



Lipid oxidation in fragrant rapeseed oil: Impact of seed roasting on the generation of key volatile compounds

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ABSTRACT

This work sought to identify the influence of roasting on lipid oxidation-derived volatile compounds in fragrant rapeseed oils (FROs) via gas chromatography–mass spectrometry and gas chromatography–ion mobility spectrometry. Seven volatiles could be regarded as aroma-active compounds by application of odor activity value (OAV ≥ 1) calculation, and caused fatty-like, nutty-like, and green-like notes. After 60 min of roasting, the OAVs of hexanal, octanal, (E,E)-2,4-heptadienal, and nonanal in FROs were greater than 3. The same compounds, including hexanal, (E,E)-2,4-heptadienal, nonanal, 1-octanol, and nonanoic acid were also detected in the model systems of lipid oxidation. Notably, the values of *p*-anisidine, conjugated dienes, and conjugated trienes increased significantly ($p < 0.05$). Furthermore, correlation analysis showed that hexanal, (E,E)-2,4-heptadienal, and nonanal have a significant positive correlation with the oxidative degree of FROs ($R = 0.70\text{--}0.94$, $p < 0.05$). Thus, the three above-mentioned aldehydes could serve as important markers for FRO quality during roasting.

1. Introduction

Fragrant rapeseed oil (FRO) is a type of hot-pressed rapeseed oil. Due to its pleasant flavor and taste, it has become popular edible oil and is commonly used as a flavor enhancer (Zhang et al., 2022a; Zhang et al., 2021). Approximately 1.5 million tons of FRO are consumed annually in China, which accounts for about 30 % of the rapeseed oil market (Zhou et al., 2019). Moreover, FRO will grow at a rate of 10 % annually.

FRO is mainly processed by cleaning, roasting, physical pressing, and centrifugation or filtration (Yu, Wang, Zhang, Liu, & Li, 2021). Among these steps, the complex reactions that occur during roasting are essential for creating flavor compounds in FROs during processing (Yang et al., 2022). The high amount of unsaturated fatty acids in FROs made it easy to oxidize them during roasting. High temperature increases the complexity of lipid oxidation since unsaturated fatty acids react more quickly at high temperatures, which could produce hydroperoxides, volatile oxidation products and other small molecular substances (Chen et al., 2017). Although classical methodologies (peroxide value, *p*-

anisidine value, conjugated dienes, etc.) have been used to assess the oxidative degree of oil, it is not easy to extract information about the chemical changes occurring in the FRO. Aspects of oxidation, volatiles are generated as a result of fatty acid chains breaking, comprising aldehydes, ketones, alcohols, acids, and esters (Zhang et al., 2021). Some of these products were important contributors to the overall flavor profiles of the oil itself. Aldehydes may greatly contribute to the flavor given their low odor threshold values. The formation of these small molecule aldehydes may be involved in the autoxidation of unsaturated fatty acids or Strecker degradation reaction of amino acids in the roasting process of rapeseed (Zhang et al., 2022b). Zhou et al. (2019) reported that the hexanal, heptanal, (E)-2-hexenal, benzaldehyde, and (E,E)-2,4-decadienal were identified as key volatile compounds in commercial FROs. These products have also been observed in the work of Yu, Wang, Zhang, Liu, and Li (2021). The majority of aldehydes reported in rapeseed oil have to do with the fatty flavor, but some lead to green, nutty, or rancid odors as well (Zhang et al., 2021). Ketones, alcohols, and acids also contribute to flavors such as 1-octene-3-one

Abbreviations: FRO, fragrant rapeseed oil; OAV, odor activity value; TAG, triglyceride; OBs, oil bodies; RI, retention index; PV, peroxide value; AV, acid value; *p*-AnV, *p*-anisidine value; K232, conjugated dienes; K268, conjugated trienes.

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(mushroom-like), 2-methoxy-4-vinylphenol (smoky), and propanoic acid (sweaty) (Jia et al., 2020). Additionally, furan-containing compounds (5-methyl-2-furancarboxaldehyde, 2-furanmethanol, and 2-pentyl-furan) also appeared, through lipid peroxidation and carbohydrates degradation (Zhang et al., 2020). These above researches were mainly focused on the overall flavor characteristics of FROs. However, there is a lack of information available in the literature regarding the association between lipid oxidation-derived volatile compounds and quality of extracted FROs during roasting.

This research was designed to identify the influences of roasting on (1) the volatile oxidation products in FRO, with a focus on both qualitative and quantitative aspects via gas chromatography–mass spectrometry (GC–MS) and gas chromatography–ion mobility spectrometry (GC–IMS), (2) determine the aroma-active volatiles in FRO identified via odor activity value (OAV) calculation, and (3) explore the relationships among the physicochemical parameters, triglycerides (TAGs), sensory evaluation, and aroma-active compounds analysis to reveal key volatile oxidation products. Moreover, we also study the production of volatile compounds in the model systems of lipid oxidation by oil bodies (OBs).

2. Materials and methods

2.1. Materials

The rapeseed varieties with low erucic acid have attracted global attention to use rapeseed as a valuable source of edible oil (Cao, Pan, Qiao, & Yuan, 2022). On the other hand, the rapeseed varieties with high erucic acid are traditional Chinese cultivars, which are favored by local people for their flavor. Moreover, high erucic acid rapeseeds still have a large proportion in China which is one of the leading producing regions. In China, rapeseed oils are called general rapeseed oil (erucic acid 3 %–60 %) and low erucic acid rapeseed oil (erucic acid < 3 %) based on the Chinese national standard (GB/T 1536-2021). Thus, two types of high-generated inbred line of seeds were included in the study. Samples 1 contained low erucic acid content (~0.5 % erucic acid), and samples 2 had high erucic acid content (~40 % erucic acid). LE and HE are the rapeseed varieties we have named for simplicity. All seeds were provided by Hybrid Rape Research Center of Shaanxi Province, China. *N*-Alkanes (C7–C30) for calculating retention index (RI) and standard 2-octanol were supplied by Sigma (St. Louis, MO, USA).

2.2. Sample preparation

Crude FRO was obtained by pressing extraction based on our prior report (Zhang et al., 2022a). Temperatures (150 °C) and times were selected in accordance with commercial processes and aimed at producing slightly under-to slightly over-roasted samples with an acceptable flavor and color. The seeds were roasted for 15, 30, 45 and 60 min. Having cooled to room temperature, FROs were extracted from roasted seeds with a small expeller. In the end, the oil samples were obtained by centrifugation (2191×g, 15 min) and stored for subsequent analysis.

2.3. Chemical analysis

Peroxide value (PV), acid value (AV), and *p*-anisidine value (*p*-AnV) were analyzed according to AOCS official methods Cd 8b-90, Cd 3d-63, and Cd 18-90, respectively. The contents of conjugated dienes (K232) and conjugated trienes (K268) were determined following GB/T 22500-2008.

