processes

MDPI

Article

Characterization of Key Odorants in Hemp Seed Oil Extracted from the Hemp Seeds Roasted Under Various Conditions

Yuchen Bai [1], Ningke You [1], Hongyu Tian [1] and Xuebing Zhao [2,*]

[1] Beijing Key Laboratory of Flavor Chemistry, Beijing Technology and Business University, Beijing 100048, China; baiyuchen@btbu.edu.cn (Y.B.); tianhy@btbu.edu.cn (H.T.)
[2] Key Laboratory of Industrial Biocatalysis, Ministry of Education, Tsinghua University, Beijing 100084, China
* Correspondence: zhaoxb@mail.tsinghua.edu.cn

Abstract: Hemp seed oil is nutritious. Besides cannabinoids, it has a higher Omega-3 fatty acid content, making it an ideal and healthy edible oil. Roasting is a key factor affecting the flavor of hemp seed oil. In this study, solvent-assisted flavor evaporation (SAFE) combined with gas chromatography–mass spectrometry (GC-O-MS) was used to analyze the volatile compounds of hemp seed oil after the hemp seeds were roasted at different temperatures (120 °C, 140 °C, 160 °C, 180 °C). α-pinene, (1*S*)-(1)-β-pinene, myrcene, and (*E*)-β-ocimene, which are typical odorants for herbs and fresh flavors, had an active odor value (OAV) ≥ 1. The sweet, nutty, and toasty flavors became more pronounced as the temperature increased. 2-pentylfuran provided the caramel flavor, and 2,5-dimethylpyrazine, 2-ethyl-6-methylpyrazine, and 2-ethyl-5-methylpyrazine provided the toasty flavor. The combination of sensory evaluation and data analysis was effective in differentiating the odors of hemp seed oil extracted at different temperatures. Reconstitution and deletion experiments showed that these odorants could mimic the overall aroma profile. This article provides a theoretical basis for the effect of over-roasting on the flavor of hemp seed oil.

Keywords: hemp seed oil; solvent-assisted flavor evaporation; odor activity values; volatile compounds

check for updates

Academic Editors: Philippe Evon and Evelien Uitterhaegen

Received: 10 January 2025
Revised: 8 February 2025
Accepted: 10 February 2025
Published: 13 February 2025

Citation: Bai, Y.; You, N.; Tian, H.; Zhao, X. Characterization of Key Odorants in Hemp Seed Oil Extracted from the Hemp Seeds Roasted Under Various Conditions. *Processes* **2025**, *13*, 530. https://doi.org/10.3390/pr13020530

Copyright: © 2025 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https://creativecommons.org/licenses/by/4.0/).

1. Introduction

Hemp seed (*Cannabis sativa* L.) is an annual herbaceous plant in the family *Cannabis* and is considered one of the most important crops. Hemp seed origin in the world is mainly distributed in Europe, North America, and the Chinese region [1]. China is the country with the largest cultivation of hemp in the world, accounting for 50% of the world's total production. Nowadays, hemp seed cultivation is widespread in China, mainly in cities such as Guangxi, Yunnan, Inner Mongolia, Shanxi, and Heilongjiang, making it the largest cannabis cultivation region in Asia [2]. Hemp seed is rich in nutritional value, mainly in its protein, fat, amino acids, etc. [3]. Hemp seed contains about 35% of the oil, as the oil is usually extracted by cold pressing hemp seed [4]. Hemp seed oil (HSO) has an ω6:3 ratio of unsaturated fatty acids of about 3:1 [5]. In comparison to other plant oils, hemp seed oil has the highest proportion of polyunsaturated fatty acids [6]. According to Matilde's research, the HSOs had a ratio of ω6:3 from 2.60 to 3.67 [7], indicating that it is an ideal vegetable oil, and its applications are spread across a variety of industries, such as cosmetics, pharmaceuticals, animal husbandry, etc., especially in the food field [8]. In the food industry, in addition to nutritional properties, flavor is an important factor in driving consumer demand.

Different physicochemical compositions were detected in hemp seeds from different geographical regions, and the characteristic flavor substances of hemp seeds from different regions were also affected. Chen studied the analytical characterization of different hemp seed oils from eight regions in China and concluded that hemp seeds in Guangxi Bama have the most nutritional value for humans [9]. Song effectively distinguished Shanxi hemp seeds by comparing the changes in volatile compounds in different regions through HS-SPME/GC-MS and the chemical composition of hemp seeds [10]. At present, there is still a lack of in-depth research on the volatile compounds extracted from hemp seed oil in Inner Mongolia.

Most commercially available hemp seed oil is obtained through cold pressing using a screw press. Borhade et al. showed that hemp seed oil has a pleasant nutty flavor and a light odor [11]. Matthäus et al. demonstrated that hemp seed oil sometimes has a slight bitterness and a rancid odor due to the deterioration of fatty acids [3]. Matilde et al. constructed a flavor sensory wheel based on sensory evaluation of 16 commercially available cold-pressed hemp seed oils, providing a basis for the study of the characteristic flavor of hemp seed oil [12]. However, fewer studies have been conducted on the flavor characteristics of hemp seed oil obtained by pressing hemp seeds after roasting.

The roasting process is essential for the formation of aroma volatiles and the improvement of the desired organoleptic characteristics [13]. The different roasting temperatures will also have varying degrees of impact on its flavor. Therefore, roasting is considered an essential process for enhancing the flavor of vegetable oils [14]. Jia et al. reported that more aroma-active volatile compounds were detected in sesame oil extracted from roasted seeds than in raw sesame oil [15]. Zhang et al. compared the aroma-active compounds of palm kernel oil extracted from unroasted and roasted seeds and reported the types of aroma active compounds and their increase in odor intensity after roasting [16]. Jeong et al. used SPME combined with E-nose to study the key flavor compounds of roasted hemp seed oil after roasting and believed that the sweetness of the sample increased with the increase of roasting temperature. Due to the Maillard reaction, the number of pyridine and pyrazine compounds increased, while the terpene compounds representing the aroma of green grass decreased [17].

The key odorants in hemp seed oil are significantly affected by an appropriate food matrix extraction method. Solvent-assisted flavor evaporation (SAFE) has now gained acceptance and has been widely used in vegetable oils such as olive oil [18], peanut oil [19], and sunflower oil [20]. To the best of the researchers' knowledge, SAFE has never been used for aroma analysis of extracted hemp seeds and HSOs.

Therefore, in this study, SAFE was used in combination with GC-O-MS and aroma extract dilution analysis (AEDA) for further in-depth investigation of the key aroma compounds. OAVs were used to quantify the odor-active compounds of Inner Mongolian hemp seed and hemp seed oil. The compounds were more comprehensively characterized by aroma recombination and omission experiments, and the effective differentiation of hemp seed oils obtained by roasting at different temperatures was based on data analysis. These results may provide a favorable profile for analyzing and evaluating the flavor changes of Inner Mongolian hemp seed and hemp seed oil roasted at different temperatures.

2. Materials and Methods

2.1. Materials

2.1.1. Plant Materials

Guangxi hemp seed (GX) was obtained by Fushuotang Company (Haozhou, China), Shanxi hemp seed (SX) was obtained by Shunshantang Herbal Shop (Shanxi, China), Heilongjiang hemp seed (HLJ) was obtained by Wanhua Baicao Farming Company (Haozhou,

China), and Inner Mongolia hemp seed (NMG) was obtained by Source of National Medicine Online Shop (Beijing, China). The hemp seeds were all harvested in September 2023. After picking, they were stored in a −4 °C refrigerator.

2.1.2. Chemicals

Hexanal (98%), hydroxyacetone (98%), 5-methylfurfural (98%), isovaleric acid (95%), valeric acid (97%), (*E*)-2-heptenal (95%), (*E*)-2-decenal (95%), 3-octanone (99%), sabinene (90%), γ-terpinene (90%), neoalloocimene (90%), α-caryophyllene (90%), acetic acid (99%), toluene (90%), 2-ethylpyridine (98%), tetramethylpyrazine (96%), pyrrole (99%), 2-pentylpyridine (98%), acetamide (98%) were obtained from Aladdin Ltd. (Shanghai, China). Benzaldehyde (98%), acetylpyrazine (98%), furfuryl alcohol (98%), 2,3-pentanedione (97%), 2-methylpyrazine (97%), 4-dimethylstyrene (95%), furfural (98%), 3-ethyl-2,5-dimethylpyrazine (98%) were from Adamas Reagent Co., Ltd., (Shanghai, China). 1-hexanol (98%), 2-acetylpyrrole (98%), ethylpyrazine (98%), 2,3-butanediol (98%), phenethyl alcohol (98%) were supplied by J&K Ltd. (Beijing, China). 2,5-dimethylpyrazine (98%), trimethylpyrazine (97%), 2-ethyl-5-methylpyrazine (95%), heptaldehyde (98%), styrene (99%), (*Z*)-β-ocimene (90%), β-caryophyllene (90%), propionic acid (99%), p-cymene (90%) and 3-octanol (99%), dichloromethane, anhydrous sodium sulfate (analytical reagent), sodium chloride were obtained from Acros Ltd. (Beijing, China). 2-octanone (98%), 2-heptanone (98%), 1-octanol (98%), fenchol (97%) were supplied by Bidepharm Ltd. (Shanghai, China). Octanal (98%), 2,6-dimethylpyrazine (98%), 2-ethyl-6-methylpyrazine (98%), dimethyl sulfoxide (98%), 1-Penten-3-ol (97%), 1-pentanol (99%), 2-heptanol (98%), linalool (98%), (*E*,*E*)-2,4-decadienal (97%), (*E*,*E*)-2,4-heptadienal (97%), (*E*,*E*)-2,4-nonadienal (97%), (*E*,*Z*)-2,4-nonadienal (97%), methylheptenone (98%), α-pinene (98%), camphene (95%), (1*S*)-(1)-β-pinene (99%), 3-carene (90%), myrcene (90%), β-phellandrene (90%), (*E*)-β-ocimene (90%), 3-ethylpyridine (99%), 2-isobutylthiazole (98%) were obtained from Macklin Ltd. (Shanghai, China). 1-octen-3-ol (98%), 2-ethylhexanol (99%) were obtained from Hwrk Ltd. (Shanghai, China). (*E*)-2-octenal (99%), γ-butyrolactone (98%), butyric acid (98%), γ-hexanolide (97%), hexanoic acid (98%), 2-pentylfuran (99%) were obtained from Yuanye Bio-Technology Co., Ltd. (Shanghai, China). (*E*)-2-hexenal (97%), (*E*)-2-nonenal (98%), (+)-dipentene (95%), terpinolene (90%), 2,3-dimethylpyrazine (98%) were purchased from TCI Ltd. (Shanghai, China). Liquid nitrogen, high purity nitrogen, helium (purity ≥99.999%) used in this experiment were supplied by Shuangquan Tianyuan Industrial Gas Co., Ltd. (Beijing, China). C7−C28 normal alkanes for retention index (RI) calculations were obtained from Tanmo Quality Inspection Technology Co., Ltd. (Beijing, China).

2.2. Preparation of Hemp Seeds

Inner Mongolian hemp seeds were selected for roasting. The hemp seeds were roasted in a high-temperature hot-air cycle through an oven (Grants KF32-G01). According to the method in the literature, with slight modifications, 1 kg of hemp seeds was evenly placed in trays, the temperatures were set at 120 °C, 140 °C, 160 °C, and 180 °C, and the seeds were tossed every 5 min to ensure the uniformity of heat and roasted for a total of 20 min [17,21]. The obtained hemp seeds and roasted hemp seeds (Figure 1) were placed in a refrigerator at −4 °C for storage.

2.3. Extraction of Hemp Seed Oil

The hemp seeds roasted at different temperatures were put into a preheated screw press machine (ZY-22A, Hongguo Technology Co., Ltd., Wenzhou, China), and the extraction temperature was set at 120 °C, so that 40 g of hemp seed oil could be extracted from each 1 kg of hemp seeds. After 72 h of resting, the virgin hemp seed oil was removed from

the precipitation, put into 100 mL light-proof brown glass bottles, and then put into the refrigerator at −4 °C for freezing.

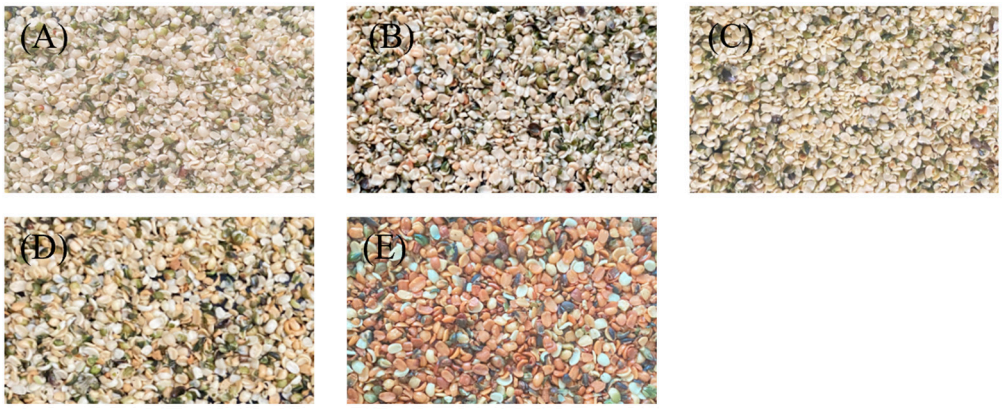

Figure 1. Inner Mongolian hemp seeds roasted at different temperatures for 20 min: (**A**) raw sample from Inner Mongolia hemp seeds in shell; (**B**) hemp seed samples after roasting at 120 °C; (**C**) hemp seed samples after roasting at 140 °C; (**D**) hemp seed samples after roasting at 160 °C; (**E**) hemp seed samples after roasting at 180 °C.

2.4. Aroma Profile Evaluation (APE)

The sensory evaluation panel consisted of 10 professional sensory judges between the ages of 20 and 40 (from the Beijing Key Laboratory of Fragrance Chemistry, Beijing, China, Beijing Technology and Business University, Beijing, China); these judges were from the specialty of flavor chemistry and had received prior training in characteristic odor analysis and quantitative descriptive analysis (QDA) [22]. QDA enables sensory evaluators to reach a consensus on the vocabulary of aroma descriptions that are similar or convergent in the same sample. The experiment was carried out at room temperature (23 °C), and 10 g of hemp seed oil samples under different temperature conditions were placed in 20 mL wide-mouth brown glass bottles with lids and odorless flavors. The panelists obtained the corresponding descriptors by ticking off the words that were similar to the descriptions in the sensory evaluation form based on the cold-pressed hemp seed oil sensory flavor wheel as a reference [12]. Seven main flavors were ultimately selected: nutty, toasted, seeds, herbs, rancid, pungent, and sweet. The intensity of each aroma was subsequently evaluated on a 9-point scale ranging from 1 (imperceptible) to 9 (strongly perceptible). All trials were repeated three times, and the average scores provided by the ten panelists were plotted on a radar chart to comparatively demonstrate the intensity of the flavors.

