	–	–	–	–	–
			LELG	–	1.58 ± 0.05	–	–	–	< 1	1.68	< 1	< 1	< 1
Octane	0.94	948	HEHG	0.02 ± 0.00	–	–	–	–	< 1	< 1	< 1	< 1	< 1
Furans													
			LELG	0.04 ± 0.00	0.02 ± 0.00	0.16 ± 0.01	0.06 ± 0.00	0.03 ± 0.01	–	–	–	–	–
5-Ethyldihydro-2(3H)-furanone	–	1103	HEHG	–	0.02 ± 0.01	0.23 ± 0.04	0.13 ± 0.02	0.06 ± 0.02	–	–	–	–	–
			LELG	–	–	–	–	0.01 ± 0.00	–	–	–	–	–
Dihydro-2-methyl-3(2H)-furan-one	–	791	HEHG	–	–	0.07 ± 0.01	–	–	–	–	–	–	–
			LELG	–	–	–	–	–	–	–	–	–	–
2(5H)-Furanone	–	823	HEHG	–	–	0.15 ± 0.03	–	–	–	–	–	–	–

Note: "-", non-detectable. *Abbreviations:* FRO, fragrant rapeseed oil; RI, retention index; OAV, odor activity value.
[a] Odor thresholds were from references (Jia et al., 2020; Xu et al., 2022; Zhou et al., 2019).
[b] The average concentration of sample.
[c] OAVs were calculated by dividing the concentrations by the odor thresholds.

volatiles in FROs, predominantly resulting from the oxidation of oleic acid (Zhang, Chen, Zhang, Sagymbek, et al., 2022). Other aldehydes observed were products of carbohydrate pyrolysis, such as furfural, benzeneacetaldehyde, and 5-methyl-2-furancarboxaldehyde. The levels and OAVs of these aldehydes in the oils generally increased with increasing microwave heating time. In addition, compounds such as 2-heptanone, 1-(2-furanyl)-ethanone, 2-furanmethanol, 1-heptanol, 1,2-butanediol, and cyclohexane were considered significant groups of oxidized and carbohydrate-degraded compounds found in the oils. The odor threshold values of these two compounds were 3, 10, 15, 20, 70, and 160 mg/kg, respectively (Jia et al., 2020; Xu et al., 2022; Zhou et al., 2019). Due to their relatively high odor thresholds, these compounds have a limited impact on the flavor profile of FROs.

Apart from oxygen-containing compounds, nitrogen-containing compounds were also identified as typical volatiles in FROs. Amino acids and reducing sugars present in rapeseeds act as substrates for the Maillard reaction, leading to the formation of pyrazines during microwave heating (Zhang, Chen, Zhao, Chen, et al., 2022). The analysis of the oil samples revealed the presence of six aroma-active pyrazines, namely, methyl pyrazine, 2,5-dimethylpyrazine, 2,3-dimethylpyrazine, 2-ethyl-6-methylpyrazine, trimethylpyrazine, and 3-ethyl-2,5-dimethylpyrazine. These pyrazines start accumulating after 6 min of treatment and are associated with nutty or roasted fragrance characteristics that significantly contribute to the distinctive aroma of FROs (Jia et al., 2020). Compared to those in HEHG, a greater variety and concentration of pyrazines were detected in the LELG samples.

The pungent, cabbage-like, and onion-like odors detected in the oil samples were primarily attributed to the presence of isothiocyanates and nitriles, which have been previously identified as key odorants in FRO (Zhou et al., 2019). A total of eight important compounds, 4-isothiocyanato-1-butene, methallyl cyanide, 2,4-pentadienenitrile, 5-(methylthio)-pentanenitrile, benzenepropanenitrile, 6-(methylthio) hexanenitrile, 5-cyano-1-pentene, and 3-methylcrotononitrile, were detected in the oil samples. Significantly increased concentrations of these substances were observed with extended treatment time. Furthermore, the isothiocyanate and nitrile species and contents identified in the HEHG were greater than those in the LELG. Consistent with the findings of previous reports, elevated levels of isothiocyanates and nitriles generally indicate GSL degradation during roasting. For example, 4-isothiocyanato-1-butene may be generated from the thermal degradation of gluconapin, while 2,4-pentadienenitrile could be generated from progoitrin when the roasting temperature exceeded 150 °C (Zhang, Chen, Zhao, Wang, & Yu, 2022; Zhang, Lv, Yang, Zheng, et al., 2022).