2.4. Fatty acid composition analysis

The fatty acid composition was identified according to the procedure determined previously (Ma et al., 2020). Samples were analyzed on a gas chromatograph (GC-2010, Shimadzu, Kyoto, Japan) equipped with an HP-88 capillary column (100 m × 0.25 mm id × 0.20 μm).

2.5. TAG profile analysis

The TAG profile of oil samples was determined by high-performance liquid chromatography (HPLC) using the method described by Chen, Chen, Wan, and Deng (2021). Analysis of the oil samples was conducted using an Agilent HPLC 1200 series system with two LiChroCART 18e columns. The HPLC mobile phases consisted of acetonitrile and acetone (25:75, v/v). The flow rate was 1.0 mL/min at the controlled temperature of 45 °C.

2.6. GC–MS analysis

The volatile compounds of FROs were determined via headspace solid-phase microextraction (HS–SPME)/GC–MS (Shimadzu-QP2010, Kyoto, Japan) equipped with a DB-17MS column (60 m × 0.25 mm id × 0.25 μm) as previously described (Zhang et al., 2022a). The FROs were accurately weighed to 4.0 ± 0.1 g and transferred into a 20-mL headspace injection vial. Four microliters of 0.49 mg/mL 2-octanol was added as the internal standard. The volatile compounds of FRO were extracted via the HS–SPME method, and the sample was left to equilibrate for 30 min at 50 °C. Immediately following the extraction, the SPME fibre was inserted into a GC–MS sample inlet after being desorbed for 3 min at 250 °C. The flow rate of helium was 1.0 mL/min. The heating program was as follows: the initial temperature was set at 40 °C and held for 3 min, then it was increased to 120 °C at a rate of 4 °C/min and finally raised to 240 °C at a rate of 6 °C/min and held for 9 min. By comparing the retention index (RI) of volatile compounds with real reference compounds and using the NIST14 library (NIST, USA) of data, volatiles were determined.

2.7. GC–IMS analysis

In accordance with Dou et al. (2022), samples were detected by a commercial GC–IMS device (FlavorSpec®, Dortmund, Germany) equipped with an autosampler unit (CTC Analytics AG, Zwingen, Switzerland). The separation was done on a DB-WAX capillary column (30 m × 0.53 mm id × 1 μm, Bellefonte, PA, USA). Each FRO sample (2.0 ± 0.1 g) was placed in a 20 mL vial and incubated at 60 °C for 15 min. Syringes were used to inject 500 μL of headspace into the GC–IMS system at 85 °C. The column temperature was at 60 °C. Ultrapure nitrogen (purity ≥ 99.999 %) was used as a carrier gas and the flow rate was 2 mL/min in 0–2 min, then increased to 100 mL/min in 2–30 min. The drift tube was 5.3 cm long and operated at a constant voltage of 500 V/cm, a temperature of 45 °C and a drift gas flow rate of 150 mL/min (ultrapure nitrogen, purity ≥ 99.999 %). The identification of volatile compounds was based on comparing RI and the drift time with the GC–IMS library. Each sample was analyzed three times.

2.8. Sensory evaluation

Normally, sensory analyses were evaluated by trained analysts. Ten well-trained analysts (five males, five females) evaluated oil samples. Six sensory qualities, namely, nutty-like, burnt-like, pickled-like, pungent-like, green-like, and fatty-like, were evaluated via a descriptive test. For each attribute, the score ranged from 0 to 10 in 0.5 increments (0: none detected; 1–2: very weak; 3–4: weak; 5–6: moderate; 7–8: strong; 9–10: very strong).

2.9. Preparation of model systems of lipid oxidation by OBs

OBs were extracted from rapeseeds using the method described by Farooq, Abdullah, Zhang, Xi, and Zhang (2022) with minor modifications. Rapeseeds were soaked in distilled water (1:9; w/v) for 18 h at 4 °C, and then grinded in a buffer solution (0.05 M Tris-HCl, pH 7, 0.4 M sucrose) (1:5, w/v). Homogenate was centrifuged at 14,000×g for 30 min. After collecting and re-suspending the OB cream layer in the buffer

solution, a second centrifugation was performed. The final products of OBs were demonstrated in Fig. S1. The obtained OBs were heated at 150 °C for 0, 15, 30, 45 and 60 min in an oven. After the heating of the model systems, GC–MS was used to investigate the formation of volatile oxidation products added with internal standard (2-octanol).

2.10. Statistical analysis

Statistically significant differences were statistically analyzed by ANOVA and Duncan's multiple comparison tests by using SPSS version 11.5 (SPSS, USA). Statistical analysis was conducted using OriginPro 9.0 (Originlab, Northampton, MA). Heatmap was carried out by R language.

3. Results and discussion

3.1. Changes in PV, AV, p-AnV, K232, and K268

The changes of lipid oxidation indices are showed in Fig. 1. PV is a common indicator of primary lipid oxidation, which represents the level of hydroperoxides. As can be seen from Fig. 1a, the PV of all the samples first increased and then decreased as the roasting time prolonged. In the heating process, hydroperoxides rapidly decompose due to their

instability, resulting in the reduction of PV (Hu et al., 2022).

Food processors indicate that AV, which determines the level of free fatty acids in oil, is a good indicator of oil deterioration. As roasting progressed, AV of oils steadily increased (Fig. 1b). After 60 min of roasting, the AVs of FROs obtained from LE and HE were 0.77 and 1.30 mg/g, respectively. This similar result was consistent with previous findings (Mildner-Szkudlarz, Róžańska, Siger, Kowalczewski, & Rudzińska, 2019), who found that roasting significantly increased the AV of the raspberry and strawberry seed oils.

A second measure of lipid oxidation is p-AnV, which measures non-volatile carbonyls, such as aldehydes and ketones, formed by the breakdown of hydroperoxides (Zhao, Zhang, Wang, & Devahastin, 2021). As shown in Fig. 1c, the p-AnV increased progressively over 60 min of roasting. Before roasting, the p-AnVs of crude oils obtained from LE and HE were 0.33 ± 0.11 and 0.41 ± 0.08 units, respectively. After 60 min of roasting at 150 °C, the p-AnVs of FROs obtained from LE and HE increased to 11.00 ± 0.49 and 34.95 ± 1.38 units, respectively. This could be explained by the fact that oils containing great amounts of polyunsaturated fatty acids (PUFAs) have a high tendency to produce oxidation products (Feng et al., 2020).