2.5. SAFE for Volatile Isolation from Sample

2.5.1. SAFE for Volatile Isolation from Hemp Seed

Twenty grams of hemp seed was frozen in liquid nitrogen for 5 min, and then the hemp seed was ground in a blender for 5 s to ensure that the husk of the hemp seed was separated from the kernel. The crushed hemp seed was extracted with 180 mL of dichloromethane in an incubator shaker for 1 h. The process was repeated three times at 200 rpm at room temperature. The odor components in the hemp seeds were successfully extracted. The volatiles were then separated by a SAFE device at 2.5×10^{-5} mbar (Edwards TIC Pumping Station from BOC Edwards, I. D. Burgess Hill, UK) [23]. The obtained distillate was thawed naturally at room temperature and dried by adding 20 g of anhydrous sodium sulfate and dried by absorption, then concentrated to 1.0−1.5 mL by passing it through a Vigreux chromatography column (50 cm length × 1 cm internal diameter) (Beijing Jingxing Glassware Co., Ltd., Beijing, China) at 47 °C, put into brown vials, and then concentrated to 0.5 mL by blowing with nitrogen. The concentrated fraction was stored in a refrigerator at −40 °C.

2.5.2. SAFE for Volatile Isolation from Hemp Seed Oil

Forty grams of flax oil was weighed and extracted in an incubator shaker with 180 mL of dichloromethane for 1 h. The process was repeated three times at 200 rpm at room temperature. The odor components in the hemp seed oil were successfully extracted, and the volatiles were separated by a SAFE device at 2.5×10^{-5} mbar. Other conditions were the same as in Section 2.5.1.

2.6. Analysis

2.6.1. GC-O-MS Aanalysis

GC-O-MS analysis was conducted with an Agilent 7890B GC connecting with an Agilent 5977 A mass spectrometric detector and sniffing port (Gerstel GmbH & Co., Mülheim an der Ruhr, Germany). Hemp seed oil was concentrated, and the distillate was extracted by SAFE and analyzed. The injection volume was 1 µL, and spitless mode was used. All samples were analyzed with a DB-WAX capillary column (30 m × 0.25 mm × 0.25 µm film thickness; Agilent Technologies Inc., Santa Clara, CA, USA). Helium was used as the carrier gas, with a constant flow rate of 3 mL/min. The temperature was programmed to increase from 40 °C (held for 1 min), then to 120 °C at a rate of 2 °C/min (held for 1 min), and finally to 220 °C at a rate of 6 °C/min (held for 5 min). The mass spectrometry analysis was performed in full scan mode with an ionization energy of 70 eV, the ion source temperature was set at 230 °C, and the quadrupole temperature was adjusted to 150 °C. Acquisition was performed in the range of 33–350 amu using full scan mode. A Y-shaped glass splitter was used between the FID and the olfactory detector to separate the fractions at the end of the capillary column in a 1:1 ratio. The temperature of one glass capillary tube connected to the inhalation port was set to 120 °C, and the temperature of the other capillary tube connected to the FID was set to 220 °C. The GC-O analysis was repeated three times for each sample to ensure the accuracy of the experiment. To avoid overlooking the aroma-active components in the isolate, GC-O-MS analysis was performed by three well-trained specialists on two columns with different polarities. All members were students of Beijing Key Laboratory of Flavor Chemistry at Beijing Technology and Business University (2 females and 1 male, aged between 20 and 25). Before GC-O-MS analysis, the members had been trained in the laboratory by smelling propylene glycol solutions of odorants with different concentrations for 90 min per day to allow them to have the abilities of differentiating and describing odor attributes. The training lasted for 1–2 months, according to the familiarity of the members with the odors of these compounds. During the GC-O-MS analyses, members were asked to record the odor characteristics they sniffed and their retention times. When a scent was smelled by at least two members, the aroma-active area was determined.

2.6.2. Characterization of Odor-Active Compounds

The retention index of each aroma compound was calculated based on the retention time of n-alkane determined by GC-MS. The compounds were identified by comparing the aroma characteristics of the olfactory compounds, mass spectrometry data, retention indices, and data from a standard reference material.

2.6.3. Determination of FD Factor by Aroma Extract Dilution Analysis (AEDA)

To ensure that potentially odor-active compounds were also detected, the dilution analysis was conducted by a total of three trained judges. The formation method of AEDA is to dilute the sample [24] with dichloromethane solvent in a gradient of $1:2^n$ (1:2, 1:4, 1:8... 1:2048) to obtain a series of gradient diluents, which are analyzed by GC-O on a chromatographic column until no odor is detected at the olfactory port. The FD factor is for the highest dilution or maximum GC separation ratio of odors perceived by the three

judges at the olfactory port. If two or more people can perceive the process, it is considered an effective result, which compensates for the loss of individual smell and determines the important aroma active ingredients in the sample.

2.6.4. Quantitative Analysis of Odor-Active Compounds

Quantitative analysis was conducted using the internal standard curve method, and the SIM model was used for analysis. Firstly, 3-octanol solution (10^{-4} g/mL) was used as the internal standard to perform semi quantitative analysis on the sample. Then, based on the semi-quantitative data obtained, five mixed solutions with different concentration levels were prepared using standard samples, and 200 μL of internal standard was added. The concentration of the compound at a known concentration of the internal standard can be calculated by measuring the peak area of the standard compound and the peak area of the internal standard (Equation (1)). Establishing a standard curve can obtain more accurate results.

$$C_i = \frac{A_i}{A_0} \times C_0 \; C = \frac{1}{k} \times C_i \quad (1)$$

where C_i is the semi-quantitative mass concentration of the compound, g/mL; C_0 is the mass concentration of the internal standard compound, g/mL; C is the concentration of the compound, g/mL; A_i and A_0 are the peak areas of the compound and the internal standard, respectively; K is the slope of the equation obtained by drawing the standard curve.

2.6.5. Threshold Determination and OAV Calculation

OAV is used to assess the magnitude of a compound's contribution to the overall aroma of a sample and is obtained by dividing the concentration by its odor threshold in the medium. In this experiment, hemp seed oil was chosen to have a threshold in odorless matrix oil for the calculation [25]. The thresholds of compounds in oil referred to ref odor thresholds are determined according to the book Compilations of Flavour Threshold Values in Water and Other Media (Second Enlarged and Revised Edition) [26]. For individual substances for which thresholds in odorless oil media could not be queried or were not available (Table 1), they were determined according to the three-point forced selection method (3-AFC) [27]. The specific formula for the calculation of the OAV values is as follows (Equation (2)):

$$OAV = \frac{C_i}{OT_i} \quad (2)$$

where C_i is the mass concentration of the compound in the sample (μg/kg), and OT_i is the threshold of the compound in water (μg/kg). The purpose of this experiment was to screen out key aroma compounds with OAV ≥ 1, and the higher the OAV value, the higher the contribution to the overall flavor of the sample.

Table 1. Threshold detection of compounds in odorless oils by the three-alternative forced-choice (3-AFC) test.

Nos.	Volatile Compounds	CAS	Threshold (μg/kg)
1	Fenchol	1632-73-1	384.73
2	2-Isobutylthiazole	18640-74-9	625
3	β-Caryophyllene	87-44-5	1403.08
4	Myrcene	123-35-3	106.07
5	1-Hexanol	111-27-3	757.70
6	(*E*,*Z*)-2,6-Dimethylocta-2,4,6-triene	7216-56-0	118.42
7	2,3-Butanediol	513-85-9	0.000015

2.7. Aroma Recombination and Deletion Experiment

Compounds with OAV ≥ 1 were dissolved in odorless propylene glycol according to their concentration in the samples (odor threshold 3360 mg/kg) and finally quantified to 10 g for the recombinant model [28]. The reconstructed model was oscillated in a constant temperature culture oscillator for 10 min to make it evenly mixed and then stood in equilibrium for 2 h. Finally, according to the selected odor properties, the sensory evaluator was asked to score the reconstructed model compared with the original sample, still using the 9-point scale (same as Section 2.4.), and the results were drawn into a radar map. An odor was omitted from the complete recombination model to establish the omission model, and significance analysis was conducted through triangle test to judge the importance of missing substances to the overall aroma [29]. Active odor compounds with large OAV were analyzed after the deletion experiments were selected according to different odor categories.

2.8. Statistical Analysis

Each experiment was repeated for three times, and the data were calculated and sorted using Microsoft Office Excel (v2016). The column chart and radar chart were made by Origin software (v2021). The clustering heat map was drawn using GraphPad Prism 10 (v2023). Orthogonal partial least squares discriminant analysis (OPLS-DA) method was determined by Swedish SIMCA14.1 software (v2015).

3. Results and Discussion

The volatile compound profiling indicates the differences between hemp seed oils depending on plant variety and seed treatment before pressing [30]. These volatile aromatic active components can directly influence the sensory quality of the oil and thus the consumer's choice [31].

3.1. Active Odor Compounds of Hemp Seeds

3.1.1. GC-O-MS Analysis of Hemp Seed from Four Different Geographical Regions

Volatile compounds from hemp seeds were separated by the SAFE method and analyzed by GC-O-MS. The compounds were also characterized based on their odor characteristics, mass spectra, and retention indices, in comparison with those of the reference compounds. The results are listed in Table 2. A total of 35 odor-active compounds were identified in the four samples, with 15 compounds in GX, 15 in SX, 17 in HLJ, and 23 compounds in NMG, and the common compounds among them were linalool (FD 8–32), α-pinene (FD 256–1024), (1*S*)-(1)-β-pinene (FD 128–256), (+)-dipentene (FD 128–256), and hexanoic acid (FD 4–64). α-pinene (FD 256–1024) and myrcene (FD 512). These compounds are important components of the unique odor of hemp seeds, and they provide herbal, light, and spiced aromas. Nissen extracted α-pinene, myrcene, (1*S*)-(1)-β-pinene, and (+)-dipentene as the most abundant compounds in different varieties of hemp seeds [32]. Malingre has also isolated compounds such as myrcene, α-pinene, (+)-dipentene, and (1*S*)-(1)-β-pinene in different hemp seeds [33]. Hemp seeds from four different geographic regions contained a wide range of volatile flavor substances, including alcohols, aldehydes, ketones, esters, terpenes, acids, furans, nitrogenous compounds, and sulfurous compounds. As shown in Table 2 and Figure 2, aldehydes, alcohols, and terpenes accounted for the highest percentage of volatile compound content in hemp seeds. GX hemp seeds had the lowest total number of compounds detected, a mild odor, and the least characteristic aroma, with hexyl acetate (FD 8) having a fruity aroma and benzaldehyde (FD 64) having the highest relative amount and a refreshing lemony aroma. SX hemp seeds have the highest content of the compound β-caryophyllene (FD 512), which provides a botanical, refreshing flavor to hemp seeds, and its unique compounds, camphene and α-caryophyllene (FD

16), which provide it with a woody flavor. HLJ hemp seed has the highest content of acid compounds, especially hexanoic acid (FD 64), which gives it a pungent and spicy odor. NMG hemp seed has the richest variety of compounds, the greatest compound content, and the greatest odor intensity. Pyrazines (FD 32), dimethyl sulfoxide (FD 8), and acetamide (FD 8) give it a nutty, fatty, and sweet flavor.

Table 2. Retention indices, sensory description, FD factor, and concentration of volatile compounds in whole hemp seeds from four regions.

Nos.	Volatile Compounds [1]	RI [2]	Sensory Description [3]	Concentrations (μg/kg) [4]				FD Value [5]			
				GX	SX	HLJ	NMG	GX	SX	HLJ	NMG
	Alcohols (9)										
1	2-Methyl-3-buten-2-ol	1033	grassy	n.d.	49.76	n.d.	n.d.	n.d.	2	n.d.	n.d.
2	1,8-Cineole	1203	herb	n.d.	82.41	31.34	n.d.	n.d.	2	1	n.d.
3	1-Pentanol	1264	fatty	1.40	n.d.	n.d.	n.d.	4	n.d.	n.d.	n.d.
4	1-Hexanol	1369	sweet	3.24	n.d.	128.31	28.89	2	n.d.	2	1
5	2-Ethylhexanol	1508	fruity	0.70	n.d.	46.06	7.08	1	n.d.	2	2
6	1-Octen-3-ol	1443	mushroom	n.d.	100.51	n.d.	n.d.	n.d.	4	n.d.	n.d.
7	Linalool	1569	fruity	0.55	8.39	32.50	6.46	8	8	4	32
8	2,3-Butanediol	1588	cream	0.54	394.22	156.76	828.1	4	8	4	128
9	Phenethyl alcohol	1885	floral	n.d.	n.d.	n.d.	5.99	n.d.	n.d.	n.d.	2
	Aldehydes (2)										
10	Hexanal	1102	almond	3.93	n.d.	86.67	20.89	16	n.d.	64	32
11	Benzaldehyde	1536	lemon	47.43	n.d.	39.07	11.61	64	n.d.	64	8
	Ketones (2)										
12	6-Methyl-5-hepten-2-one	1392	fruity	n.d.	n.d.	n.d.	17.64	n.d.	n.d.	n.d.	1
13	(-)-α-Thujone	1411	woody	n.d.	34.85	n.d.	n.d.	n.d.	2	n.d.	n.d.
	Esters (3)										
14	Hexyl acetate	1301	fruity	0.62	n.d.	n.d.	n.d.	1	n.d.	n.d.	n.d.
15	Gamma butyrolactone	1515	sweet	n.d.	n.d.	n.d.	30.80	n.d.	n.d.	n.d.	2
16	4-Hexanolide	1713	coconut	n.d.	n.d.	98.53	n.d.	n.d.	n.d.	2	n.d.
	Terpenes (11)										
17	α-Pinene	1042	grassy	5.25	136.71	198.47	230.09	256	512	512	1024
18	Camphene	1077	orange	n.d.	n.d.	n.d.	2.08	n.d.	n.d.	n.d.	2
19	(1S)-(1)-β-Pinene	1126	grassy	3.78	76.82	101.97	121.49	256	256	256	128
20	Myrcene	1159	spice	n.d.	167.97	n.d.	183.07	n.d.	512	n.d.	512
21	(+)-Dipentene	1179	grassy	0.29	56.81	159.21	49.47	128	256	128	128
22	(*E*)-β-Ocimene	1246	floral	n.d.	68.74	36.03	5.11	n.d.	128	64	64
23	Styrene	1253	floral	n.d.	n.d.	n.d.	103.40	n.d.	n.d.	n.d.	2
24	Terpinolene	1274	orange	n.d.	73.59	n.d.	n.d.	n.d.	8	n.d.	n.d.
25	(-)-Isocaryophyllene	1595	spice	0.99	n.d.	n.d.	n.d.	64	n.d.	n.d.	n.d.
26	β-Caryophyllene	1620	woody	22.39	909.35	122.40	n.d.	512	512	512	n.d.
27	α-Caryophyllene	1662	woody	n.d.	342.33	n.d.	n.d.	n.d.	16	n.d.	n.d.
	Acids (4)										
28	Acetic acid	1454	acidity	4.13	n.d.	299.25	292.52	8	n.d.	16	4
29	Propionic acid	1548	acidity	n.d.	n.d.	76.33	7.63	n.d.	n.d.	2	1
30	Isovaleric acid	1605	cheese	n.d.	n.d.	n.d.	12.82	n.d.	n.d.	n.d.	4
31	Hexanoic acid	1861	acidity	10.16	249.41	703.45	87.32	4	8	64	4
	Furans (2)										
32	2-Pentylfuran	1249	fruity	n.d.	n.d.	21.15	n.d.	n.d.	n.d.	8	n.d.
33	Tetramethylpyrazine	1411	nutty	n.d.	n.d.	n.d.	2.43	n.d.	n.d.	n.d.	8
	Sulfur-containing compounds (1)										
34	Dimethyl sulfoxide	1504	mushroom	n.d.	n.d.	n.d.	9.11	n.d.	n.d.	n.d.	1
	Nitrogen-containing compounds (1)										
35	Acetamide	1702	salty	n.d.	n.d.	n.d.	4.45	n.d.	n.d.	n.d.	2

[1] Odorants were identified by comparing its odor quality and retention indices on capillary DB-wax with the data of the reference compounds. [2] Retention index. [3] Odor descriptions were from Flavor.net (www.flavornet.org, www.chemicalbook.com and www.thegoodscentscompany.com (accessed on 1 September 2022). n.d., not detected. [4] Compound content by semi-quantitative methods. [5] FD factor means flavor dilution factor determined on a DB-Wax column.

