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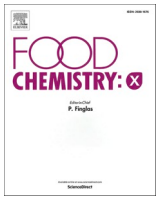

Unveiling the flavor and quality variations in dried *Zanthoxylum bungeanum maxim* from China's diverse regions

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ABSTRACT

Dried *Zanthoxylum bungeanum Maxim* (DZM) is one of the popular categories in spices and condiments market. The flavor and quality of DZM products determine its value and application scope. This study evaluated the flavor and quality of DZMs from various origins, considering physical, chemical, and safety attributes. Notable variations were observed, with DZMs from Maoxian and Jinyang excelling in aroma, pungency, and appearance. Chromatic analysis distinguished green and red DZMs from Meishan and Hancheng. HPLC results revealed high pungency compound levels in Maoxian and Wudu samples, while GC–MS identified 173 volatile compounds, dominated by linalool and D-limonene. Microbial contamination was minimal in DZMs from Hanyuan and Jiangjin, and the lowest heavy metal levels were in samples from Hanyuan, Hancheng, and Jingyang, indicating superior environmental conditions. The research offered insights into origin and processing influences on DZM, aiding in selection and food safety assurance.

1. Introduction

Zanthoxylum bungeanum Maxim, commonly known as huajiao, belongs to the Rutaceae family and the Zanthoxylum genus (Ji, Li, & Ho, 2019). The fresh fruits of this plant are abundant in amide compounds and essential oils, imparting them with a distinctive numbing sensation and fragrance (Liang et al., 2024). These characteristics have rendered them a significant spice and seasoning in a variety of culinary traditions (Ji et al., 2019). DZM, first mentioned in the ancient Chinese text "Er Ya," has historically been referred to as "li" or "da jiao" (Chang et al., 2024). The southwestern region of China, including provinces such as Sichuan, Guizhou, Gansu, Shaanxi, and Chongqing, is the primary global source for DZM, thanks to its unique geographical environment which is conducive to the cultivation of diverse DZM varieties (Shao et al., 2023). Globally, over 250 varieties of DZM. have been identified, with China alone boasting 45 species and 13 varieties (Liang et al., 2024). Approximately a dozen of these varieties have been used as culinary spices, including *Zanthoxylum bungeanum* (red prickly ash), *Zanthoxylum armatum* (bamboo leaf prickly ash), *Zanthoxylum schinifolium* (green prickly ash), *Zanthoxylum simulans* (wild prickly ash), and *Zanthoxylum piasezkii* (Sichuan-Shaanxi prickly ash). Moreover, DZM is not only prevalent in China but also extensively distributed in other Asian countries such as Japan, India, and South Korea (Zhang et al., 2017). The unique numbing sensation and aroma of DZM have long been sought and valued by culinary aficionados worldwide.

The tingling sensation and aromatic profile of DZM are pivotal in its assessment and are crucial drivers of its consumption in the high-end market. The distinctive tingling effect of DZM. is primarily derived from alkylamide compounds found in its fruits, such as hydroxy-α-sanshool, hydroxy-β-sanshool, and hydroxy-γ-sanshool. These compounds stimulate the tactile nerves on the tongue, creating a unique numbing sensation (Bader, Stark, Dawid, Lösch, & Hofmann, 2014). The diversity and concentration of these characteristic tingling components typically determine the overall tingling intensity of DZM. Additionally, the plant is abundant in volatile essential oils, More than 100 volatile organic compounds passed through GC–MS, GC-IMS, About 70 of these incense-like compounds were identified as with notable aroma constituents, including 1, 8-cineole, (E)-2-heptenal, β-myrcene, β-ocimene, limonene, and linalool etc., all of which enhance its complex sensory profile (Yang, 2008). Furthermore, factors such as the variety, origin, climate, and

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processing techniques significantly influence the final product's flavor components and sensory attributes of DZM (Feng et al., 2024; Shao et al., 2023; Zheng et al., 2022).

The harvest season for fresh DZM typically spans from July to October, with green DZM being harvested mainly from July to September, and red DZM from August to October. Fresh DZM often contains moisture exceeding 50 %, which makes it prone to mechanical damage, compression, light exposure, and oxidation during the picking process. Currently, the primary method of harvesting is manual picking. Freshly picked DZM. has a limited shelf life, with discoloration occurring within 6–12 h at room temperature and a storage duration of 3–5 days when kept with stems and leaves at 4 °C. In the long-term production and processing of DZM, efforts are made to preserve its flavor characteristics as much as possible. The market's main processing methods include solvent extraction, drying, and vacuum color protection and preservation for storage. Solvent extraction techniques encompass supercritical carbon dioxide extraction, fried process extraction, fresh-pressed process extraction, and a novel low-temperature continuous phase change extraction, with the application of the products varying according to consumer demands (Liu et al., 2023). Drying methods such as freeze-drying, solar drying, shaded drying, far-infrared drying, and hot air drying are all utilized in practical production and processing (Feng et al., 2024). Additionally, low-temperature vacuum color protection preservation involves enzyme deactivation through steam sterilization, followed by vacuum low-temperature preservation to isolate oxygen, thereby maintaining the color and freshness of fresh DZM.