The evolutions in K232 and K268 are efficient parameters for estimating lipid oxidation. K232 refers to conjugated dienes produced by

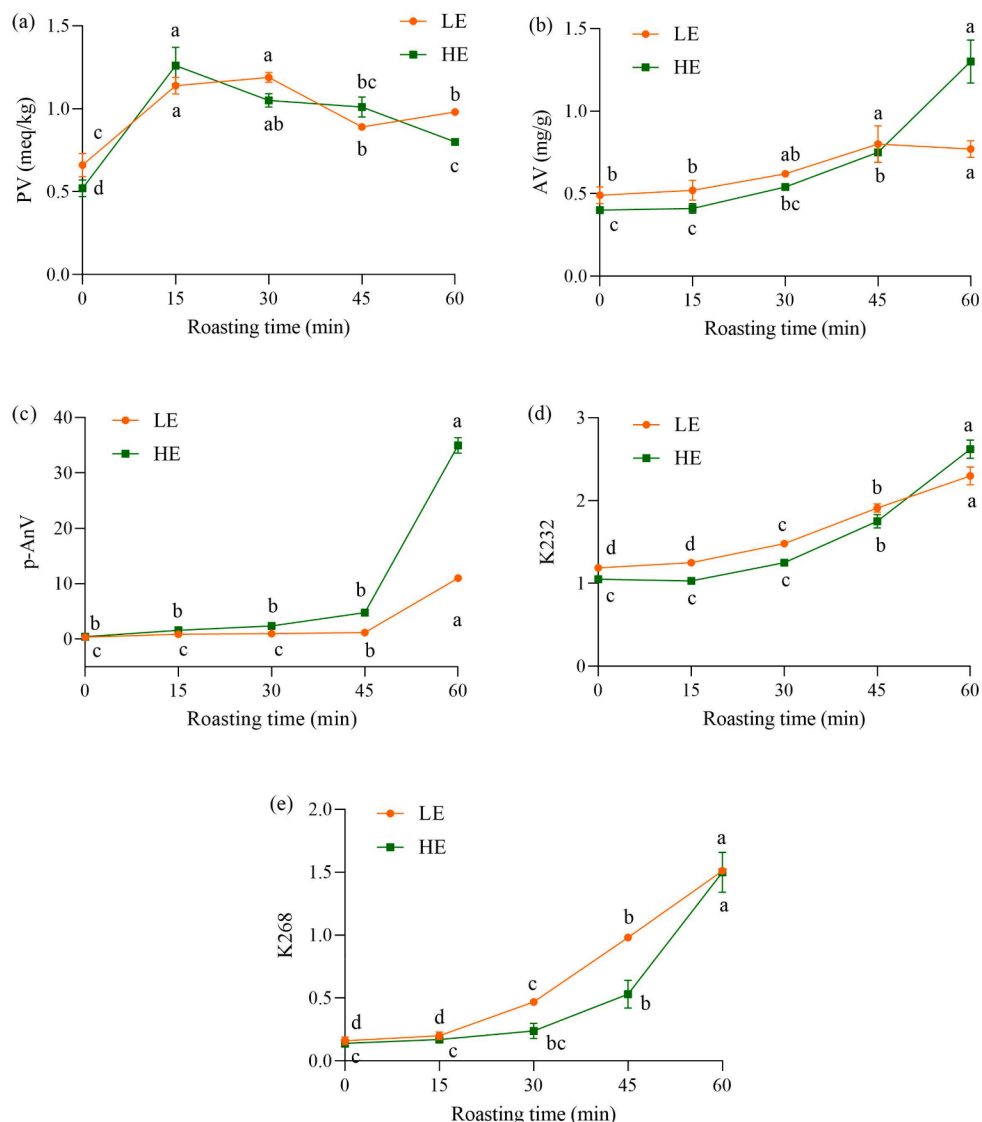


Fig. 1. Quality changes in FROs with different roasting time.

PUFA degradation, while K268 refers to aldehydes and ketones (Zhao, Zhang, Wang, & Devahastin, 2021). According to Fig. 1d and 1e, K232 and K268 were present in the fresh FRO in small quantities, with increases in both with extended roasting time, as has been seen previously in other reports (Ahmad Tarmizi, Niranjani, & Gordon, 2013; Urbančić, Kolar, Dimitrijević, Demšar, & Vidrih, 2014).

3.2. Changes in fatty acid composition

The FROs prepared from roasted seeds were subjected to fatty acid composition analysis to determine the effects of roasting on the fatty acid profile. Table S1 illustrated that fatty acid composition for oils obtained from LE and HE had a simple pattern consisting of palmitic, stearic, oleic, linoleic, linolenic, and erucic acids. The two oils enormously differed in their fatty acid composition. The oil sample from raw LE contained predominantly oleic (60.16 %), linoleic (17.00 %), and linolenic (10.28 %) acids. The major fatty acids of oil sample from raw HE were oleic (23.01 %), linoleic (9.55 %), linolenic (16.45 %), and erucic (44.55 %) acids. The erucic acid content was very low for crude oil from LE (0.51 %), in contrast to the high content in crude oil from HE (44.55 %). Moreover, there were rare levels of palmitic acid (2.91–4.04 %) and stearic acid (1.22–2.23 %) in the crude oils. From Table S1, it also can be seen that roasting treatment could increase stearic and oleic acids of FROs. The amount of linoleic, linolenic, and erucic acids in samples from the seeds heated at 150 °C decreased with the roasting duration. The reason for this phenomenon may be that thermal treatment could lead to oxidative deterioration of unsaturated fats (Lin et al., 2016). Żyżelewicz et al. (2014) also found that the level of unsaturated fatty acids significantly decreased in roasted cocoa beans at the temperature of 135 and 150 °C, which was consistent with our findings.

3.3. Changes in TAG profile

The TAGs make up 95–98 % of the total oil composition (Zeb, 2012). The quantitative result of TAGs in oils from LE and HE is listed in Table 1. Some TAGs were determined in all the samples, including LLLn, LLL, OLL, OLLP, OOLn, LOP, OOL, OOP, and OOO. Nevertheless, some TAGs were specific to a certain oil, such as ErLnP, ErLP, ErOL, ErOO, ErErLn, ErErO, ErErL, and ErErO, which was only present in oils from HE. This was in line with the data presented above. It can be observed that OLLn, OLL, OOLn, OOL, and OOO were the major TAGs in crude oil from LE present in 8.90 %, 8.73 %, 11.74 %, 18.02 %, and 20.42 %, respectively; ErOO, ErErLn, ErErO, ErErL, and ErErO were the major TAGs in crude oil from HE present in 6.13 %, 9.16 %, 7.81 %, 10.51 %, and 12.76 %, respectively. As illustrated in Table 1, roasting process at various times significantly altered the TAG profile, especially reducing the contents of OLLn (1.39 %), OLL (2.66 %), OOL (1.10 %), OOO (1.68 %), ErErLn (1.04 %), and ErErL (1.34 %). These findings were in line with those of Chen, Chen, Wan, and Deng (2021) who found that OLL, OLLn, OLnO, OOO, OLO, and OLLn were significantly decreased in rapeseed oil after heating at 180 °C. In fact, there is a direct correlation between the number of double bonds in the carbon chain and the rate of fatty acid degradation. It was found that PUFAs, especially linoleic and linolenic acids, were more susceptible to oxidative damage than others (Ma et al., 2020; Ren et al., 2021). Together, these findings showed that the roasting procedure caused PUFA in rapeseeds to degrade oxidatively, which increased the quantity of oxidation products.