3.8. *E*–nose analysis of FROs

PCA is a multivariate analysis technique that reduces dimensionality or converts multiple indicators into a few comprehensive indicators (Xu et al., 2016). As shown in Fig. 2a, a PCA distribution diagram was constructed based on the *E*-nose data collected from ten sensors for FROs. A total of eight components were extracted from the E-nose data by PCA, accounting for 100% of the total variance. The first two principal components (PC1 and PC2) explained 87.3% of the variance (76.2% and 11.1%, respectively). Among the treated samples, those roasted for 6 min (only HEHG) or 8 min were distributed on the positive

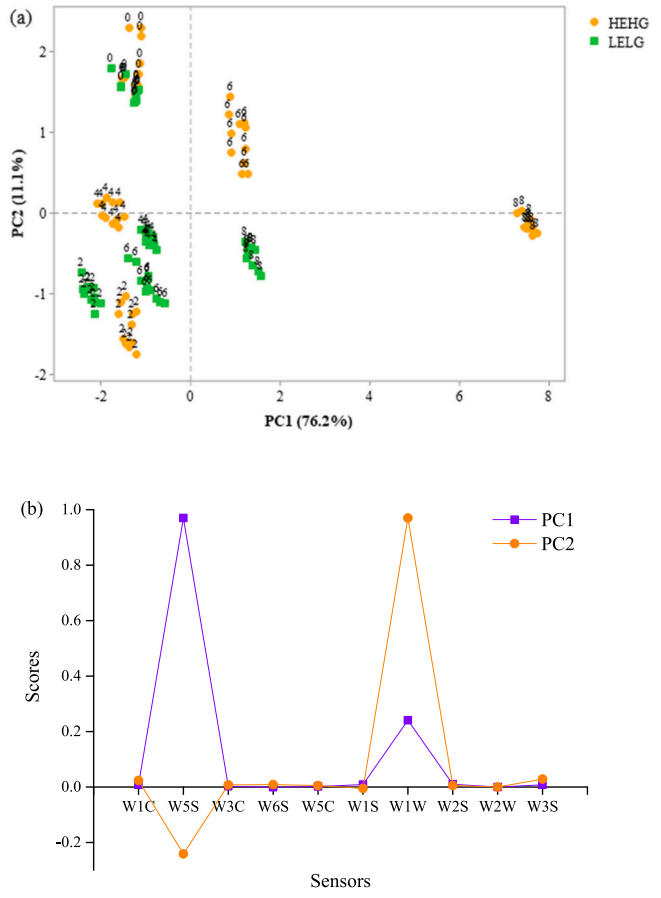

Fig. 2. The PCA sample distribution diagram (a) and loading plot (b) based on sensor responds of FROs with different pretreatment time via E–nose.

side of PC1, while the remaining samples were located on the negative side of PC1. Based on Fig. 2b, the differences among the samples were mainly detected by the response values of W5S (sensitive to nitrogen oxide compounds) and W1W (sensitive to sulfides). The mean response signals of each sensor at a response time of 57–60 s were investigated. Correlation analysis (Fig. S3) revealed a significant positive correlation ($R = 0.64$–0.86, $p < 0.05$) between the response values of W1W and W5S and the concentrations of relevant volatiles, particularly sulfides (4-isothiocyanato-1-butene) and nitrogen oxide compounds (pyrazines and nitriles). This difference may be attributed to the accelerated Maillard reaction and GSL degradation rates with increasing treatment time (He et al., 2021).

3.9. Sensory evaluation analysis

A spider diagram was generated to depict the distribution of sensory characteristics for each oil sample (Fig. S4). In line with previous research by Jing, Guo, Wang, Zhang, and Yu (2020), the primary aromas detected in virgin rapeseed oils were green-like and pickled-like notes. However, with increased microwave time, there was a gradual intensification of burnt-like, fatty-like, and nutty-like odor attributes in all the samples. This can be attributed to the higher concentrations of aldehydes and pyrazines, as observed in the study by Zhou et al. (2019). Analysis of Fig. S4 revealed that the pungent-like attribute played a key role in distinguishing between oil samples, with the highest intensity observed in the HEHG sample and a lesser intensity in the other LELG samples. This discrepancy may be attributed to the increase in isothiocyanates and nitriles, which are dominant in HEHG samples and are responsible for the pungent aroma, as suggested by Zhang et al. (2021). Additionally, Jia et al. (2023) reported that the degradation of GSL induced by microwave treatment significantly contributed to the pungent odor of radish seed oils.