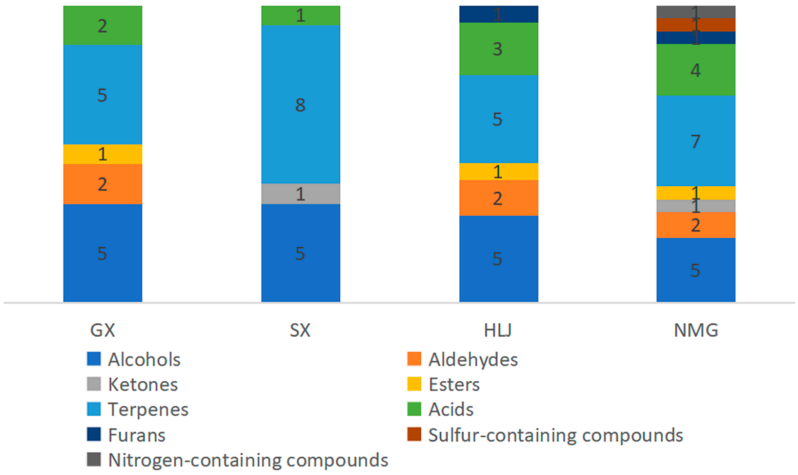

Figure 2. Classification of compounds and quantitative statistics of hemp seeds from four different geographic regions. The Arabic numerals on the bars indicate the numbers of corresponding compounded identified.

Comparing the volatile compound content (0.29–703.45 μg/kg) and FD factor (8–1024) of NMG with other three different geographical sources of hemp seed, we can conclude that linalool, α-pinene, myrcene, (+)-dipentene, (1*S*)-(1)-β-pinene, and hexanoic acid are the sources of the characteristic flavor compounds of fresh and grassy, but the different geographical sources give different characteristic flavors to the hemp seed. Since NMG hemp seed has the highest volatile content, more prominent and distinctive flavor, NMG hemp seed was selected for the subsequent study to press hemp seed oil.

3.1.2. GC-O-MS Analysis of Shelled Hemp Seed (SHS) and Unshelled Hemp Seed (USHS)

It is known from the research that all the current commercial hemp seed oils are obtained by shelled pressing, and according to the above, NMG hemp seeds were selected for the study to compare the active odor compounds of shelled and unshelled hemp seeds.

In the work, SAFE combined with GC-O-MS was used to analyze the volatile compounds of shelled and unshelled hemp seeds, and a total of 33 compounds were identified, 23 in SHS and 19 in USHS. Nine compounds were common to both samples, three alcohols, one aldehyde, and five terpenes. 1-Hexanol, 2-ethylhexanol, linalool and hexanal provided sweet and grassy flavor and almond aroma for both samples. It can be seen from Table 3 that the content of these three alcohols in USHS relatively increased while the content of compounds of hexanal decreased. Alcohols and aldehydes are associated with the degradative oxidation products of unsaturated fatty acids initiated by lipoxygenase and/or free radicals [34]. The results of the study are in agreement with Shen results that hexanal is the main oxidation product of linoleic acid and dehulling brings about secondary oxidation of hemp seed [35]. 2,3-Butanediol appeared in SHS and its relative content (8.38 ± 12.69 μg/g), FD factor (128) and OAV (6.91) reflected that it contributed an effective creamy flavor. Six compounds in the SHS had an OAV ≥ 1 and seven compounds in the USHS had an OAV ≥ 1, mainly concentrated in terpenoids. Terpenoids are the major constituents of volatile compounds in hemp seeds [36]. More terpenes were detected in USHS. α-Pinene, (1*S*)-(1)-β-pinene, myrcene, (+)-dipentene, and (*E*)-β-ocimene are terpene compounds they share. According to Table 3, combining the FD factor with the OAV shows that the overall odor of terpenes is heavier in USHS. Four terpenes had an OAV ≥ 1 in SHS and six in USHS. USHS have more terpenes and heterocyclic aromatics, and (+)-dipentene and terpinolene give USHS a grassy odor, as does p-cymene [37]. Four acidic compounds were detected in SHS, with acetic acid having an OAV ≥ 1, providing a sour flavor. Tetramethylpyrazine, dimethyl sulfoxide, and acetamide, which imparted the characteristic nutty, fatty flavor to NMG hemp seeds, had FD ≥ 4 although the OAV < 1, perhaps because of the larger threshold. They were all undetected in USHS.

Table 3. Retention index, sensory description, FD factor, volatile compound concentration, and OAV values for shelled and unshelled hemp seeds.

Nos.	Volatile Compounds	RI	Sensory Description	OT (μg/kg) [1]	Concentrations(μg/g) [2]		FD Factor [3]		OAV Values [4]	
					SHS [5]	USHS [6]	SHS	USHS	SHS	USHS
	Alcohols (9)									
1	pentanol	1213	fatty	3000	n.d.	0.86 ± 0.02	n.d.	4	n.d.	<1
2	1-Hexanol	1369	sweet	757.7	0.27 ± 0.07	0.92 ± 0.01	1	2	<1	<1
3	2-Ethylhexanol	1508	fruity	25,482	0.06 ± 0.03	0.26 ± 0.01	2	2	<1	<1
4	Linalool	1569	fruity	37	0.19 ± 0.26	0.35 ± 0.02	32	8	<1	<1
5	Fenchol	1583	woody	369.34	n.d.	0.27 ± 0.01	n.d.	16	n.d.	<1
6	2,3-Butanediol	1588	cream	668	8.38 ± 12.69	n.d.	128	n.d.	6.91	n.d.
7	Phenethyl alcohol	1885	floral	211	0.06 ± 0.07	n.d.	2	n.d.	<1	n.d.
	Aldehydes (2)									
8	Hexanal	1102	almond	80	2.96 ± 1.78	0.12 ± 0.01	32	64	<1	<1
9	Benzaldehyde	1536	lemon	60	0.15 ± 0.05	n.d.	8	n.d.	<1	n.d.
	Ketones (1)									
10	6-Methyl-5-hepten-2-one	1392	fruity	1000	7.95 ± 1.34	n.d.	1	n.d.	<1	n.d.
	Esters (1)									
11	γ-Butyrolactone	1515	sweet	12,500	0.68 ± 0.98	n.d.	2	n.d.	<1	n.d.
	Terpenes (11)									
12	α-Pinene	1042	grassy	274	1.10 ± 0.16	9.37 ± 0.19	1024	1024	4.2	7.69
13	Camphene	1077	orange	26,000	0.03 ± 0.01	n.d.	2	n.d.	<1	n.d.
14	(1S)-(1)- β -Pinene	1126	grassy	206.17	0.67 ± 0.02	4.19 ± 0.15	128	128	2.95	5.59
15	Myrcene	1159	spice	112.5	1.78 ± 0.14	21.20 ± 0.15	512	256	8.14	7.13
16	(+)-Dipentene	1179	grassy	507.66	0.40 ± 0.07	3.54 ± 0.01	128	128	<1	5.85
17	(*E*)- β -Ocimene	1220	floral	24.05	63.93 ± 30.54	0.06 ± 0.00	64	8	1.06	3.53

Table 3. *Cont.*

Nos.	Volatile Compounds	RI	Sensory Description	OT (μg/kg) [1]	Concentrations(μg/g) [2]		FD Factor [3]		OAV Values [4]	
					SHS [5]	USHS [6]	SHS	USHS	SHS	USHS
18	γ-Terpinene	1224	grassy	3.5	n.d.	0.15 ± 0.00	n.d.	2	n.d.	<1
19	(Z)- β-Ocimene	1236	fruity	600	n.d.	0.57 ± 0.01	n.d.	2	n.d.	<1
20	Terpinolene	1244	grassy	200	n.d.	5.54 ± 0.07	n.d.	16	n.d.	4.12
21	Styrene	1253	floral	3100	0.57 ± 0.09	n.d.	2	n.d.	<1	n.d.
22	(*E*,*Z*)-2,6-Dimethylocta-2,4,6-triene	1329	floral	38,000	n.d.	0.20 ± 0.01	n.d.	2	n.d.	<1
23	β-Caryophyllene	1532	sweet	640	n.d.	7.92 ± 0.39	n.d.	8	n.d.	<1
24	α-Caryophyllene	1600	woody	160	n.d.	0.33 ± 0.02	n.d.	2	n.d.	<1
	Acids (4)									
25	Acetic acid	1454	acidity	720	2.42 ± 0.46	n.d.	4	n.d.	2.93	n.d.
26	Propionic acid	1548	acidity	140	0.34 ± 0.49	n.d.	1	n.d.	<1	n.d.
27	Isovaleric acid	1605	cheese	61	0.25 ± 0.35	n.d.	4	n.d.	<1	n.d.
28	Hexanoic acid	1861	acidity	700	0.55 ± 0.68	n.d.	4	n.d.	<1	n.d.
	Benzene compounds (2)									
29	p-cymene	1229	grassy	11.4	n.d.	0.12 ± 0.00	n.d.	64	n.d.	3.93
30	4-Dimethylstyrene	1383	spice	85	n.d.	0.10 ± 0.01	n.d.	2	n.d.	<1
	Furans (1)									
31	Tetramethylpyrazine	1411	nutty	38,000	0.04 ± 0.04	n.d.	32	n.d.	<1	n.d.
	Sulfur-containing compounds (1)									
32	Dimethyl sulfoxide	1504	mushroom	100	0.52 ± 0.52	n.d.	8	n.d.	<1	n.d.
	Nitrogen-containing compounds (1)									
33	Acetamide	1702	salty	140,000	0.14 ± 0.18	n.d.	4	n.d.	<1	n.d.

[1] The thresholds of compounds in oil referred to ref odor thresholds are determined according to the book Compilations of Flavour Threshold Values in Water and Other Media (Second Enlarged and Revised Edition). Thresholds not found are self-tested using the 3-AFC method in Section 2.6.5. [2] Calculation of compound content (μg/g) by internal standard method. [3] FD factor means flavor dilution factor determined on a DB-Wax column. [4] Odor activity values were calculated by dividing the concentration by the odor threshold. [5] USHS: unshelled hemp seeds. [6] SHS: shelled hemp seeds.

In order to retain a more complete flavor of NMG hemp seed, SHS was selected for the subsequent roasting and pressing of hemp seed oil for the study.

3.2. Active Odor Compounds of Hemp Seed Oils

According to Section 3.1., the SHS from NMG was selected for roasting experiments. The NMG hemp seeds were roasted at different temperatures (120 °C, 140 °C, 160 °C, and 180 °C) for 20 min and then immediately put into a screw press machine to extract the hemp seed oil. The oil was subjected to sensory evaluation with raw samples, and the odor-active compounds of the oil were extracted using SAFE and analyzed in combination with GC-O-MS. OAV values were calculated, and recombination and deletion experiments and data analyses were performed to determine the key odor compounds.

3.2.1. Sensory Evaluation

Figure 3 shows the sensory evaluation results of the raw samples and four HSO samples. In the HS, the seeds scored the highest at 6.9, while the sweet scored the lowest at 2. Maximum retention of the seeds of hemp seed was scored at 120 °C HSO with a score of 6, and the score for sweet was the lowest, at 2.2. In the 140 °C HSO, the fatty score was the highest, at 5.3, and the rancid score was the lowest, at 1.1. In the 160 °C HSO, the sweet score was the highest at 5.3, and the pungent score was the lowest at 2.2. In the 180 °C HSO, the nutty score was the highest at 7.8, while the herbs scored the lowest at 2. In this study, as the roasting temperature of the samples increased, the sweet, fatty, and nutty flavors of the samples increased with roasting time, while the herbs and seeds of the raw sample that had not been roasted gradually decreased relatively with roasting temperature.

Hemp seed oil has a nutty flavor and slight bitterness [38], and by roasting hemp seed, it will have a better flavor and aroma [39]. It has been found that almonds have more greasy and toasted flavor compounds produced by a Maillard reaction through oil roasting [40]. Canola oil improves sour flavor and increases toasted and fatty flavors through roasting [41]. Additionally, red pepper seeds can also impart sweet and roasted flavors to the seeds through thermal processing [41]. There was a tendency for both sweet and toasted flavors to increase in the roasted treated seed samples with increasing roasting temperature and time. In addition, sweetness, which is one of the characteristics of hemp seed oil flavor compounds, increased at lower roasting temperatures than the raw sample sweet, but the plant's characteristic seeds

and herbs flavors tended to decrease with increasing roasting temperatures, presumably because they were masked by sweet and roasting aroma.