3.4. Quantification of volatile oxidation products by GC–MS

The oxidation of lipids during roasting is one of the essential factors leading to the flavor of FRO (Zhang et al., 2022b). By using GC–MS, the relative amounts of volatile compounds in oil samples were determined (Table 2). Based on MS data, they have previously been identified as important secondary oxidation products of FRO (Zhou et al., 2019). The most prevalent type of volatile in FROs, which constituted the majority

Table 1
Evolution of the absolute content (%) of TAGs in FROs during roasting.

Rapeseed varieties	TAG (g/100 g)	Roasting time (min)				
		0	15	30	45	60
LE	L-Ln-	0.99 ±	0.91 ±	0.87 ±	0.81 ±	0.76 ±
	Ln	0.04a	0.01ab	0.02bc	0.01 cd	0.02d
	L-L-Ln	1.77 ±	1.64 ±	1.48 ±	1.30 ±	0.86 ±
		0.03a	0.02b	0.04c	0.03d	0.01e
	O-Ln-	3.14 ±	3.06 ±	2.70 ±	2.60 ±	2.43 ±
	Ln	0.27a	0.25ab	0.15ab	0.02ab	0.05b
	L-L-L	1.54 ±	1.15 ±	0.85 ±	1.88 ±	1.19 ±
		0.01b	0.03c	0.03d	0.03a	0.06c
	O-L-Ln	8.90 ±	7.51 ±	7.47 ±	7.50 ±	7.51 ±
		0.26a	0.03b	0.02b	0.13b	0.08b
	O-L-L	8.73 ±	8.13 ±	7.63 ±	6.50 ±	6.07 ±
		0.13a	0.04b	0.15c	0.05d	0.04e
	L-L-P	3.38 ±	2.95 ±	2.93 ±	2.90 ±	2.89 ±
		0.02a	0.01b	0.04b	0.05b	0.01b
	O-O-	11.74	13.12 ±	13.45	12.59	13.70
	Ln	± 1.44a	0.1a	± 0.12a	± 0.01a	± 0.08a
	L-O-P	4.90 ±	4.29 ±	4.17 ±	4.19 ±	4.12 ±
		0.12a	0.11b	0.08b	0.1b	0.16b
	O-O-L	18.02	17.80 ±	17.33	17.14	16.92
		± 0.25a	0.24ab	±	± 0.05c	± 0.05c
O-O-P	5.55 ±	5.15 ±	4.95 ±	4.94 ±	4.94 ±	
	0.12a	0.01b	0.02b	0.05b	0.03b	
O-O-O	20.42	19.49 ±	19.13	19.06	18.74	
	± 0.20a	0.04b	±	± 0.05 cd	± 0.04d	
HE	L-Ln-	–	–	–	–	–
	Ln	–	–	–	–	–
	L-L-Ln	0.28 ±	0.26 ±	0.25 ±	0.25 ±	0.23 ±
		0.02a	0.01ab	0.01ab	0.02ab	0.01b
	O-Ln-	–	–	–	–	–
	Ln	–	–	–	–	–
	L-L-L	0.58 ±	0.58 ±	0.55 ±	0.55 ±	0.54 ±
		0.03b	0.01b	0.03b	0.01b	0.03b
	O-L-Ln	–	–	–	–	–
	O-L-L	0.37 ±	0.39 ±	0.38 ±	0.33 ±	0.32 ±
		0.02a	0.00a	0.01a	0.00b	0.01b
	L-L-P	0.65 ±	0.64 ±	0.47 ±	0.54 ±	0.49 ±
		0.03a	0.02a	0.02b	0.0b	0.02b
	O-O-	0.76 ±	0.69 ±	0.67 ±	0.73 ±	0.59 ±
	Ln	0.01a	0.01bc	0.01c	0.01ab	0.02d
	L-O-P	0.82 ±	0.77 ±	0.71 ±	0.86 ±	0.74 ±
		0.02ab	0.01abc	0.05c	0.02a	0.03bc
	O-O-L	1.03 ±	1.02 ±	0.96 ±	0.88 ±	0.77 ±
		0.02a	0.02a	0.01a	0.02b	0.01c
	O-O-P	0.85 ±	0.84 ±	0.77 ±	0.84 ±	0.79 ±
	0.02a	0.02a	0.06a	0.03a	0.03a	
O-O-O	2.01 ±	1.99 ±	1.94 ±	1.85 ±	1.72 ±	
	0.03a	0.01a	0.02ab	0.04b	0.01c	
Er-Ln-	1.77 ±	1.95 ±	1.86 ±	1.85 ±	1.76 ±	
P	0.12a	0.30a	0.29a	0.17a	0.36a	
Er-L-P	4.64 ±	4.46 ±	4.02 ±	4.71 ±	3.53 ±	
	0.05ab	0.02b	0.04c	0.06a	0.08d	
Er-O-L	4.91 ±	4.90 ±	4.75 ±	4.67 ±	4.44 ±	
	0.03a	0.02a	0.04b	0.00b	0.02c	
Er-O-	6.13 ±	6.03 ±	7.39 ±	5.71 ±	6.08 ±	
O	0.03a	0.02a	1.52a	0.02a	0.53a	
Er-Er-	9.16 ±	8.53 ±	8.54 ±	8.39 ±	8.12 ±	
Ln	0.04a	0.04b	0.05b	0.08b	0.05c	
Er-Er-	7.81 ±	7.34 ±	7.02 ±	7.26 ±	7.70 ±	
O	0.08a	0.34a	0.47a	0.88a	0.09a	
Er-Er-	10.51	10.04 ±	9.86 ±	9.54 ±	9.17 ±	
L	± 0.01a	0.18ab	0.21b	0.12bc	0.16c	
Er-Er-	12.76	12.16 ±	11.14	11.65	12.08	
O	± 0.30a	0.33a	± 0.74a	± 0.87a	± 0.75a	

Note: “–”, not detected. Results are means ± SD of triplicate determinations. Values with different letters in the same row are significantly different ($p < 0.05$). Abbreviations: FRO, fragrant rapeseed oil; TAG, triglycerides; P, palmitic acid; O, oleic acid; L, linoleic acid; Ln, linolenic acid; Er, erucic acid.

Table 2
The concentrations and OAVs of volatile oxidation products.