Table S2 presents the results of the investigation of rapeseed variety and time effects based on *F* value evaluation. A higher *F* value indicates a more significant difference between the variables (Chen et al., 2020). For the attributes of nutty-like, burnt-like, pickled-like, green-like, and fatty-like, the effect of time was more significant than that of rapeseed variety. However, in terms of the pungent-like attribute, contrasting results were observed. Overall, these findings align with the volatile component analysis results discussed earlier.

3.10. Relationship between aroma precursors and aroma-active compounds

The relationships between 24 aroma-active compounds and aroma precursors are depicted in Fig. S5. Notably, the content of aldehydes in octanal exhibited a significant negative correlation with the content of linolenic acid ($p < 0.01$; Fig. S5a). Notably, aroma-active aldehydes are not direct byproducts of the Maillard reaction. However, certain aldehydes resulting from lipid oxidation can contribute to this reaction (Zhou et al., 2019). Moreover, the type of amino acid plays a crucial role in determining the backbone structure of pyrazines, as the nitrogen atoms in pyrazines originate from amino acids (Bi et al., 2022). According to Fig. S5b, sucrose, cysteine, lysine, and isoleucine exhibited the strongest negative correlations with the six aroma-active pyrazines ($p < 0.05$). These findings are consistent with previous studies in which sucrose, cysteine, lysine, and isoleucine were identified as important precursors in Maillard reaction model systems (Shrestha & De Meulenaer, 2014; Zhang, Chen, Zhao, Chen, et al., 2022). In comparison to conventional roasting, microwave treatment results in the generation of fewer thermorelated nitriles and isothiocyanates (Jia et al., 2020). This outcome can be attributed to the different roasting intensities employed, as well as the distinct microstructures and compositions of the raw materials. As indicated in Fig. S5c, only benzenepropanenitrile exhibited a significant negative correlation with 4-hydroxyglucobrassicin ($p < 0.05$). Overall, these results reveal a close linear relationship between aroma-active pyrazines and their aroma precursors.

4. Conclusions

In summary, the levels of polyunsaturated fatty acids, sugars, protein-bound amino acids, and GSLs decreased during the microwave heating process. In contrast, elevated amounts of FFAs, *p*-AnV, K232, K268, saturated fatty acids, and HMF were detected. GC–MS analysis revealed a total of 66 volatile compounds in the oil samples, 24 of which were identified as key odorants (OAV ≥ 1). Aldehydes, pyrazines, isothiocyanates, and nitriles were found to substantially contribute to the flavor of the evaluated FROs. Among the 10 odor sensors tested, W1W and W5S exhibited the highest sensitivity in distinguishing the flavor characteristics of FROs. Understanding the effect of microwave pretreatment on the alteration of aroma-active compounds and studying aroma precursors will be beneficial for enhancing the flavor of FROs and facilitating their industrial application.

Statement of consent

Before carrying out experiments, participants were informed about the background, process, possible benefits and risks of this research. Written informed consent was obtained from all the participants prior to the enrollment of this study.

CRediT authorship contribution statement

Lingyan Zhang: Writing – original draft, Methodology, Investigation, Conceptualization. **Jia Chen:** Methodology, Investigation, Data curation. **Xingfeng Guo:** Visualization, Investigation. **Yongsheng Cao:**

Supervision, Validation. **Guoyi Qu:** Supervision, Validation. **Qi Li:** Validation, Supervision. **Yuan Gao:** Validation, Supervision. **Xiuzhu Yu:** Writing – review & editing, Validation, Supervision, Methodology.

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. All authors have seen the manuscript and approved to submit to your journal.

Data availability

No data was used for the research described in the article.

Acknowledgement

We gratefully acknowledge the Key Research and Development Projects of Shaanxi Province (2023-YBNY-164, 2024NC-YBXM-135), the National Natural Science Foundation of China (NO. 32072260), and Shaanxi Science and Technology Innovation Team Project (2024RS-CXTD-70).

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.fochx.2024.101381.

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