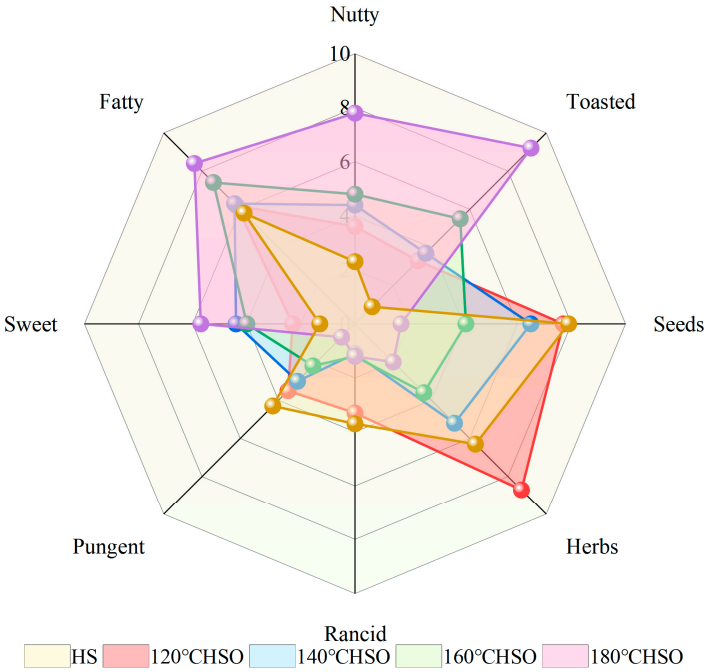

Figure 3. Sensory evaluation of five samples with different processing. (HS: Raw sample from Inner Mongolian hemp seeds in shell; 120 °C HSO: hemp seed oil obtained by pressing raw samples after roasting at 120 °C; 140 °C HSO: hemp seed oil obtained by pressing raw samples after roasting at 140 °C; 160 °C HSO: hemp seed oil obtained by pressing raw samples after roasting at 160 °C; 180 °C HSO: hemp seed oil obtained by pressing raw samples after roasting at 180 °C).

3.2.2. Identification of Odor-Active Compounds in Hemp Seed Oil

According to Table 4 and Figure 4, a total of 69 active odor compounds were detected in the five samples (HS, 120 °C HSO, 140 °C HSO, 160 °C HSO, 180 °C HSO) of which 23 compounds were detected in the HS, 34 compounds in the 120 °C HSO, 27 compounds in the 140 °C HSO, 32 compounds in the 160 °C HSO, and 41 compounds in the 180 °CHSO, and the number of extracted flavor compounds of hemp seed oil showed a general increasing trend with the increase of the roasting temperature of the hemp seed.

Table 4. Retention index, odor description, and volatile compound concentration for five samples with different processing.

Nos.	Volatile Compounds	RI	Sensory Description	Concentrations(μg/g) [1]				
				HS	120 °C HSO	140 °C HSO	160 °C HSO	180 °C HSO
	Alcohols (10)							
1	1-Penten-3-ol	1154	grassy	n.d.	n.d.	n.d.	n.d.	0.65 ± 0.01
2	1-Pentanol	1231	fatty	n.d.	n.d.	n.d.	6.91 ± 0.63	1.08 ± 0.02
3	1-Hexanol	1369	sweet	0.27 ± 0.07	3.1 ± 0.64	n.d.	2.09 ± 0.26	3.77 ± 0.04
4	1-Octen-3-ol	1435	mushroom	n.d.	1.5 ± 0.03	4.92 ± 0.44	8.26 ± 1.09	2.45 ± 0.01
5	2-Ethylhexanol	1448	sweet	0.06 ± 0.03	n.d.	n.d.	n.d.	n.d.
6	Linalool	1527	floral	0.19 ± 0.26	1.9 ± 1.21	n.d.	n.d.	n.d.
7	1-Octanol	1535	floral	n.d.	n.d.	n.d.	n.d.	0.51 ± 0.00
8	2,3-Butanediol	1588	sweet	8.38 ± 12.69	n.d.	n.d.	n.d.	n.d.
9	Furfuryl alcohol	1630	acidity	n.d.	5.0 ± 2.55	15.95 ± 0.87	34.52 ± 3.68	n.d.
10	Phenethyl alcohol	1805	floral	0.06 ± 0.0745	n.d.	n.d.	n.d.	n.d.
	Aldehydes (15)							
11	Hexanal	1056	fruity	2.96 ± 1.78	12.1 ± 0.12	12.18 ± 1.60	12.77 ± 0.44	4.72 ± 0.03
12	Heptaldehyde	1084	fatty	n.d.	n.d.	n.d.	n.d.	0.72 ± 0.04
13	(*E*)-2-Hexenal	1096	grassy	n.d.	2.1 ± 4.43	ND	8.63 ± 0.64	26.25 ± 1.75
14	Octanal	1266	orange	n.d.	n.d.	n.d.	n.d.	2.16 ± 0.99
15	(*E*)-2-Heptenal	1296	vegetables	n.d.	n.d.	n.d.	n.d.	6.98 ± 0.03
16	(*E*)-2-Octenal	1396	greasy	n.d.	0.5 ± 0.15	1.85 ± 0.2	n.d.	1.48 ± 0.01
17	(*E*,*E*)-2,4-Heptadienal	1431	greasy	n.d.	2.7 ± 0.13	5.58 ± 0.32	210.48 ± 27.24	5.90 ± 0.05
18	Furfural	1432	sweet	n.d.	n.d.	4.12 ± 0.32	14.34 ± 1.84	3.67 ± 0.04
19	Benzaldehyde	1479	nutty	0.15 ± 0.05	n.d.	n.d.	n.d.	n.d.
20	(*E*)-2-Nonenal	1501	grassy	n.d.	n.d.	n.d.	n.d.	2.35 ± 0.07
21	5-Methyl furfural	1536	spice	n.d.	1.1 ± 4.59	3.03 ± 0.27	8.95 ± 1.28	0.28 ± 0.00

Table 4. *Cont.*

Nos.	Volatile Compounds	RI	Sensory Description	Concentrations(μg/g) [1]				
				HS	120 °C HSO	140 °C HSO	160 °C HSO	180 °C HSO
22	(*E*)-2-Decenal	1636	grassy	n.d.	n.d.	n.d.	n.d.	2.71 ± 0.02
23	(*E*,*E*)-2,4-Nonadienal	1692	floral	n.d.	n.d.	n.d.	n.d.	2.78 ± 0.1
24	(*E*,*Z*)-2,4-Decadienal	1730	greasy	n.d.	n.d.	n.d.	10.45 ± 2.39	13.66 ± 0.24
25	(*E*,*E*)-2,4-Decadienal	1775	grassy	n.d.	1.9 ± 2.47	n.d.	9.63 ± 1.50	1.41 ± 0.12
	Ketones (6)							
26	2,3-Pentanedione	1093	creamy	n.d.	n.d.	n.d.	1.99 ± 0.16	n.d.
27	2-Heptanone	1164	sweet	n.d.	8.0 ± 0.16	n.d.	n.d.	0.86 ± 0.02
28	2-Octanone	1262	grassy	n.d.	1.2 ± 0.02	1.14 ± 0.13	n.d.	n.d.
29	3-Octanone	1288	fruity	n.d.	n.d.	n.d.	n.d.	1.49 ± 0.02
30	Hydroxyacetone	1286	caramel	n.d.	n.d.	n.d.	n.d.	4.62 ± 4.99
31	Methylheptenone	1312	lemon	7.95 ± 1.34	n.d.	n.d.	n.d.	n.d.
	Esters (1)							
32	γ-Butyrolactone	1515	sweet	0.68 ± 0.98	n.d.	n.d.	n.d.	n.d.
	Terpenes (13)							
33	α-Pinene	1017	sweet	1.10 ± 0.16	3.1 ± 0.08	3.85 ± 0.50	12.83 ± 1.67	6.97 ± 0.15
34	Camphene	1207	grassy	0.03 ± 0.01	n.d.	n.d.	n.d.	n.d.
35	(1S)-(1)- β -Pinene	1094	grassy	0.67 ± 0.02	2.2 ± 0.01	2.92 ± 0.39	8.63 ± 0.57	0.32 ± 0.02
36	3-Carene	1142	woody	n.d.	0.1 ± 0.14	0.28 ± 0.15	n.d.	n.d.
37	Myrcene	1152	fruity	1.78 ± 0.14	20.0 ± 0.94	12.60 ± 1.54	28.08 ± 2.39	0.89 ± 1.17
38	(+)-Dipentene	1179	orange	0.40 ± 0.07	2.4 ± 0.06	1.62 ± 0.19	4.25 ± 0.38	0.27 ± 0.03
39	β -Phellandrene	1180	cedarwood	n.d.	19.0 ± 1.61	n.d.	n.d.	n.d.
40	(*E*)- β -Ocimene	1220	floral	63.93 ± 30.54	0.9 ± 1.31	3.86 ± 0.58	4.25 ± 0.39	32.21 ± 15.20
41	γ-Terpinene	1224	grassy	n.d.	0.1 ± 0.01	n.d.	n.d.	n.d.
42	Styrene	1213	sweet	0.57 ± 0.09	n.d.	n.d.	n.d.	n.d.
43	(*Z*)- β -ocimene	1236	fruity	n.d.	3.15 ± 6.20	n.d.	n.d.	n.d.
44	Terpinolene	1244	grassy	n.d.	4.1 ± 0.13	3.74 ± 0.40	162.51 ± 19.25	n.d.
45	β -Caryophyllene	1517	sweet woody	n.d.	4.8 ± 0.03	n.d.	5.12 ± 0.90	0.99 ± 0.02
	Acids (5)							
46	Acetic acid	1414	spice	2.42 ± 0.46	n.d.	27.97 ± 2.20	177.89 ± 31.36	n.d.
47	Propionic acid	1500	spice	0.34 ± 0.49	n.d.	n.d.	n.d.	n.d.
48	Butyric Acid	1590	cheese	n.d.	0.04 ± 0.00	n.d.	n.d.	n.d.
49	Isovaleric acid	1605	cheese	0.25 ± 0.35	n.d.	n.d.	n.d.	n.d.
50	Hexanoic acid	1813	acidity	0.55 ± 0.68	3.6 ± 0.04	0.56 ± 0.08	2.27 ± 0.36	16.18 ± 10.65
	Furans (1)							
51	2-Pentylfuran	1215	sweet	n.d.	1.7 ± 0.10	2.10 ± 1.74	3.00 ± 0.36	1.92 ± 0.06
	Pyrroles (2)							
52	Pyrrole	1483	nutty	n.d.	n.d.	n.d.	7.62 ± 1.02	n.d.
53	2-Acetyl pyrrole	1921	caramel	n.d.	n.d.	1.72 ± 0.10	2.04 ± 0.34	n.d.
	Pyrazines (11)							
54	2-Methylpyrazine	1247	nutty	n.d.	14.0 ± 5.50	27.39 ± 4.14	n.d.	2.43 ± 0.09
55	2,5-Dimethylpyrazine	1303	roasty	n.d.	9.90 ± 1.67	9.97 ± 1.08	270.10 ± 24.01	26.66 ± 34.49
56	2,6-Dimethylpyrazine	1310	fatty	n.d.	3.3 ± 2.72	5.07 ± 0.57	n.d.	6.57 ± 8.55
57	Ethylpyrazine	1314	roasty	n.d.	2.4 ± 1.59	2.89 ± 0.32	2.74 ± 0.32	0.03 ± 0.00
58	2,3-Dimethylpyrazine	1324	nutty	n.d.	1.8 ± 0.25	2.38 ± 0.39	3.51 ± 0.44	0.04 ± 0.00
59	2-ethyl-6-methylpyrazine	1363	roasty	n.d.	1.4 ± 0.03	2.18 ± 0.22	3.30 ± 0.46	0.01 ± 0.00
60	2-Ethyl-5-methylpyrazine	1368	coffee	n.d.	4.9 ± 0.08	1.85 ± 0.17	4.30 ± 0.78	0.06 ± 0.00
61	Trimethyl-pyrazine	1380	roasty	n.d.	5.4 ± 0.11	6.51 ± 0.63	10.05 ± 1.29	n.d.
62	3-Ethyl-2,5-diMethylpyrazine	1410	roasty	n.d.	n.d.	n.d.	9.33 ± 1.27	8.96 ± 11.27
63	Tetramethylpyrazine	1411	nutty	0.04 ± 0.04	n.d.	n.d.	n.d.	n.d.
64	Acetylpyrazine	1581	caramel	n.d.	n.d.	n.d.	2.74 ± 0.49	n.d.
	Pyridine (3)							
65	2-Ethylpyridine	1272	fatty	n.d.	n.d.	n.d.	n.d.	3.29 ± 0.08
66	3-Ethylpyridine	1361	fatty	n.d.	n.d.	n.d.	n.d.	0.21 ± 0.02
67	2-Pentylpyridine	1522	mushroom	n.d.	n.d.	n.d.	n.d.	2.25 ± 0.01
	Sulfur-containing compounds (1)							
68	Dimethyl sulfoxide	1504	mushroom	0.52 ± 0.52	n.d.	n.d.	n.d.	n.d.
	Nitrogen-containing compounds (1)							
69	Acetamide	1702	odor	0.14 ± 0.18	n.d.	n.d.	n.d.	n.d.

[1] Concentrations (μg/g) determined by internal standard method.

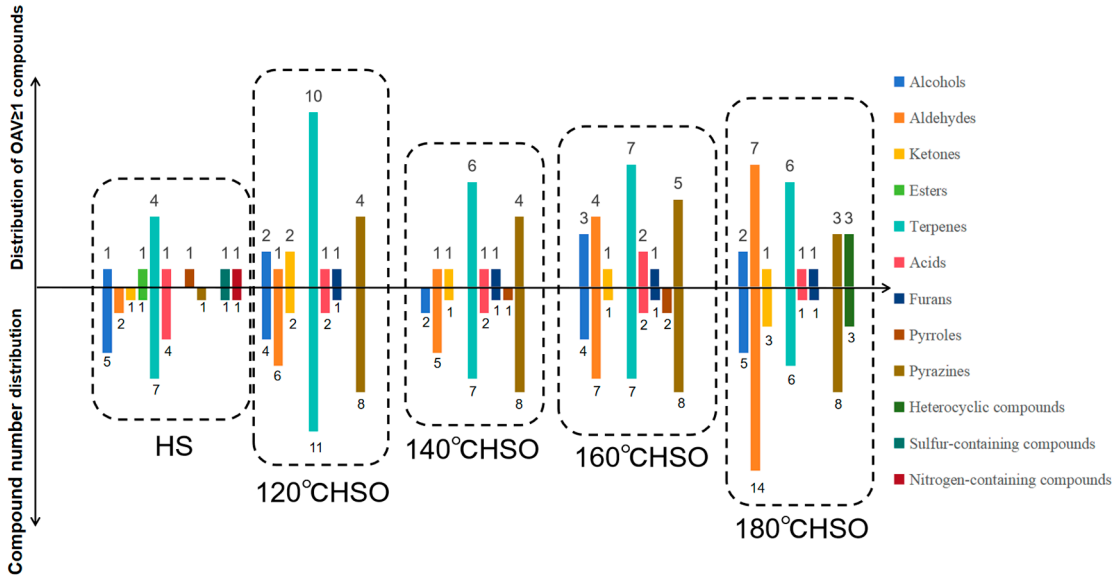

Figure 4. Classification and number counts of 69 compounds in 5 samples and OAV ≥ 1 compounds. The Arabic numerals indicate the number of corresponding compounded identified.