Compounds	^a Odor threshold (mg/kg)	RI	Variety	^b Concentration (mg/kg)					^c OAV				
				0	15	30	45	60	0	15	30	45	60
Aldehydes													
2-methylbutanal	0.11	647	LE	0.02 ± 0.00	0.03 ± 0.00	0.07 ± 0.02	0.50 ± 0.21	0.82 ± 0.04	<1	<1	<1	4.55	7.45
			HE	–	0.02 ± 0.00	0.11 ± 0.03	0.70 ± 0.05	1.07 ± 0.06	<1	<1	1.00	6.36	9.73
pentanal	0.24	670	LE	0.04 ± 0.01	0.05 ± 0.01	0.06 ± 0.01	0.09 ± 0.05	0.11 ± 0.03	<1	<1	<1	<1	<1
			HE	–	–	–	–	–	<1	<1	<1	<1	<1
hexanal	0.073	771	LE	0.12 ± 0.03	0.14 ± 0.02	0.18 ± 0.04	0.35 ± 0.07	0.48 ± 0.05	1.64	1.92	2.47	4.79	6.58
			HE	0.08 ± 0.02	0.09 ± 0.02	0.09 ± 0.02	0.13 ± 0.04	0.28 ± 0.06	1.10	1.23	1.23	1.78	3.84
2-ethylhexanal	0.041	968	LE	0.02 ± 0.01	–	–	–	–	<1	<1	<1	<1	<1
			HE	–	–	0.02 ± 0.00	–	–	<1	<1	<1	<1	<1
furfural	0.7	801	LE	–	–	0.13 ± 0.06	0.86 ± 0.05	36.79 ± 2.03	<1	<1	<1	1.23	52.56
			HE	–	0.06 ± 0.02	0.08 ± 0.01	0.93 ± 0.11	53.46 ± 4.28	<1	<1	<1	1.33	76.37
heptanal	0.05	879	LE	0.07 ± 0.01	0.05 ± 0.01	0.05 ± 0.02	0.09 ± 0.02	0.11 ± 0.01	1.40	1.00	1.00	1.80	2.20
			HE	0.04 ± 0.01	0.03 ± 0.01	0.02 ± 0.01	0.06 ± 0.01	0.08 ± 0.01	<1	<1	<1	1.20	1.60
octanal	0.0007	983	LE	0.08 ± 0.03	0.09 ± 0.02	0.13 ± 0.02	0.25 ± 0.09	0.34 ± 0.05	114.29	128.57	185.71	357.14	485.71
			HE	0.01 ± 0.00	0.05 ± 0.01	0.05 ± 0.01	0.09 ± 0.04	0.13 ± 0.01	14.29	71.43	71.43	128.57	185.71
(E,E)-2,4-heptadienal	0.05	945	LE	–	0.05 ± 0.02	0.08 ± 0.03	0.22 ± 0.06	0.83 ± 0.04	<1	1.00	1.60	4.40	16.60
			HE	–	0.05 ± 0.01	0.12 ± 0.01	0.36 ± 0.02	1.11 ± 0.21	<1	1.00	2.40	7.20	22.20
(E)-2-octenal	0.12	1039	LE	0.02 ± 0.00	0.04 ± 0.01	–	–	–	1.67	3.33	<1	<1	<1
			HE	–	–	–	–	–	<1	<1	<1	<1	<1
nonanal	0.15	1087	LE	0.19 ± 0.05	0.19 ± 0.05	0.20 ± 0.02	0.27 ± 0.04	0.89 ± 0.16	1.27	1.27	1.33	1.80	5.93
			HE	0.12 ± 0.01	0.15 ± 0.03	0.18 ± 0.02	2.84 ± 0.61	3.01 ± 0.04	<1	1.00	1.20	18.93	20.07
(E)-2-nonenal	0.14	1127	LE	–	–	0.02 ± 0.00	0.06 ± 0.01	0.13 ± 0.02	<1	<1	<1	<1	<1
			HE	–	0.04 ± 0.01	0.10 ± 0.02	0.33 ± 0.01	0.61 ± 0.05	<1	<1	<1	2.36	4.36
decanal	0.65	1189	LE	0.01 ± 0.00	0.01 ± 0.00	0.05 ± 0.00	0.41 ± 0.05	0.55 ± 0.06	<1	<1	<1	<1	<1
			HE	–	0.03 ± 0.00	0.05 ± 0.01	0.87 ± 0.06	1.44 ± 0.11	<1	<1	<1	1.34	2.22
5-methyl-2-furancarboxaldehyde	0.26	938	LE	–	–	–	–	5.27 ± 0.38	<1	<1	<1	<1	20.27
			HE	–	–	–	–	85.21 ± 2.37	<1	<1	<1	<1	327.73
(Z)-2-heptenal	0.25	932	LE	0.06 ± 0.02	0.18 ± 0.02	0.18 ± 0.06	0.35 ± 0.05	–	<1	<1	<1	1.40	<1
			HE	0.03 ± 0.00	–	–	–	–	<1	<1	<1	<1	<1
Ketones													
2-heptanone	3	868	LE	–	0.02 ± 0.00	–	0.15 ± 0.02	0.21 ± 0.03	<1	<1	<1	<1	<1
			HE	–	–	–	–	–	<1	<1	<1	<1	<1
2,3-pentanedione	0.003	768	LE	–	–	–	0.64 ± 0.08	1.00 ± 0.05	<1	<1	<1	213.33	333.33
			HE	–	–	0.06 ± 0.01	0.08 ± 0.02	0.62 ± 0.05	<1	<1	20.00	26.67	206.67
2-octanone	12	971	LE	–	–	–	–	–	<1	<1	<1	<1	<1
			HE	–	–	–	0.05 ± 0.01	0.09 ± 0.01	<1	<1	<1	<1	<1
1-(2-furanyl)-ethanone	10	884	LE	–	–	–	–	0.89 ± 0.04	<1	<1	<1	<1	<1
			HE	–	–	–	–	6.85 ± 0.56	<1	<1	<1	<1	<1
	170	1045	LE	–	–	–	–	–	<1	<1	<1	<1	<1

(continued on next page)

Table 2 (continued)

Compounds	^a Odor threshold (mg/kg)	RI	Variety	^b Concentration (mg/kg)					^c OAV				
				0	15	30	45	60	0	15	30	45	60
1-(1H-pyrrol-2-yl)-ethanone			HE	–	–	–	–	1.85 ± 0.12 3.22 ± 0.51	<1	<1	<1	<1	<1
Alcohols													
3-methyl-1-butanol	0.1	716	LE	–	–	–	–	–	<1	<1	<1	<1	<1
			HE	0.04 ± 0.01	0.07 ± 0.01	0.18 ± 0.03	–	–	0.40	0.70	1.80	<1	<1
1-methoxy-2-propanol	4	659	LE	–	–	–	–	–	<1	<1	<1	<1	<1
			HE	0.02 ± 0.00	–	–	–	–	<1	<1	<1	<1	<1
1-pentanol	1.5	746	LE	0.17 ± 0.05	0.16 ± 0.03	0.18 ± 0.05	0.57 ± 0.06	0.64 ± 0.05	<1	<1	<1	<1	<1
2,3-butanediol	668	767	HE	–	–	–	–	–	<1	<1	<1	<1	<1
			LE	–	–	–	–	–	<1	<1	<1	<1	<1
			HE	0.02 ± 0.00	0.03 ± 0.00	0.02 ± 0.00	–	–	<1	<1	<1	<1	<1
2-(methylthio)ethanol	0.12	813	LE	–	–	–	–	–	<1	<1	<1	<1	<1
			HE	0.01 ± 0.00	–	0.06 ± 0.02	–	–	<1	<1	<1	<1	<1
2-furanmethanol	15	835	LE	–	–	–	0.16 ± 0.03	–	<1	<1	<1	<1	<1
			HE	–	–	–	0.64 ± 0.02	7.22 ± 0.21	<1	<1	<1	<1	<1
1-hexanol	10	854	LE	0.19 ± 0.05	–	0.17 ± 0.06	0.34 ± 0.03	0.23 ± 0.06	<1	<1	<1	<1	<1
			HE	0.04 ± 0.01	0.07 ± 0.01	–	–	–	<1	<1	<1	<1	<1
4-methyl-1-pentanol	0.82	824	LE	–	0.19 ± 0.05	–	–	–	<1	<1	<1	<1	<1
			HE	–	–	–	–	–	<1	<1	<1	<1	<1
1-heptanol	20	958	LE	0.01 ± 0.00	0.03 ± 0.02	0.04 ± 0.00	0.08 ± 0.00	0.11 ± 0.02	<1	<1	<1	<1	<1
			HE	0.01 ± 0.00	0.07 ± 0.03	0.34 ± 0.06	0.68 ± 0.06	1.26 ± 0.21	<1	<1	<1	<1	<1
1-octanol	0.027	1060	LE	0.05 ± 0.02	0.03 ± 0.01	0.04 ± 0.00	0.11 ± 0.02	0.25 ± 0.03	1.85	1.11	1.48	4.07	9.26
phenylethyl alcohol	1.2	1094	HE	–	–	–	–	–	<1	<1	<1	<1	<1
			LE	–	0.09 ± 0.02	0.07 ± 0.02	–	–	<1	<1	<1	<1	<1
			HE	0.11 ± 0.02	0.19 ± 0.04	0.22 ± 0.08	–	–	<1	<1	<1	<1	<1
1,2-butanediol	70	798	LE	–	–	–	0.14 ± 0.02	0.21 ± 0.03	<1	<1	<1	<1	<1
			HE	–	–	–	–	–	<1	<1	<1	<1	<1
Acids													
propanoic acid	0.72	687	LE	0.06 ± 0.00	–	–	0.13 ± 0.02	–	<1	<1	<1	<1	<1
			HE	0.03 ± 0.01	–	–	–	–	<1	<1	<1	<1	<1
butanoic acid	0.135	777	LE	0.01 ± 0.00	–	–	–	–	<1	<1	<1	<1	<1
			HE	0.01 ± 0.00	0.02 ± 0.00	–	–	–	<1	<1	<1	<1	<1
3-methylbutanoic acid	0.022	837	LE	0.02 ± 0.00	0.02 ± 0.00	–	–	–	<1	<1	<1	<1	<1
			HE	0.14 ± 0.02	0.11 ± 0.02	0.11 ± 0.03	–	–	6.36	5.00	5.00	<1	<1
2-methylbutanoic acid	0.11	845	LE	0.02 ± 0.00	0.01 ± 0.00	0.02 ± 0.00	–	–	<1	<1	<1	<1	<1
			HE	0.04 ± 0.02	0.03 ± 0.00	–	–	–	<1	<1	<1	<1	<1
pentanoic acid	0.6	876	LE	0.02 ± 0.00	0.02 ± 0.00	0.03 ± 0.02	–	–	<1	<1	<1	<1	<1
			HE	0.01 ± 0.00	0.01 ± 0.00	–	–	–	<1	<1	<1	<1	<1
hexanoic acid	0.46	972	LE	0.05 ± 0.01	0.09 ± 0.03	0.14 ± 0.03	–	–	<1	<1	<1	<1	<1
			HE	0.03 ± 0.00	–	–	–	–	<1	<1	<1	<1	<1
nonanoic acid	0.05	1253	LE	–	–	–	–	–	<1	<1	<1	<1	<1
			HE	–	–	0.05 ± 0.00	0.37 ± 0.05	2.14 ± 0.13	<1	<1	1.00	7.40	42.80

Note: “-”, not detected. Abbreviations: RI, retention index; OAV, odor activity value.

^a Odor thresholds were from reference (Xu et al., 2022).

^b The average concentration of sample.

^c OAVs were calculated by dividing the concentrations by the odor thresholds.

of these volatiles, was aldehyde. As shown in Table 2, some aroma-active aldehydes, including hexanal (grass, green, and fat), octanal (citrus-like, fat, grassy, and green), (E,E)-2,4-heptadienal (nut and fat), and nonanal (fatty, green, soapy, and sweet), were identified in all oil samples. Zhou et al. (2019) also confirmed that hexanal, octanal, and (E,E)-2,4-heptadienal were present in commercial FROs. The OAVs and contents of these volatiles in samples significantly increased with roasting time, which was consistent with the results of *p*-AnV and K268. The OAVs of hexanal (6.58 in LE, 3.84 in HE), octanal (485.71 in LE, 185.71 in HE), (E,E)-2,4-heptadienal (16.60 in LE, 22.20 in HE), and nonanal (5.93 in LE, 20.07 in HE) in oil samples extracted from rapeseeds roasted for 60 min were observed in average values higher than 3. Of note, the (E,E)-2,4-heptadienal content in HE samples was higher than that in LE samples, however, which was contrary in the octanal. Various fatty acids are oxidized differently, resulting in this difference. (E,E)-2,4-Heptadienal is a compound that is produced by oxidizing linolenic acid, whereas octanal is produced by degrading oleic acid (Zhang et al., 2021).

Ketones, alcohols, and acids are also considered important for the

flavor of FROs. The three kinds of aroma-active acid compounds, i.e. 2,3-pentanedione (buttery), 1-octanol (citrus-like and soapy), and nonanoic acid (moldy, rancid, and pungent) were found in higher OAVs in some oil samples. Jing, Guo, Wang, Zhang, and Yu (2020) found that the 2,3-pentanedione content increased dramatically after roasting at 150 °C for 40 min and showed high OAVs. Cold-pressed rapeseed oil also contained 1-octanol, which plays an important role as an odorant (Zhang et al., 2021). Aroma-active compounds in heated oil are produced as a result of oxidation of fatty acids, and nonanoic acid is a representative acid of this group (Xu et al., 2022). Overall, a major part of the oxidation products that increased obviously in OAV after 60 min of roasting were aldehydes.