According to Figure 5, it can be seen that there are fourteen unique compounds of HS, four unique compounds at 120 °C HSO, three unique compounds at 160 °C HSO, and thirteen unique compounds at 180 °C HSO, which shows a trend of increasing, then decreasing, and then increasing. With the increase of roasting temperature, the characteristic flavor of hemp seed oil changes. Unique compounds are concentrated in terpenes, acids, and heterocyclic compounds. Seven compounds were identical in the five samples. As can be seen from Table 4, there were hexanal, α-pinene, (1*S*)-(1)-β-pinene, myrcene, (+)-dipentene, (*E*)-β-ocimene, and hexanoic acid. The terpenes were higher than the other kinds of compounds, especially α-pinene, myrcene, and (*E*)-β-ocimene. The main volatile compounds of HS are terpenes, which are mainly synthesized naturally in the chloroplasts and cytoplasm of plant cells [42]. They can synergistically interact with cannabinoids in hemp seeds to produce a unique odor in plants [43]. The main volatile compounds extracted from cannabis (Cannabis sativa L) also include a variety of terpenes such as α-pinene, β-pinene, terpinolene, α-caryophyllene, and β-caryophyllene [44,45]. Key volatile compounds of hemp seed oil also include terpenes such as α-pinene, β-pinene, myrcene, terpinolene, α-caryophyllene, and β-caryophyllene, which exhibit fresh, herbal, and lemony aromas [34,46,47]. It is worth mentioning that a large amount of β-phellandrene is produced in 120 °C HSO. β-phellandrene is a natural compound found in various volatile oils such as eucalyptus oil, fennel oil, cinnamon leaf oil, orange peel oil, pepper, bay oil, elephantine oil, etc. [48–50]. According to Table 4, the content of limonene and terpinolene reaches its maximum in 160 °C HSO (4.25 ± 0.38 μg/g). This is consistent with Hanus' research conclusion that the main volatile compounds in hemp seeds include monoterpenes, sesquiterpenes, and their oxygenated derivatives, with the highest content of myrcene, (+)-limonene, and β-caryophyllene [51]. At high temperatures, the C-C double bond undergoes ring strain through dissociation. β-pinene and α-pinene are partially degraded to monocyclic structural olefins [52]. Content of their compounds shows an increasing and then decreasing trend with increasing roasting temperature, with 160 °C HSO having the highest content of terpene compounds.

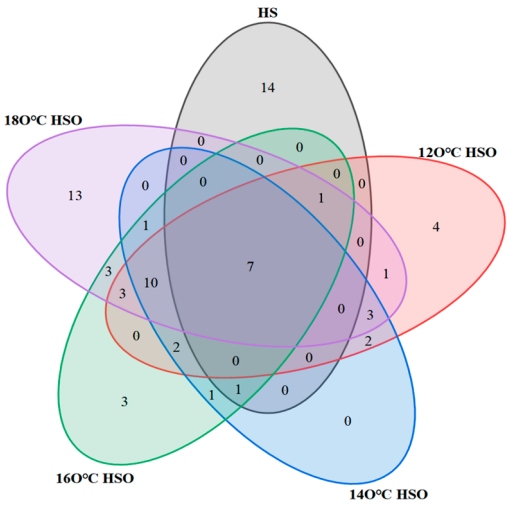

Figure 5. The Venn diagram of 69 total volatile compounds in five samples. The Arabic numerals indicate the numbers of compounds shared by different samples.

The four hemp seed oils obtained after roasting at different temperatures had a total of 16 compounds identical to each other. In addition to the above seven, 1-octen-3-ol, (*E*,*E*)-2,4-heptadienal, 5-methylfurfural, 2-pentylfuran, 2,5-dimethylpyrazine, ethylpyrazine, 2,3-dimethylpyrazine, 2-ethyl-6-methylpyrazine, and 2-ethyl-5-methylpyrazine were present, due to the Metabolic reaction produced by roasting. During the roasting process, Metabolic

reactions and lipid oxidation result in the formation of a large number of volatile aromatic compounds, including aldehydes, ketones, and pyrazines. The source of these aldehydes is mostly formed by degradation reactions of amino acids or oxidation of unsaturated fatty acids, which play an important role in the flavor improvement of roasted seeds. Hexenal is the key flavor compound in hemp seeds and contributes a high value to the specific flavor of hemp seeds [10]. (*E*)-2-hexenal, (*E*)-2-octenal, (*E*,*E*)-2,4-heptadienal, furfural, 5-methylfurfural, and (*E*,*E*)-2,4-decadienal occur in hemp seed oil obtained after roasting. In the oxidation of oils and fats, oxidative degradation of oleic, linoleic, and linolenic acids is the main cause of changes in aroma attributes, and the volatiles in oils and fats have a certain correlation with their fatty acid compositions [53]. Most of the aldehydes listed above are converted from linoleic acid, which is an oxidized product obtained by heating vegetable oils [54]. Oxidative cleavage reactions of unsaturated fatty acids are generally accompanied by the production of alcohols in addition to aldehydes. Hexanol, 1-octen-3-ol, and furfuryl alcohol have been detected in creosote. 1-octen-3-ol is derived from lipid β-oxidation [55]. Hexanol may be derived from the oxidative cleavage of unsaturated fatty acids during reheating. Furfural is a key intermediate in the formation of furfuryl alcohol, and furfuryl aldehyde yields furfural [56]. They are all products of the Maillard reaction, and the compound content of furfural and furfuryl alcohol increased with increasing roasting temperature. In 160 °C HSO, 2,3-pentanedione was produced by roasting. This is a completely new compound produced by heating the sucrose in the vegetable oil and is generated throughout the roasting process mainly through the intact sugar skeleton [57].

Heterocyclic compounds are important flavor compounds associated with roasting, including furans, pyridines, pyrazines, etc., and are usually produced during the heat treatment of food products through a Maillard reaction [58]. The content of heterocyclic compounds also increased, with 180 °C HSO having the largest content and the largest variety of heterocyclic compounds. Furan formation can occur through lipid oxidation, carbohydrate degradation, and Maillard re-activation. 2-Pentylfuran, a typical furan compound, is produced by oxidation of linoleic acid and is considered a marker of lipid oxidation in vegetable oils with a grassy aroma [59]. The sugars in oilseeds form dicarbonyl compounds during heat treatment, which then react with peptides or free amino acids to produce alpha aminoketones, ultimately forming pyrazines through condensation [60]. The highest level of 2,5-dimethylpyrazine was detected at 160 °C HSO, with nutty and toasted flavors. 2,3,5-Trimethylpyrazine can be produced by heating threonine, and 2,5-dimethylpyrazine and 2,3,5-trimethylpyrazine are key aromatic compounds in hot pressed rapeseed oil [41]. 3-Ethyl-2,5-methylpyrazine is synthesized by a condensation reaction between two amino ketones, or one amino aldehyde and one amino ketone, and then reacts with hexanal to form dihydropyrazine as an intermediate, with earthy and baking flavors [61]. 2-Pentylpyridine is a typical product produced by the thermal reaction condensation of amino acids with 2,4-decadienal. It forms a pyridine ring through electrocyclic reaction and aromatization and has a unique grassy aroma [62].

3.2.3. Quantitation of Odor-Active Compounds in Hemp Seed Oil

The contribution of a compound to the aroma of a sample depends not only on the content of the compound, but also on the odor threshold of the compound. OAV is the ratio of the concentration of an aroma presenting substance in hemp seed oil to the threshold value of the substance in the oil. OAV reflects the intensity of the aroma-presenting substance, and OAV ≥ 1 will theoretically be perceived as a substance [63]. When the OAV ≥ 1, the compound has a significant degree of contribution to the aroma of the sample. The key odor compound intensity in hemp seed oils was determined by using the AEDE method to measure the FD factor and the OAV calculation method. The number of compounds with OAV ≥ 1 in

the five samples is 6, 21, 14, 23, and 24, respectively, and the number of compounds with FD $\geq$ 4 is 17, 23, 14, 20, and 30, respectively. As we can see from Figure 6, in the five samples, the number of compounds with OAV $\geq$ 1 and FD $\geq$ 4 both show an upward trend.

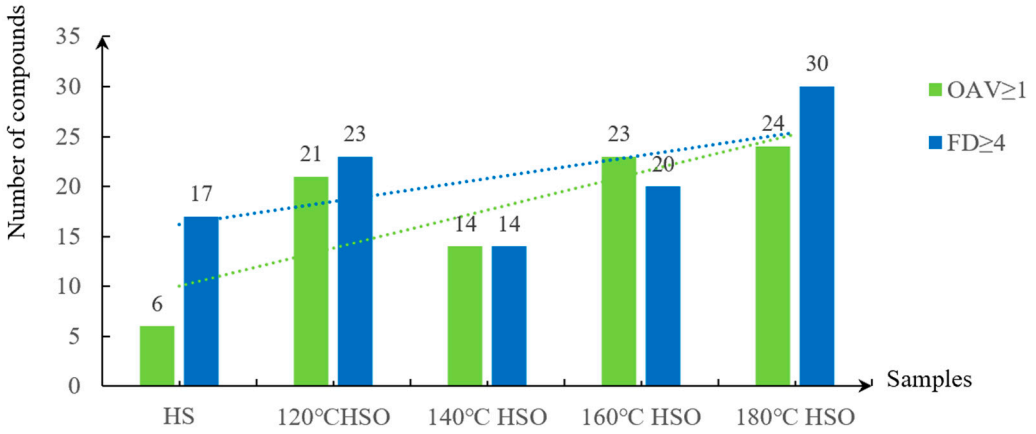

Figure 6. Number of compounds with OAV $\geq$ 1 and FD $\geq$ 4 in five different samples.

The specific results are shown in Table 5. It can be seen that α-pinene, (1*S*)-(1)-β-pinene, myrcene, and (*E*)-β-ocimene were the common compounds of the five samples, and they had OAV $\geq$ 1. With OAV values between 1.06 and 216.01 and FD values between 32 and 1024, they had a high value of contribution to the flavor of hemp seeds. The above compounds provide a refreshing herbal aroma to hemp and are considered the primary aroma compounds [17]. In roasted hemp seeds, hexanal, (*E*,*Z*)-2,4-decadienal, and (*E*)-2-heptenal were also found to have large OAV values ranging from 29.05 to 217.14 and FD values ranging from 2 to 1024, and they provided fatty flavor to the hemp seed oil [64]. Both their OAV value and FD factors increased with increasing temperature, and the two have a corresponding relationship, thus showing that they are very important odor compounds in the roasting process of hemp seeds. In 160 °C HSO, the FD factor of hexanal, (*E*,*Z*)-2,4-decadienal, and (*E*,*E*)-2,4-decadienal were more prominent. Among the ketones, 2,3-butanedione appeared at 160 °C HSO with a FD of 64 and an OAV of 3.41, with an attractive butter aroma, and the content increased considerably during roasting of rapeseed oil [65]. Acids had a significantly larger FD factor in HS, with acetic and propionic acids occurring only in HS. Hexanoic acid had the largest FD factor in 120 °C HSO, and hexanoic acid, which adds a pungent odor to hemp seed oil, had the largest OAV value in 180 °C HSO. Therefore, in the choice of temperature for roasting hemp seeds, attention should be paid to the size of the roasting temperature. Roasting temperatures that are too low or too high will produce negative odors that are detrimental to the overall flavor of the hemp seed oil. Heterocyclic compounds appeared after roasting of hemp seeds, and the FD factor from 2 to 128 and OAV from 1.94 to 52.04 increased with increasing roasting time, with furans and pyrazines being more prominent. 2-Pentylfuran provides caramel flavor; 2,5-dimethylpyrazine, 2-ethyl-6-methylpyrazine, and 2-ethyl-5-methylpyrazine provide toasted flavor. It is worth mentioning that the highest FD factor and OAV values of acetyl pyrazine that appeared in 160 °C HSO were the largest among all compounds with 2048 and 162.52, respectively, which provided a strong caramelized aroma to the 160 °C HSO. In 180 °C HSO, 2-ethylpyridine and 2-pentylpyridine have OAV $\geq$ 1. They are flavor compounds unique to 180 °C HSO, with 2-ethylpyridine having a greasy flavor and 2-pentylpyridine having a meaty flavor [66]. They are both produced by a Metabolic reaction and have a low threshold in the oil that can be easily detected by smell, so the roasted and fatty aroma of 180 °C HSO is more prominent than any other samples. As can be seen from Table 4, most of the compounds have the same trend of OAV value change and FD factor change, but there are exceptions. For example, the FD factor of dimethyl sulfoxide and acetamide is greater than 8,

but the OAV value is less than 1. This is due to the difference between AEDA sniffing and OAV calculation in analyzing the aroma active substances. The matrix and threshold of the active odor compounds will bring a certain impact on the calculation of OAV value, and the difference in the analysts' sniffing will also affect the analysis results. The differences can be partly attributed to the fact that these compounds may have a very high odor threshold. It could be related to the difference of thresholds in air and water; the oil matrix may influence the aroma release. Noteworthily, the flavor of each compound was dependent not only on its concentration but also on its interaction with the surrounding matrix due to the synergistic and antagonistic effects (masking effect) of a single compound [67]. As can be seen from Table 5, the thresholds of tetramethylpyrazine and acetamide in oil are extremely high. Considering their low contents in hemp seed samples, the calculated OAVs are less than 1. According to Wang's research on Longjing tea, it is speculated that dimethyl sulfoxide is mainly formed by Strecker degradation and subsequent oxidation processes, exhibiting high odor intensity characteristics [68]. Therefore, even at low concentrations of dimethyl sulfoxide in hemp seeds, its FD value is still greater than 4, providing an effective odor in hemp seeds.

Table 5. FD factor and quantitative results of OAV calculation for five samples with different processing.