3.5. Identification of volatile oxidation products by GC-IMS

GC-IMS was applied to look at the differences between test samples based on their volatile fingerprints (Fig. 2). In Fig. 2a, the entire spectrum represents the total volatiles, and each bright signal on the reaction ion peak's right represents a particular compound. Signals with blue

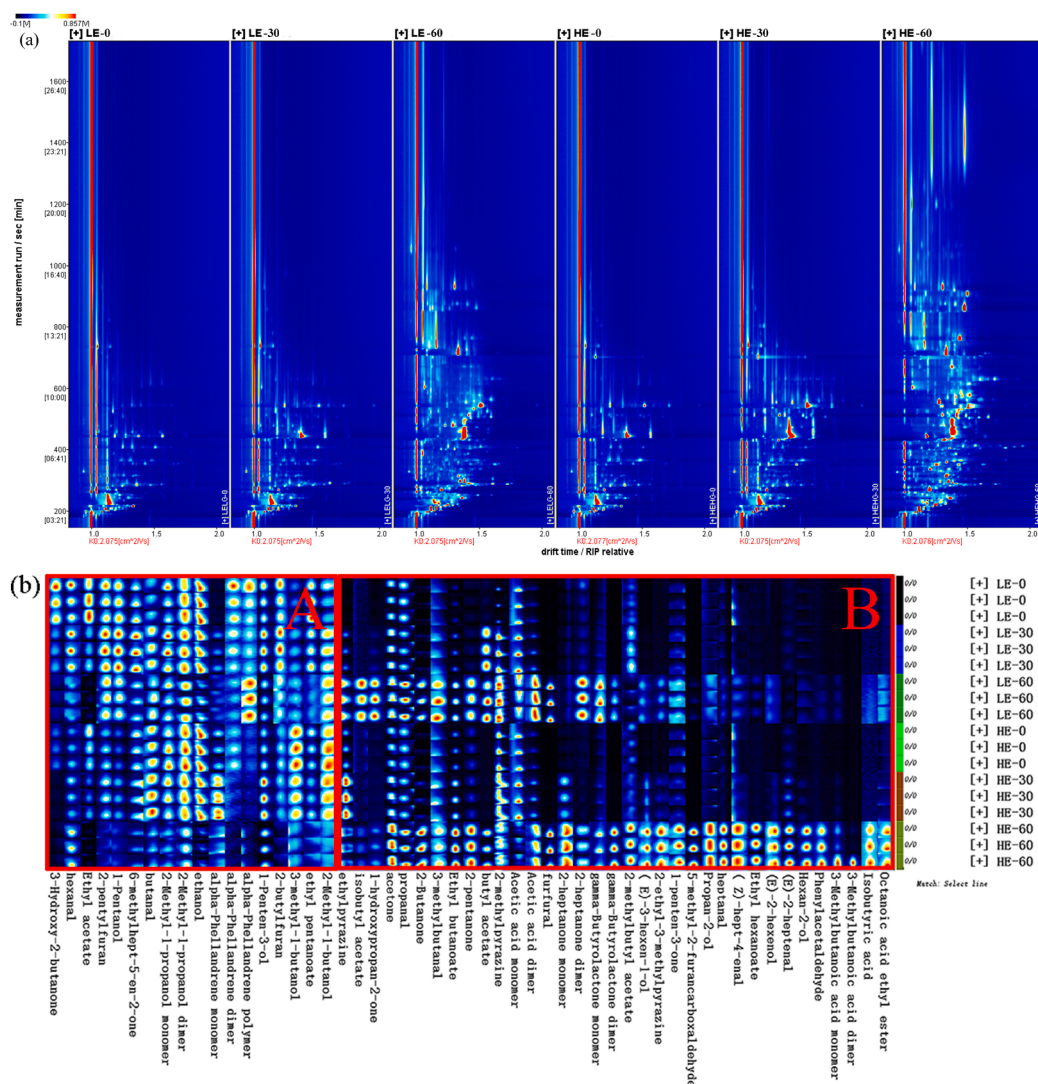


Fig. 2. Top-view plot (a) and gallery plot (b) based on the signal intensity of oils from roasted seeds with different processing time via GC-IMS.

hues indicated weak intensity, while signals with red hues indicated strong intensity. In the samples roasted 60 min, the concentration and number of volatile compounds were significantly higher than in samples roasted for shorter periods. To compare the differences among the volatiles that are present in roasted seeds, the sample information was plotted in a gallery plot (Fig. 2b). An intensified signal of each compound is indicated by a brighter signal, whereas a weaker signal is indicated by a darker signal. From region A, it is clear that the concentrations of these compounds in the sample of 0 min were much higher and they represented typical volatiles in natural rapeseed oil from seeds without roasting treatment. Region B is the higher concentration of volatile compounds in the 60 min sample, and these compounds grew significantly as the roasting time increased (especially for HE samples). Similar results were obtained in GC–MS analysis. Also, five common volatiles were observed by using GC–MS and GC–IMS, including hexanal, furfural, heptanal, 2-heptanone, and 1-pentanol. Linoleic acid undergoes oxidation to form hexanal and oleic acid undergoes oxidation to produce heptanal (Lee & Choe, 2012). He, Wu, and Yu (2021) also found the number of volatiles identified by GC–IMS was not as large as GC–MS. As a GC detector, the IMS responds non-linearly, which poses challenges for providing accurate concentration measurements at ppb and ppt levels (Wang, Chen, & Sun, 2020).

3.6. Sensory evaluation analysis

The spider diagram was used to plot the sensory scores of oil samples, and six sensory notes were chosen for evaluation (Fig. S2). The intensity of sensory attributes exhibited distinct differences among the oils prepared with different seeds and roasting time. Original samples exhibited the weakest burned-like, fatty-like, nutty-like, as well as the strongest green-like sensory attributes, as described in previous studies (Gracka, Jelen, Majcher, Siger, & Kaczmarek, 2016; Zhang et al., 2022). By contrast, the aroma profile of FROs was significantly changed as the roasting time increased. All these samples had increased burned-like,

fatty-like, and nutty-like notes, but exhibited decreased green-like note. These results were strongly consistent with the volatile profile analysis mentioned above. The stronger fatty-like note in FROs was mainly caused by the high levels of aldehydes (Zhang et al., 2021). As for the pungent-like note, it was the strongest profile for HE, but less in other LE samples. This result could be related to the content of isothiocyanate, nitrile, and heterocyclic compounds in HE samples (Jia et al., 2020).

3.7. Determination of volatile oxidation products in model systems

Many plant tissues contain natural oil bodies (OBs); they are particularly abundant in oleaginous seeds. Generally, OBs are made up of a TAG core that is surrounded by a monolayer membrane of phospholipids and OB-associated proteins (Zhang et al., 2022). The GC–MS analysis of model systems yielded the following volatile oxidation products (Table 3). Comparing their RIs and mass spectra with the data above, 13 volatile oxidation products were identified. The same volatiles, including hexanal, (E,E)-2,4-heptadienal, nonanal, 1-octanol, and nonanoic acid were identified from the model systems. With roasting time, their concentrations and OAVs increased, causing an overall flavor of FROs that smells nutty-like and fatty-like odors (Jing, Guo, Wang, Zhang, & Yu, 2020). Thus, these results further confirmed these compounds are involved in the flavor formation of FROs when heated, especially for aldehydes.

3.8. Pearson correlation analysis

The data was plotted as a heat map, with the color visualizing the differences among the paired indices (Fig. 3). As for volatile oxidation products, hexanal, (E,E)-2,4-heptadienal, and nonanal were discovered to significantly correlate positively with *p*-AnV, K232, or K268 ($R = 0.70\text{--}0.94$, $p < 0.05$). According to these findings, they were highly correlated with the degree of deterioration of samples. Furthermore,

Table 3

The volatile oxidation products in model systems.

Compounds	Variety	Concentration (mg/kg)					OAV				
		0	15	30	45	60	0	15	30	45	60
Aldehydes											
hexanal	LE	–	–	1.50 ± 0.05	2.05 ± 0.23	3.31 ± 0.15	<1	<1	20.55	28.08	45.34
	HE	–	–	0.34 ± 0.02	1.68 ± 0.05	2.66 ± 0.08	<1	<1	4.66	23.01	36.44
heptanal	LE	–	–	0.84 ± 0.05	1.06 ± 0.03	2.28 ± 0.02	<1	<1	16.80	21.20	45.60
	HE	–	–	0.34 ± 0.02	0.94 ± 0.01	2.47 ± 0.04	<1	<1	6.80	18.80	49.40
(E,E)-2,4-heptadienal	LE	–	2.13 ± 0.06	4.82 ± 0.13	5.89 ± 0.02	15.53 ± 0.34	<1	42.60	96.40	117.80	310.60
	HE	–	3.49 ± 0.04	5.33 ± 0.11	13.78 ± 0.21	19.45 ± 0.41	<1	69.80	106.60	275.60	389.00
nonanal	LE	–	2.09 ± 0.07	4.84 ± 0.26	5.88 ± 0.10	8.51 ± 0.22	<1	13.93	32.27	39.20	56.73
	HE	–	1.52 ± 0.04	3.93 ± 0.06	8.33 ± 0.27	14.56 ± 0.38	<1	10.13	26.20	55.53	97.07
(E)-2-octenal	LE	–	0.22 ± 0.01	0.40 ± 0.02	0.53 ± 0.02	1.81 ± 0.05	<1	1.57	2.86	3.79	12.93
	HE	–	–	0.07 ± 0.01	2.81 ± 0.02	5.20 ± 0.01	<1	<1	20.07	37.14	
decanal	LE	–	–	0.15 ± 0.02	0.13 ± 0.01	0.20 ± 0.00	<1	<1	<1	<1	<1
	HE	–	0.10 ± 0.00	0.21 ± 0.03	1.36 ± 0.04	4.43 ± 0.07	<1	<1	<1	2.09	6.82
Ketones											
2-heptanone	LE	–	0.09 ± 0.01	0.11 ± 0.02	0.15 ± 0.01	0.23 ± 0.01	<1	<1	<1	<1	<1
	HE	–	–	–	–	–	<1	<1	<1	<1	<1
2-octanone	LE	–	–	–	–	–	<1	<1	<1	<1	<1
	HE	–	0.16 ± 0.01	0.37 ± 0.02	0.44 ± 0.01	0.81 ± 0.03	<1	<1	<1	<1	<1
Alcohols											
1-pentanol	LE	–	–	–	–	1.06 ± 0.02	<1	<1	<1	<1	<1
	HE	–	–	–	–	2.45 ± 0.04	<1	<1	<1	<1	1.63
4-methyl-1-pentanol	LE	–	–	–	–	–	<1	<1	<1	<1	<1
	HE	–	–	–	–	0.04 ± 0.00	<1	<1	<1	<1	<1
1-heptanol	LE	–	–	0.70 ± 0.05	0.94 ± 0.03	1.82 ± 0.01	<1	<1	<1	<1	<1
	HE	–	0.61 ± 0.01	1.25 ± 0.04	3.17 ± 0.10	4.51 ± 0.05	<1	<1	<1	<1	<1
1-octanol	LE	–	0.23 ± 0.02	0.74 ± 0.01	0.89 ± 0.03	2.46 ± 0.04	<1	8.52	27.41	32.96	91.11
	HE	–	–	–	–	–	<1	<1	<1	<1	<1
Acids											
nonanoic acid	LE	–	–	–	0.38 ± 0.02	0.74 ± 0.05	<1	<1	<1	7.60	14.80
	HE	–	–	–	0.52 ± 0.05	0.97 ± 0.03	<1	<1	<1	10.40	19.40

Note: “–”, not detected. Abbreviation: OAV, odor activity value.

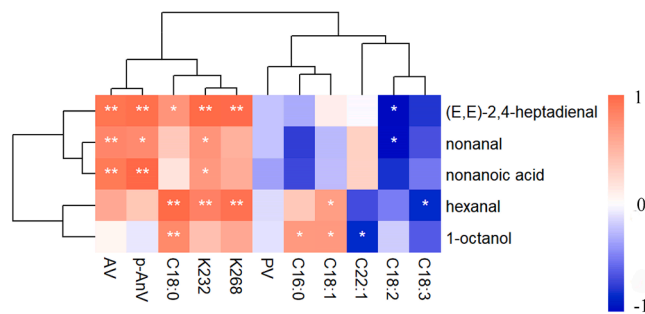


Fig. 3. Heat map of Pearson correlation analysis.

there is a negative correlation between PUFAs (linoleic acid and linolenic acid) and the above-mentioned aldehydes should be noted ($p < 0.05$), which indicates that roasting increased lipid oxidation of PUFA in rapeseeds. Hu et al. (2022) also reported that roasting scallops increased levels of PUFA oxidation and free radicals, both in relation to time and temperature. Taken together, these data confirmed that hexanal, (E,E)-2,4-heptadienal, and nonanal could serve as markers for oil stability during roasting.

4. Conclusions

In the present study, we demonstrate an examination of the quality of FRO roasted for 60 min, not only in terms of lipid oxidation parameters, but also in terms of degradation indicators of volatile. Roasting decreased the level of PUFAs in samples, while *p*-AnV, K232, and K268 increased. GC-MS and GC-IMS were applied to detect seven aromatic compounds. Moreover, GC-MS is a more comprehensive and accurate way of detecting volatile compounds. Also, five mutual compounds had been identified in model systems of lipid oxidation by OBS via GC-MS, including hexanal, (E,E)-2,4-heptadienal, nonanal, 1-octanol, and nonanoic acid. Pearson correlation analysis further supported that hexanal, (E,E)-2,4-heptadienal, and nonanal were identified as key volatile oxidation products for FRO during roasting. The results obtained in the present study will form a basis for future attempts to investigate the interactions between aroma-active compounds and lipid oxidation and to regulate the flavors of foods with rapeseeds as the main ingredients.

Declaration of Competing Interest

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

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.fochx.2022.100491>.

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