Nos.	Volatile Compounds	OT [1] (μg/kg)	OAV Value [2]					FD Factor [3]				
			HS	120 °C HSO	140 °C HSO	160 °C HSO	180 °C HSO	HS	120 °C HSO	140 °C HSO	160 °C HSO	180 °C HSO
	Alcohols (10)											
1	1-Penten-3-ol	400	n.d.	n.d.	n.d.	n.d.	1.88	n.d.	n.d.	n.d.	n.d.	32
2	1-Pentanol	3000	n.d.	n.d.	n.d.	1.92	<1	n.d.	n.d.	n.d.	2	4
3	1-Hexanol	757.7	<1	3.26	n.d.	2.22	8.14	128	16	n.d.	4	4
4	1-Octen-3-ol	32,000	n.d.	<1	<1	<1	<1	n.d.	2	1	4	2
5	2-Ethylhexanol	25,482	<1	n.d.	n.d.	n.d.	n.d.	2	n.d.	n.d.	n.d.	n.d.
6	Linalool	37	<1	7.35	n.d.	n.d.	n.d.	8	32	n.d.	n.d.	n.d.
8	2,3-Butanediol	668	6.91	n.d.	n.d.	n.d.	n.d.	64	n.d.	n.d.	n.d.	n.d.
9	Furfuryl alcoho	15,000	n.d.	<1	<1	1.03	n.d.	n.d.	2	1	2	n.d.
10	Phenethyl alcohol	211	<1	n.d.	n.d.	n.d.	n.d.	8	n.d.	n.d.	n.d.	n.d.
	Aldehydes (13)											
11	Hexanal	80	<1	29.05	90.93	113.98	90.56	8	64	256	512	512
12	Heptaldehyde	500	n.d.	n.d.	n.d.	n.d.	1.49	n.d.	n.d.	n.d.	n.d.	8
15	(*E*)-2-Heptenal	50	n.d.	n.d.	n.d.	n.d.	121.69	n.d.	n.d.	n.d.	n.d.	64
16	(*E*)-2-Octenal	7000	n.d.	<1	<1	n.d.	<1	n.d.	2	1	n.d.	2
17	(*E*,*E*)-2,4-Heptadienal	10,000	n.d.	<1	<1	<1	<1	n.d.	4	1	1	2
18	Furfural	15,157	n.d.	n.d.	<1	<1	<1	n.d.	n.d.	1	2	2
19	Benzaldehyde	60	<1	n.d.	n.d.	n.d.	n.d.	16	n.d.	n.d.	n.d.	n.d.
20	(*E*)-2-Nonenal	150	n.d.	n.d.	n.d.	n.d.	8.8	n.d.	n.d.	n.d.	n.d.	8
21	5-Methyl furfural	16,000	n.d.	<1	<1	<1	<1	n.d.	2	2	4	4
22	(*E*)-2-Decenal	3220	n.d.	n.d.	n.d.	n.d.	<1	n.d.	n.d.	n.d.	n.d.	2
23	(*E*,*E*)-2,4-Nonadienal	1500	n.d.	n.d.	n.d.	n.d.	<1	n.d.	n.d.	n.d.	n.d.	2
24	(*E*,*Z*)-2,4-Decadienal	10	n.d.	n.d.	n.d.	217.14	165.2	n.d.	n.d.	n.d.	1024	256
25	(*E*,*E*)-2,4-Decadienal	2500	n.d.	<1	n.d.	3.44	<1	n.d.	4	n.d.	8	4
	Ketones (6)											
26	2,3-Pentanedione	300	n.d.	n.d.	n.d.	3.41	n.d.	n.d.	n.d.	n.d.	64	n.d.
27	2-Heptanone	300	n.d.	2.8	n.d.	n.d.	6.18	n.d.	8	n.d.	n.d.	32
28	2-Octanone	510	n.d.	3.45	3.73	n.d.	n.d.	n.d.	2	1	n.d.	n.d.
29	3-Octanone	21,400	n.d.	n.d.	n.d.	n.d.	<1	n.d.	n.d.	n.d.	n.d.	2
30	Hydroxyacetone	10,000	n.d.	n.d.	n.d.	n.d.	<1	n.d.	n.d.	n.d.	n.d.	4
31	Methylheptenone	1000	<1	n.d.	n.d.	n.d.	n.d.	2	n.d.	n.d.	n.d.	n.d.
	Esters (1)											
32	γ-Butyrolactone	12,500	<1	n.d.	n.d.	n.d.	n.d.	8	n.d.	n.d.	n.d.	n.d.
	Terpenes (13)											
33	α-Pinene	274	4.2	20.35	14.74	46.98	35.05	512	1024	1024	1024	1024
34	Camphene	26,000	<1	n.d.	n.d.	n.d.	n.d.	2	n.d.	n.d.	n.d.	n.d.
35	(1S)-(1)-beta-Pinene	206.17	2.95	18.82	16.51	47.98	5.05	128	128	64	128	128
36	3-Carene	9300	n.d.	<1	<1	n.d.	n.d.	n.d.	2	2	n.d.	n.d.
37	Myrcene	112.5	8.14	194.19	95.03	216.01	33.69	64	1024	256	256	512
38	(+)-Dipentene	507.66	<1	10.34	3.9	10.99	1.95	32	256	512	512	128
39	β-Phellandrene	36	n.d.	13.43	n.d.	n.d.	n.d.	n.d.	128	n.d.	n.d.	n.d.
40	(*E*)-β-Ocimene	24.05	1.06	87.88	16.78	42.77	43.66	128	256	32	128	128
41	γ-Terpinene	3.5	n.d.	131.4	n.d.	n.d.	n.d.	n.d.	256	n.d.	n.d.	n.d.
42	Styrene	3100	<1	n.d.	n.d.	n.d.	n.d.	32	n.d.	n.d.	n.d.	n.d.
43	(*Z*)-β-ocimene	600	n.d.	9.11	n.d.	n.d.	n.d.	n.d.	32	n.d.	n.d.	n.d.
44	Terpinolene	200	n.d.	25.87	16.49	43.07	n.d.	n.d.	16	16	32	n.d.
45	β-Caryophyllene	640	n.d.	11.85	n.d.	9.76	5.17	n.d.	32	n.d.	64	64
	Acids (5)											
46	Acetic acid	720	2.93	n.d.	14.76	33.51	n.d.	256	n.d.	2	1	n.d.
47	Propionic acid	140	<1	n.d.	n.d.	n.d.	n.d.	8	n.d.	n.d.	n.d.	n.d.
48	Butyric Acid	66.2	n.d.	<1	n.d.	n.d.	n.d.	n.d.	1	n.d.	n.d.	n.d.
49	Isovaleric acid	61	<1	n.d.	n.d.	n.d.	n.d.	2	n.d.	n.d.	n.d.	n.d.
50	Hexanoic acid	700	<1	4.05	<1	2.78	13.84	32	128	2	8	8
	Furans (1)											
51	2-Pentylfuran	100	n.d.	12.18	22.55	40.56	52.04	n.d.	32	64	64	64
	Pyrroles (2)											
52	Pyrrole	20,000	n.d.	n.d.	n.d.	<1	n.d.	n.d.	n.d.	n.d.	2	n.d.
53	2-Acetyl pyrrole	58,585	n.d.	n.d.	<1	<1	n.d.	n.d.	n.d.	1	2	n.d.
	Pyrazines (11)											
54	2-Methylpyrazine	27,000	n.d.	<1	<1	n.d.	<1	n.d.	4	4	n.d.	32
55	2,5-Dimethylpyrazine	2600	n.d.	4.19	2.73	4.7	4.16	n.d.	64	64	8	128
56	2,6-Dimethylpyrazine	8000	n.d.	<1	<1	n.d.	1.07	n.d.	4	4	n.d.	8
57	Ethylpyrazine	17,000	n.d.	<1	<1	<1	<1	n.d.	2	1	2	2
58	2,3-Dimethylpyrazine	900	n.d.	2.01	1.94	3.28	<1	n.d.	2	4	64	2
59	2-ethyl-6-3-methylpyrazine	400	n.d.	6.4	5.84	6.21	2.25	n.d.	32	16	16	8

Table 5. *Cont.*

Nos.	Volatile Compounds	OT [1] (μg/kg)	OAV Value [2]					FD Factor [3]				
			HS	120 °C HSO	140 °C HSO	160 °C HSO	180 °C HSO	HS	120 °C HSO	140 °C HSO	160 °C HSO	180 °C HSO
60	2-Ethyl-5-2-methylpyrazine	320	n.d.	8.47	4.48	8.48	4.63	n.d.	64	4	64	128
61	Trimethyl-pyrazine	27,000	n.d.	<1	<1	<1	n.d.	n.d.	2	2	2	n.d.
62	4-Ethyl-2,5-5-diMethylpyrazine	24,000	n.d.	n.d.	n.d.	<1	<1	n.d.	n.d.	n.d.	2	4
63	Tetramethylpyrazine	38,000	<1	n.d.	n.d.	n.d.	n.d.	2	n.d.	n.d.	n.d.	n.d.
64	Acetylpyrazine	10	n.d.	n.d.	n.d.	162.56	n.d.	n.d.	n.d.	n.d.	2048	n.d.
	Pyridine (3)											
65	2-Ethylpyridine	57	n.d.	n.d.	n.d.	n.d.	108.73	n.d.	n.d.	n.d.	n.d.	32
66	3-Ethylpyridine	20	n.d.	n.d.	n.d.	n.d.	5.2	n.d.	n.d.	n.d.	n.d.	64
67	2-Pentylpyridine	5	n.d.	n.d.	n.d.	n.d.	588.69	n.d.	n.d.	n.d.	n.d.	1024
	Sulfur-containing compounds (1)											
68	Dimethyl sulfoxide	100	<1	n.d.	n.d.	n.d.	n.d.	16	n.d.	n.d.	n.d.	n.d.
	nitrogen–containing compounds (1)											
69	Acetamide	140,000	<1	n.d.	n.d.	n.d.	n.d.	8	n.d.	n.d.	n.d.	n.d.

[1] The thresholds of compounds in water/oil referred to ref odor thresholds are determined by according to the book Compilations of Flavour Threshold Values in Water and Other Media (Second Enlarged and Revised Edition). Thresholds not found are self-tested using the 3-AFC method in Section 2.6.5. [2] Odor activity values were calculated by dividing the concentration by the odor threshold. [3] FD factor means flavor dilution factor determined on a DB-Wax column.

3.2.4. Recombination and Deletion Experiments

In order to demonstrate the effect of key odor compounds on the overall odor of the samples, recombination models with OAV $\geq$ 1 were developed based on the natural concentrations of the compounds and presented to the sensory judges for aroma characterization. As shown in Figure 7, the similarity of the aroma profiles of the recombinant model and the corresponding HSO samples in Figure 2 were all above 90%, indicating that the recombinant model was able to effectively simulate the aroma of the hemp seed oil. The overall reduction of seeds, herbs, rancid, and toasted flavors is relatively high, perhaps due to the fact that seeds and herbs flavors are the main flavors of hemp seeds, and they have lower relative thresholds to be perceived more distinctly and with the distinctive greasy aroma of hemp seeds. However, the aroma compounds with a rancid odor are relatively single, mainly concentrated in acidic compounds, while the compounds that provide a toasted aroma are mainly concentrated in heterocyclic compounds, which have high compound content and higher intensity of active odor.

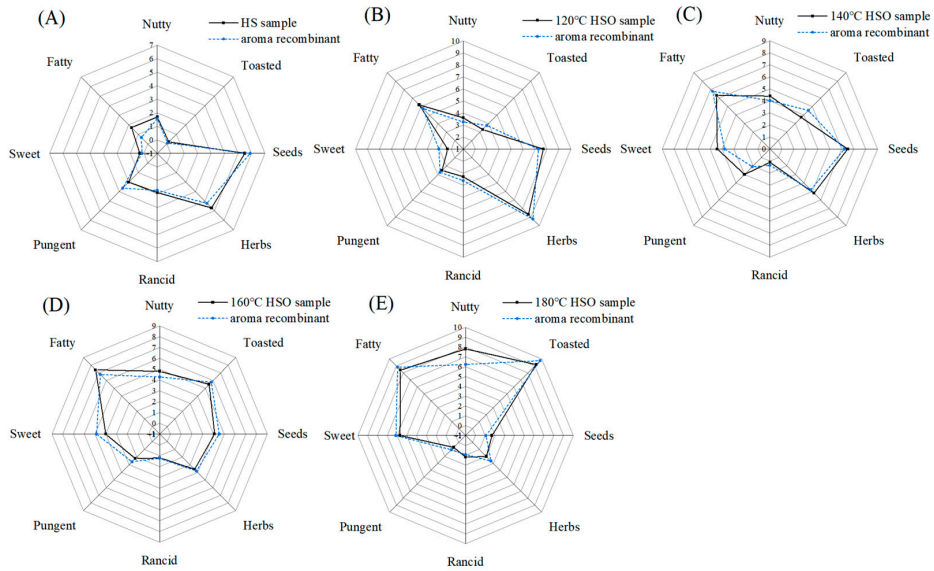

Figure 7. Odor profile analyses. (**A**) Aroma profile of the HS sample and aroma recombinant; (**B**) aroma profile of the 120 °C HSO sample and aroma recombinant; (**C**) aroma profile of the 140 °C HSO sample and aroma recombinant; (**D**) aroma profile of the 160 °C HSO sample and aroma recombinant; (**E**) aroma profile of the 180 °C HSO sample and aroma recombinant.

To further investigate the contribution of compounds to flavor, 23 sets of deletion samples were formulated and evaluated by triangulation experiments, and the results are shown in Table 6. α-pinene, (1*S*)-(1)-β-pinene, myrcene, and (*E*)-β-ocimene showed significance for all samples, with (*E*)-β-ocimene showing the highest significance at $p \leq 0.01$. The results indicate that terpenes can be used as key flavor compounds in the hemp seed series of samples and that they provide hemp seed with a refreshing herbal and sweet aroma. In the hemp seed oil obtained from roasting over pressing, 2-pentylfuran, hexanal, 2,5-dimethylpyrazine, 2-ethyl-6-methylpyrazine, and 2-ethyl-5-methylpyrazine had a significant effect. The above compounds were found to be the key aroma-active compounds in the roasted hemp seed oil, and they provided the roasted hemp seed oil with toasted and fatty flavor.

Table 6. Results of the omission experiments for recombinant models.

Nos.	Volatile Compounds	Significance [1]				
		HS	120 °C HSO	140 °C HSO	160 °C HSO	180 °C HSO
1	α-Pinene	**	*	*	**	*
2	2-Pentylfuran	n.d.	**	**	**	**
3	2,3-Butanediol	**	n.d.	n.d.	n.d.	n.d.
4	Hexanal	n.d.	*	**	***	**
5	Myrcene	**	***	**	**	*
6	(+)-Dipentene	n.d.	**	**	**	*
7	(1S)-(1)-β-Pinene	**	*	*	*	*
8	γ-Terpinene	n.d.	**	n.d.	n.d.	n.d.
9	2-Ethylpyridine	n.d.	n.d.	n.d.	n.d.	***
10	Terpinolene	n.d.	*	*	*	n.d.
11	(*E*)-2-Heptenal	n.d.	n.d.	n.d.	n.d.	*
12	β-Caryophyllene	n.d.	*	n.d.	n.s	n.s [2]
13	(*E*,*Z*)-2,4-Decadienal	n.d.	n.d.	n.d.	**	**
14	Acetic acid	**	n.d.	n.d.	**	**
15	Hexanoic acid	n.s	n.s	n.s	n.s	*
16	(*E*)-β-Ocimene	***	***	***	***	***
17	Linalool	n.d.	*	n.d.	n.d.	n.d.
18	2,5-Dimethylpyrazine	n.d.	**	***	**	**
19	2-ethyl-6-methylpyrazine	n.d.	***	***	**	**
20	Acetylpyrazine	n.d.	n.d.	n.d.	***	n.d.
21	2,3-Dimethylpyrazine	n.d.	*	*	*	n.d.
22	2-Ethyl-5-methylpyrazine	n.d.	*	*	*	*
23	2-Pentylpyridine	n.d.	n.d.	n.d.	n.d.	***

[1] Significance: "*", $p \leq 0.05$; "**", $p \leq 0.01$; "***", $p \leq 0.001$. [2] n.s: no significant difference.

3.2.5. Results of Multivariate Analysis of Hemp Seed Oil

After the initial analysis of aroma substances, multivariate analysis using OPLS-DA was performed to further identify the differences in aroma compounds of hemp seed oil processed at different temperatures. OPLS-DA analysis of the aroma components of hemp seed oil at four different thermal processing temperatures (A) resulted in R^2 (cum) and Q^2 (cum) values of 0.970 and 0.924, respectively, for the model, demonstrating availability of data and predictability of methods. From Figure 8, it can be seen that the hemp seed oils from the four different processing methods can be effectively differentiated. 120 °C HSO and 180 °C HSO can be clearly differentiated. This is due to the fact that 120 °C HSO was found to have a high concentration of (*Z*)-β-ocimene, as well as β-phellandrene, 2-heptanone, linalool, and γ-terpinene, all of which had a strong fruity, sweet, grassy flavor. 180 °C HSO was found to have more heterocyclic compounds; 2-ethylpyridine, 3-ethylpyridine, and 2-pentylpyridine were detected, bringing out a fatty and meaty aroma. 160 °CHSO was also able to efficiently separate out the (*Z*)-β-ocimene, as well as β-phellandrene, 2-heptanone, linalool, and γ-terpinene, which were all detected at high concentrations. 160 °C HSO was also able to be efficiently separated, which contained higher concentrations of (*E*)-β-ocimene, 1-penten-3-ol, 2-ethylpyridine, 3-ethyl-2,5-dimethylpyrazine, and acetic acid, with a floral and fatty aroma.

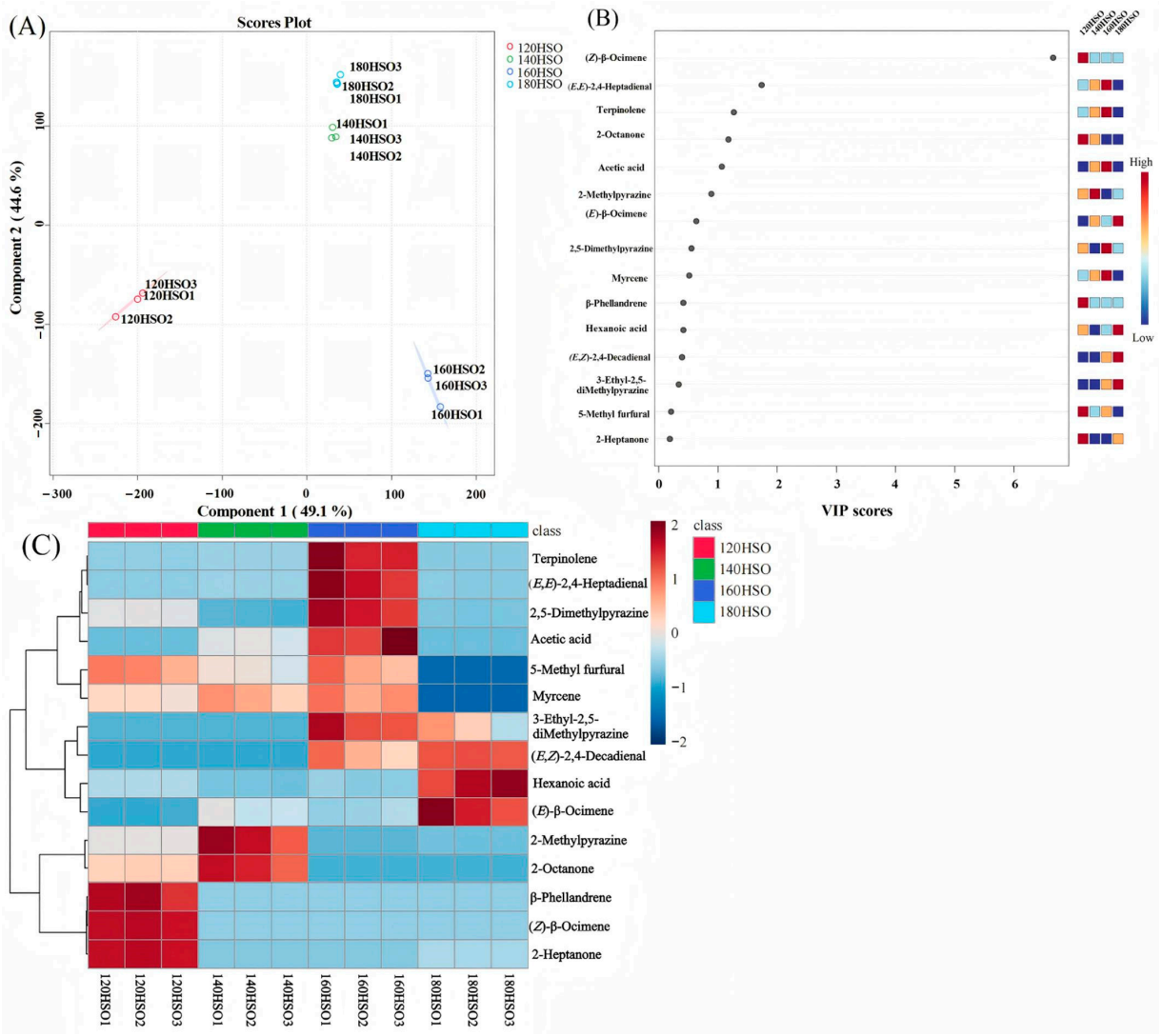

Figure 8. Multivariate analysis of hemp seed oil under processing at five different temperatures: (**A**) the OPLS-DA score plot; (**B**) differential volatile compounds ranked by VIP scores; (**C**) a heat map of VIP $\geq$ 1 differential volatile compound.

The variable OPLS-DA significant projection (VIP) method can be applied to determine the effect of different substances on differentiation per group of samples. Each compound with a VIP $\geq$ 1 is considered to have a significant effect on the differences between groups. As shown in (B), 15 odor compounds were screened for VIP $\geq$ 1. Among them, (*Z*)-β-ocimene, (*E*,*E*)-2,4-heptadienal, terpinolene, 2-octanone, and acetic acid, which are important volatiles of hemp seed oil, had VIP values greater than 2. (*Z*)-β-ocimene had the highest content at 120 °C HSO, and (*E*,*E*)-2,4-heptadienal appeared in all four samples with high compound content. Their odors all fall into the category of floral, grassy, and spicy aromas, representing the size of the compounds they contain that can effectively influence the flavor of the hemp seed oil. Combined with the VIP values of the compounds, flavor differences in hemp seed oil could be effectively discerned across temperatures.

Heatmapping can also effectively explore the differences between samples, as shown in (C). The heatmap was drawn for the compounds with VIP $\geq$ 1, arranged according to the size of VIP value from top to bottom, and the darker color represents the higher content of the compounds. It can be seen that β-phellandrene, 2-heptanone, and (*Z*)-β-ocimene can serve as effective compounds to distinguish 120 °C HSO. 2-methylpyrazine and 2-octanone were able to serve as effective differentiating compounds for 140 °C HSO, terpinolene and

acetic acid in 160 °C HSO. (*E*)-β-ocimene and hexanoic acid had the highest representation in 180 °C HSO. Combining the results of the three multivariate analyses, the hemp seed oils extracted after the four different roasting temperatures were better identified.

4. Conclusions

In this study, we comprehensively determined the main odorants of HS and HSO by SAFE extraction, combined with GC-O-MS, determination of the FD factor by AEDA, calculation of OAVs, aroma recombination experiments, and deletion experiments. A total of 69 volatiles were identified in the HS and the HSOs, and six odor-active compounds with OAV ≥ 1 were identified as the key odorants of Inner Mongolian hemp seeds. They were 2,3-butanediol, α-pinene, (1*S*)-(1)-β-pinene, myrcene, (*E*)-β-ocimene, acetic acid. Hexanal, (*E*,*Z*)-2,4-decadienal, and (*E*)-2-heptenal provided fatty flavors to the hemp seed oils with roasting. 2-pentylfuran, 2,5-dimethylpyrazine, 2-ethyl-6-methylpyrazine, and 2-ethyl-5-methylpyrazine provided roasted, sweet, and nutty flavors. They are considered to be key flavor compounds in roasted hemp seed oils. Sensory evaluation and the results of aroma restructuring experiments showed that 120 °C HSO was dominated by terpenoids, with heavier grassy and fruity flavors; the aromatic compounds in 160 °C HSO were dominated by alcohols, aldehydes, and ketones, mainly flavored with fatty and floral aromas; and 180 °C HSO had more pyrazines and pyridines, which mainly provided roasted, fatty, and meaty flavors. Finally, four hemp seed oils extracted at different roasting temperatures were effectively identified through multivariate analysis, corroborating the above analytical results. In order to reduce the negative flavor of the hemp seed oil obtained after roasting and to be able to retain more of the characteristic flavor of Inner Mongolian hemp seeds, it was concluded that hemp seeds roasted at 160 °C for 20 min possessed a better flavor of hemp seed oil.

Author Contributions: Conceptualization, Y.B. and X.Z.; methodology, Y.B. and N.Y.; investigation, validation, N.Y.; formal analysis Y.B. and H.T.; writing—editing, Y.B., N.Y. and X.Z.; funding acquisition, Y.B. and H.T. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by the National Natural Science Foundation of China (No. 32301973), the R&D Program of Beijing Municipal Education Commission (No. KM202310011008), and the National Natural Science Foundation of China (No. 32130083).

Data Availability Statement: The data presented in this study are available on request from the corresponding author.

Conflicts of Interest: The authors declare no conflicts of interest.

References

1. Frassinetti, S.; Moccia, E.; Caltavuturo, L.; Gabriele, M.; Longo, V.; Bellani, L.; Giorgi, G.; Giorgetti, L. Nutraceutical potential of hemp (*Cannabis sativa* L.) seeds and sprouts. *Food Chem.* **2018**, *262*, 56–66. [CrossRef] [PubMed]
2. Kolodziejczyk, P.; Ozimek, L.; Kozłowska, J. 11-The Application of Flax and Hemp Seeds in Food, Animal Feed and Cosmetics Production. In *Handbook of Natural Fibres*; Kozłowski, R.M., Ed.; Woodhead Publishing: Cambridge, UK, 2012; Volume 2, pp. 329–366.
3. Matthäus, B.; Brühl, L. Virgin hemp seed oil: An interesting niche product. *Eur. J. Lipid Sci. Technol.* **2008**, *110*, 655–661. [CrossRef]
4. Martini, N. Hempseed oil. *J. Prim. Health Care* **2021**, *13*, 375–376. [CrossRef]
5. Xu, J.; Bai, M.; Song, H.; Yang, L.; Zhu, D.; Liu, H. Hemp (*Cannabis sativa* subsp. sativa) Chemical Composition and the Application of Hempseeds in Food Formulations. *Plant Foods Hum. Nutr.* **2022**, *77*, 504–513. [CrossRef]
6. Callaway, J.C. Hempseed as a nutritional resource: An overview. *Euphytica* **2004**, *140*, 65–72. [CrossRef]
7. Tura, M.; Mandrioli, M.; Valli, E.; Toschi, T.G. Quality indexes and composition of 13 commercial hemp seed oils. *J. Food Compost. Anal.* **2023**, *117*, 105112. [CrossRef]
8. Shariatmadari, F. Emergence of hemp as feed for poultry. *Worlds Poult. Sci. J.* **2023**, *79*, 769–782. [CrossRef]
9. Chen, T.; He, J.; Zhang, J.; Zhang, H.; Qian, P.; Hao, J.; Li, L. Analytical characterization of Hempseed (seed of *Cannabis sativa* L.) oil from eight regions in China. *J. Diet. Suppl.* **2010**, *7*, 117–129. [CrossRef]

10. Song, W.; Yin, H.; Zhong, Y.; Wang, D.; Xu, W.; Deng, Y. Regional differentiation based on volatile compounds via HS-SPME/GC-MS and chemical compositions comparison of hemp (*Cannabis sativa* L.) seeds. *Food Res. Int.* **2022**, *162*, 112151. [CrossRef]
11. Borhade, S.S. Chemical Composition and Characterization of Hemp (*Cannabis sativa*) Seed oil and essential fatty acids by HPLC Method. *Arch. Appl. Sci. Res.* **2013**, *5*, 5–8.
12. Matilde, T.; Mara, M.; Enrico, V.; Caterina, D.; Tullia Gallina, T. Sensory Wheel and Lexicon for the Description of Cold-Pressed Hemp Seed Oil. *Foods* **2023**, *12*, 661. [CrossRef] [PubMed]
13. Xu, Y.; Li, J.; Zhao, J.; Wang, W.; Griffin, J.; Li, Y.; Bean, S.; Tilley, M.; Wang, D. Hempseed as a nutritious and healthy human food or animal feed source: A review. *Int. J. Food Sci. Technol.* **2021**, *56*, 530–543. [CrossRef]
14. Yu, Z.; Xiaolong, L.; Xinzhu, L.; Hao, S.; Fengjun, W. Effect of oilseed roasting on the quality, flavor and safety of oil: A comprehensive review. *Food Res. Int.* **2021**, *150*, 110791.
15. Jia, X.; Zhou, Q.; Wang, J.; Liu, C.; Huang, F.; Huang, Y. Identification of key aroma-active compounds in sesame oil from microwaved seeds using E-nose and HS-SPME-GCxGC-TOF/MS. *J. Food Biochem.* **2019**, *43*, e12786. [CrossRef] [PubMed]
16. Wencan, Z.; Rui, W.; Yonghong, Y.; Tiankui, Y.; Shaoquan, L. Changes in volatiles of palm kernel oil before and after kernel roasting. *LWT* **2016**, *73*, 432–441.
17. Jeong, H.; Yoon, S.; Jo, S.M.; Hong, S.J.; Ban, Y.; Park, H.; Youn, M.Y.; Shin, E.-C. Chemosensory of hemp seed oil extracted with hemp seed (*Cannabis sativa* L.) roasted under various conditions using electronic sensors and GC-MS/ Olfactometry. *Food Chem. X* **2024**, *21*, 101226. [CrossRef]
18. Zhou, Q.; Liu, S.; Liu, Y.; Song, H. Comparative Analysis of Volatiles of 15 Brands of Extra-Virgin Olive Oils Using Solid-Phase Micro-Extraction and Solvent-Assisted Flavor Evaporation. *Molecules* **2019**, *24*, 1512. [CrossRef]
19. Guohao, S.; Yulan, L.; Yuxiang, M.; Xiaohua, Y.; Qiang, Y. Effects of high-oleic acid and regular peanuts on the flavor and quality of fragrant peanut oil. *Food Sci. China* **2022**, *43*, 232–241.
20. Wen-Ting, Y.; Rui, S.; Ke, L.; Xue-De, W.; An-Na, W.; Yu-Hang, Z.; Zhuo-Qing, Z. Effect of microwave pretreatment of sunflower kernels on the aroma-active composition, sensory quality, lipid oxidation, tocopherols, heterocyclic amines and polycyclic aromatic hydrocarbons of sunflower oil. *LWT* **2022**, *170*, 114077.
21. Farid, M.; Aymane, A.; Abdessamad Ben, M.; Chaymae, B.; Yassine, T.; Kamal, B.; Mohamed, A.; Christophe, H.; Marie-Laure, F.; Hana Serghini, C.; et al. Laboratory-Scale Optimization of Hemp Seed Roasting Temperature and Time for Producing a High-Quality Pressed Oil. *J. Food Process. Preserv.* **2023**, *2023*, 8261279.
22. Wu, Y.; Xin, R.; Liang, M.; Zhang, Z.; Guan, W.; Wang, R.; Liu, Y. Investigation on the changes of odor-active compounds of Amomum tsao-ko during natural drying. *J. Food Compost. Anal.* **2023**, *122*, 105432. [CrossRef]
23. Jie, S.; Baoguo, S.; Fazheng, R.; Haitao, C.; Ning, Z.; Yuyu, Z. Characterization of key odorants in Hanyuan and Hancheng fried pepper (*Zanthoxylum bungeanum*) oil. *J. Agric. Food Chem.* **2020**, *68*, 6403–6411.
24. Kesen, S.; Kelebek, H.; Selli, S. Characterization of the Key Aroma Compounds in Turkish Olive Oils from Different Geographic Origins by Application of Aroma Extract Dilution Analysis (AEDA). *J. Agric. Food Chem.* **2014**, *62*, 391–401. [CrossRef] [PubMed]
25. Wen-ting, Y.; Xue-ting, M.; Shi-jia, L.; Xue-de, W.; Hua-min, L.; Rui, S. Comparison of key aroma-active compounds between roasted and cold-pressed sesame oils. *Food Res. Int.* **2021**, *150*, 110794.
26. Czerny, M.; Christlbauer, M.; Christlbauer, M.; Fischer, A.; Granvogl, M.; Hammer, M.; Hartl, C.; Hernandez, N.M.; Schieberle, P. Re-investigation on odour thresholds of key food aroma compounds and development of an aroma language based on odour qualities of defined aqueous odorant solutions. *Eur. Food Res. Technol.* **2008**, *228*, 265–273. [CrossRef]
27. Nicolotti, L.; Mall, V.; Schieberle, P. Characterization of Key Aroma Compounds in a Commercial Rum and an Australian Red Wine by Means of a New Sensomics-Based Expert System (SEBES)-An Approach to Use Artificial Intelligence in Determining Food Odor Codes. *J. Agric. Food Chem.* **2019**, *67*, 4011–4022. [CrossRef]
28. Seyfried, C.; Granvogl, M. Characterization of the Key Aroma Compounds in Two Commercial Dark Chocolates with High Cocoa Contents by Means of the Sensomics Approach. *J. Agric. Food Chem.* **2019**, *67*, 5827–5837. [CrossRef]
29. Zhou, Z.; Jian, D.; Gong, M.; Zhu, S.; Li, G.; Zhang, S.; Zhong, F.; Mao, J. Characterization of the key aroma compounds in aged Zhenjiang aromatic vinegar by gas chromatography-olfactometry-mass spectrometry, quantitative measurements, aroma recombination and omission experiments. *Food Res. Int.* **2020**, *136*, 109434. [CrossRef]
30. Gaca, A.; Kludska, E.; Hradecky, J.; Hajslova, J.; Jelen, H.H. Changes in Volatile Compound Profiles in Cold-Pressed Oils Obtained from Various Seeds during Accelerated Storage. *Molecules* **2021**, *26*, 285. [CrossRef]
31. Marcus, J.B. *Aging, Nutrition and Taste: Nutrition, Food Science and Culinary Perspectives for Aging Tastefully*; Academic Press: Cambridge, MA, USA, 2019.
32. Nissen, L.; Zatta, A.; Stefanini, I.; Grandi, S.; Sgorbati, B.; Biavati, B.; Monti, A. Characterization and antimicrobial activity of essential oils of industrial hemp varieties (*Cannabis sativa* L.). *Fitoterapia* **2010**, *81*, 413–419. [CrossRef]
33. Malingre, T.; Hendriks, H.; Batterman, S.; Bos, R.; Visser, J. The essential oil of Cannabis sativa. *Planta Med.* **1975**, *28*, 56–61. [CrossRef]

34. Peiyi, S.; Zili, G.; Minwei, X.; Jae-Bom, O.; Jiajia, R.; Bingcan, C. The impact of hempseed dehulling on chemical composition, structure properties and aromatic profile of hemp protein isolate. *Food Hydrocoll.* **2020**, *106*, 105889.
35. Bertoli, A.; Tozzi, S.; Pistelli, L.; Angelini, L.G. Fibre hemp inflorescences: From crop-residues to essential oil production. *Ind. Crops. Prod.* **2010**, *32*, 329–337. [CrossRef]
36. Pellati, F.; Brighenti, V.; Sperlea, J.; Marchetti, L.; Bertelli, D.; Benvenuti, S. New Methods for the Comprehensive Analysis of Bioactive Compounds in *Cannabis sativa* L. (hemp). *Molecules* **2018**, *23*, 2639. [CrossRef] [PubMed]
37. Won Ho, S.; Hyung Hee, B. Identification of characteristic aroma-active compounds from water dropwort (*Oenanthe javanica* DC.). *J. Agric. Food Chem.* **2005**, *53*, 6766–6770.
38. Cerino, P.; Buonerba, C.; Cannazza, G.; D'Auria, J.; Ottoni, E.; Fulgione, A.; Di Stasio, A.; Pierri, B.; Gallo, A. A Review of Hemp as Food and Nutritional Supplement. *Cannabis Cannabinoid Res.* **2021**, *6*, 19–27. [CrossRef]
39. Ma, S.; Ding, C.; Zhou, C.; Shi, H.; Bi, Y.; Zhang, H.; Xu, X. Peanut oils from roasting operations: An overview of production technologies, flavor compounds, formation mechanisms, and affecting factors. *Heliyon* **2024**, *10*, e34678. [CrossRef]
40. Erten, E.S.; Cadwallader, K.R. Identification of predominant aroma components of raw, dry roasted and oil roasted almonds. *Food Chem.* **2017**, *217*, 244–253. [CrossRef]
41. Gracka, A.; Jelen, H.H.; Majcher, M.; Siger, A.; Kaczmarek, A. Flavoromics approach in monitoring changes in volatile compounds of virgin rapeseed oil caused by seed roasting. *J. Chromatogr. A* **2016**, *1428*, 292–304. [CrossRef]
42. Daletos, G.; Katsimpouras, C.; Stephanopoulos, G. Novel Strategies and Platforms for Industrial Isoprenoid Engineering. *Trends Biotechnol.* **2020**, *38*, 811–822. [CrossRef]
43. Russo, E.B. Taming THC: Potential cannabis synergy and phytocannabinoid-terpenoid entourage effects. *Br. J. Pharmacol.* **2011**, *163*, 1344–1364. [CrossRef] [PubMed]
44. Da Porto, C.; Decorti, D.; Natolino, A. Separation of aroma compounds from industrial hemp inflorescences (*Cannabis sativa* L.) by supercritical CO_2 extraction and on-line fractionation. *Ind. Crops. Prod.* **2014**, *58*, 99–103. [CrossRef]
45. Iseppi, R.; Brighenti, V.; Licata, M.; Lambertini, A.; Sabia, C.; Messi, P.; Pellati, F.; Benvenuti, S. Chemical Characterization and Evaluation of the Antibacterial Activity of Essential Oils from Fibre-Type *Cannabis sativa* L. (Hemp). *Molecules* **2019**, *24*, 2302. [CrossRef] [PubMed]
46. Fei, H.; YanPing, L.Q.; Qian, M.C. Flavor and chiral stability of lemon-flavored hard tea during storage. *Food Chem.* **2018**, *239*, 622–630.
47. Diaz-Montana, E.J.; Aparicio-Ruiz, R.; Morales, M.T. Effect of Flavorization on Virgin Olive Oil Oxidation and Volatile Profile. *Antioxidants* **2023**, *12*, 242. [CrossRef]
48. Sunic, L.; Ilic, Z.S.; Stanojevic, L.; Milenkovic, L.; Stanojevic, J.; Kovac, R.; Milenkovic, A.; Cvetkovic, D. Comparison of the Essential Oil Content, Constituents and Antioxidant Activity from Different Plant Parts during Development Stages of Wild Fennel (*Foeniculum vulgare* Mill.). *Acta Hortic.* **2023**, *9*, 364.
49. Weeratunge, H.D.; Sirimal Premakumara, G.A.; de Silva, E.D.; Prasadini Rodrigo, W.W. Comparative study on chemical characteristics of essential oils and genetic characteristics of true Cinnamon (*Cinnamomum zeylanicum* Blume) and three wild Cinnamon species of Sri Lanka. *J. Essent. Oil Res.* **2024**, *36*, 234–246. [CrossRef]
50. Minh Tu, N.T.; Onishi, Y.; Choi, H.S.; Kondo, Y.; Ukeda, H.; Sawamura, M. Characteristic odour components of *Citrus* sp. (Kiyookadaidai) cold-pressed peel oil. *Flavour Fragr. J.* **2003**, *18*, 515–520. [CrossRef]
51. Hanus, L.O.; Hod, Y. Terpenes/Terpenoids in Cannabis: Are They Important? *Cannabis Cannabinoid Res.* **2020**, *3*, 25–60. [CrossRef]
52. Jakab, E.; Blazso, M.; Barta-Rajnai, E.; Babinszki, B.; Sebestyen, Z.; Czegeny, Z.; Nicol, J.; Clayton, P.; McAdam, K.; Liu, C. Thermo-oxidative decomposition of lime, bergamot and cardamom essential oils. *J. Anal. Appl. Pyrolysis* **2018**, *134*, 552–561. [CrossRef]
53. Juita; Dlugogorski, B.Z.; Kennedy, E.M.; Mackie, J.C. Identification and Quantitation of Volatile Organic Compounds from Oxidation of Linseed Oil. *Ind. Eng. Chem. Res.* **2012**, *51*, 5645–5652.
54. Goicoechea, E.; Guillen, M.D. Volatile compounds generated in corn oil stored at room temperature. Presence of toxic compounds. *Eur. J. Lipid Sci. Technol.* **2014**, *116*, 395–406. [CrossRef]
55. El Adab, S.; Hassouna, M. Proteolysis, Lipolysis and Sensory Characteristics of a Tunisian Dry Fermented Poultry Meat Sausage with Oregano and Thyme Essential Oils. *J. Food Saf.* **2016**, *36*, 19–32. [CrossRef]
56. Ran, W.; Chao, Y.; Huanlu, S. Key meat flavour compounds formation mechanism in a glutathione–xylose Maillard reaction. *Food Chem.* **2012**, *131*, 280–285.
57. Poisson, L.; Schaerer, A.; Spreng, S.; Mestdagh, F.; Blank, I.; Davidek, T. Generation of α-Diketones and 4-Hydroxy-2,5-dimethyl-3(2H)-furanone upon Coffee Roasting-Impact of Roast Degree on Reaction Pathways. *J. Agric. Food Chem.* **2019**, *67*, 13829–13839. [CrossRef]
58. Seong Jun, H.; Chang Guk, B.; Jookyeong, L.; Seong Wook, H.; Seong Min, J.; Hyangyeon, J.; Sojeong, Y.; Youngseung, L.; Sung-Soo, P.; Eui-Cheol, S. Chemosensory approach supported-analysis of wintering radishes produced in Jeju island by different processing methods. *Food Sci. Biotechnol.* **2021**, *30*, 1033–1049.

59. Min, D.B.; Callison, A.L.; Lee, H.O. Singlet oxygen oxidation for 2-pentylfuran and 2-pentenyfuran formation in soybean oil. *J. Food Sci.* **2003**, *68*, 1175–1178. [CrossRef]
60. Umano, K.; Hagi, Y.; Nakahara, K.; Shyoji, A.; Shibamoto, T. Volatile Chemicals Formed in the Headspace of a Heated D-Glucose/L-cysteine Maillard Model System. *J. Agric. Food Chem.* **1995**, *43*, 2212–2218. [CrossRef]
61. Jian, Z.; Meng, W.; Jianchun, X.; Mengyao, Z.; Li, H.; Jingjing, L.; Shi, W.; Jie, C. Volatile flavor constituents in the pork broth of black-pig. *Food Chem.* **2017**, *226*, 51–60.
62. Young-Suk, K.; Hartman, T.G.; Chi-Tang, H. Formation of 2-pentylpyridine from the thermal interaction of amino acids and 2,4-decadienal. *J. Agric. Food Chem.* **1996**, *44*, 3906–3908.
63. Cao, W.; Shu, N.; Wen, J.; Yang, Y.; Jin, Y.; Lu, W. Characterization of the Key Aroma Volatile Compounds in Nine Different Grape Varieties Wine by Headspace Gas Chromatography-Ion Mobility Spectrometry (HS-GC-IMS), Odor Activity Values (OAV) and Sensory Analysis. *Foods* **2022**, *11*, 2767. [CrossRef] [PubMed]
64. Zhang, N.; Sun, B.; Mao, X.; Chen, H.; Zhang, Y. Flavor formation in frying process of green onion (*Allium fistulosum* L.) deep-fried oil. *Food Res. Int.* **2019**, *121*, 296–306. [CrossRef] [PubMed]
65. Jing, B.; Guo, R.; Wang, M.; Zhang, L.; Yu, X. Influence of seed roasting on the quality of glucosinolate content and flavor in virgin rapeseed oil. *LWT* **2020**, *126*, 109301. [CrossRef]
66. Adams, A.; Kitryte, V.; Venskutonis, R.; De Kimpe, N. Model Studies on the Pattern of Volatiles Generated in Mixtures of Amino Acids, Lipid-Oxidation-Derived Aldehydes, and Glucose. *J. Agric. Food Chem.* **2011**, *59*, 1449–1456. [CrossRef]
67. Sánchez-Palomo, E.; García-Carpintero, E.G.; Gómez Gallego, M.; González Viñas, M. The Aroma of Rojal Red Wines from La Mancha Region-Determination of Key Odorants. In *Gas Chromatography in Plant Science, Wine Technology, Toxicology and Some Specific Applications*; InTechOpen: London, UK, 2012; pp. 147–170.
68. Wang, M.-Q.; Ma, W.-J.; Shi, J.; Zhu, Y.; Lin, Z.; Lv, H.-P. Characterization of the key aroma compounds in Longjing tea using stir bar sorptive extraction (SBSE) combined with gas chromatography-mass spectrometry (GC-MS), gas chromatography-olfactometry (GC-O), odor activity value (OAV), and aroma recombination. *Food Res. Int.* **2020**, *130*, 108908. [CrossRef]

Disclaimer/Publisher's Note: The statements, opinions and data contained in all publications are solely those of the individual author(s) and contributor(s) and not of MDPI and/or the editor(s). MDPI and/or the editor(s) disclaim responsibility for any injury to people or property resulting from any ideas, methods, instructions or products referred to in the content.