Due to the challenging storage conditions, dried DZM has consistently been the mainstream product in the market, with its sensory attributes and application range widely accepted and appreciated by the public. However, the characteristic flavors of dried DZM from different regions can vary significantly due to variations in processing methods and varieties. To guide production and consumer application, based on the actual product quality specifications, the flavor and quality of different sources were evaluated, encompassing basic, physical, and chemical indexes (moisture, ash, opening rate), color, volatile oil, and hemp composition, as well as microbial and heavy metal indexes. Furthermore, the variable importance projection (VIP) method of the partial least squares discriminant analysis model was employed to study the key aroma components of DZM from different origins, which provided reference guidance for the identification and production application of dried DZM.

2. Materials and methods

2.1. Zanthoxylum bungeanum maxim. Material

Samples were gathered from seven primary producing regions: Meishan (MS) in Sichuan, Jingyang (JY) in Sichuan, Hanyuan (HY) in Sichuan, and Maoxian (MX) in Sichuan; Jiangjin (JJ) in Chongqing; Hancheng (HC) in Shaanxi; and Wudu (WD) in Gansu, as depicted in Fig. 1C. Green and red DZM belong to different species. All of the samples were uniformly dried at 40 °C conditions by heated-air drying, and stored in dry, cool glass bottles for subsequent analysis. The material information was added in Supplementary documentation 1.

2.2. Reagents

Analytical standards, encompassing C7-C30 normal alkanes (O2SI, American), as well as hydroxy-α-sanshool and hydroxy-β-sanshool, were acquired from Yuanye Co., Ltd. (Shanghai, China). The internal standard, cyclohexanone (purity >99 %), was sourced from Aladdin Biochemical Technology (Shanghai, China). Furthermore, chromatographic grade methanol and acetonitrile were procured from Fisher Scientific (Springfield, NJ, USA). Additional chemicals of analytical grade were obtained from the Guangzhou Chemical Reagent Factory (Guangzhou, China).

2.3. Sensory evaluation

Sensory analysis was employed to evaluate the flavor profiles of DZM samples from various origins, following the reported method with slight modifications (Ni, Yan, Tian, Zhan, & Zhang, 2022). A panel consisting of 10 well-trained members (all of Chinese descent; comprising 5 males and 5 females, aged between 20 and 30 years) underwent sensory training at the Food Flavor Laboratory of South China Agricultural University. For each flavor attribute, the intensity was rated on a 10-point scale. Appropriate agreements were executed to safeguard the rights and privacy of all participants involved in this study. The DZMs utilized in the research were safe and consisted of ordinary food items that met national standard requirements. Based on the regulations of our institution and country, ethical permission was not required for conducting human sensory studies. Our institution did not have a dedicated sensory research ethics committee, and according to our institutional policies and national laws, such research was not subject to an ethical review process. We had ensured that all participants provided written informed consent prior to their involvement in the study, and we had strictly adhered to the regulations concerning the protection of participants' rights and interests throughout the research process.

2.4. Analysis of basic indicator

2.4.1. Color difference determination

The samples were pulverized using a traditional chinese medicine grinder for 20 s, then sifted through an 80-mesh sieve. Subsequently, 5 g of the powdered sample was placed in a glass dish, and the L*, a*, and b* values were measured and recorded using colorimeter (colorimeter, DC-P3A, China). The color difference meter was preheated for 30 min, and then calibrated with a standard blackboard and whiteboard before measuring the samples.

2.4.2. Moisture determination

Referencing GB 5009.3–2016, the moisture content of all samples was measured using the drying method. Take a clean glass dish and put it in 105 °C drying box to dry with constant weight, put 2.000 g Chinese pepper sample into this glass dish, the sample thickness does not exceed 5 mm, dry it in 105 °C drying box to constant weight, calculate the moisture content.

2.4.3. Ash determination

In accordance with GB 5009.4–2016, approximately 1.000 g of each sample was weighed into a crucible, and the crucible's weight was recorded. The crucible containing the sample was then placed in an automatic temperature-controlled furnace and heated to 600 °C for 2 h to ensure complete carbonization of the sample until it was smokeless, resulting in a gray-white ash. Once the furnace temperature dropped below 200 °C, the crucible was transferred to a dryer to cool to room temperature.

2.4.4. Analysis of microbiological

The total colony count was determined in accordance with GB 4789.2–2016, "Determination of National Standard for Food Safety." Coliform bacteria detection was conducted as per GB/T 4789.3–2003, "Determination of coliform bacteria in Food Hygiene." Mold detection was carried out according to GB/T 4789.15–2016, "Count of bacteria and yeast in National Standard for Food Safety." A representative sample was aseptically taken and appropriately diluted in sterile diluents to achieve a countable range of colonies on the agar plate. Using a sterile pipette, 1 mL of the diluted sample was plated onto a sterile plate count agar/lauryl sulfate broth tubes/potato dextrose agar. The plates were inverted and incubated at 36 ± 1 °C for 48 ± 2 h/37 ± 1 °C for 24 ± 2 h/25 ± 1 °C for 5 days. After incubation, the colonies were counted, and the number of colony-forming units (CFUs) per gram or milliliter of the original sample was calculated.

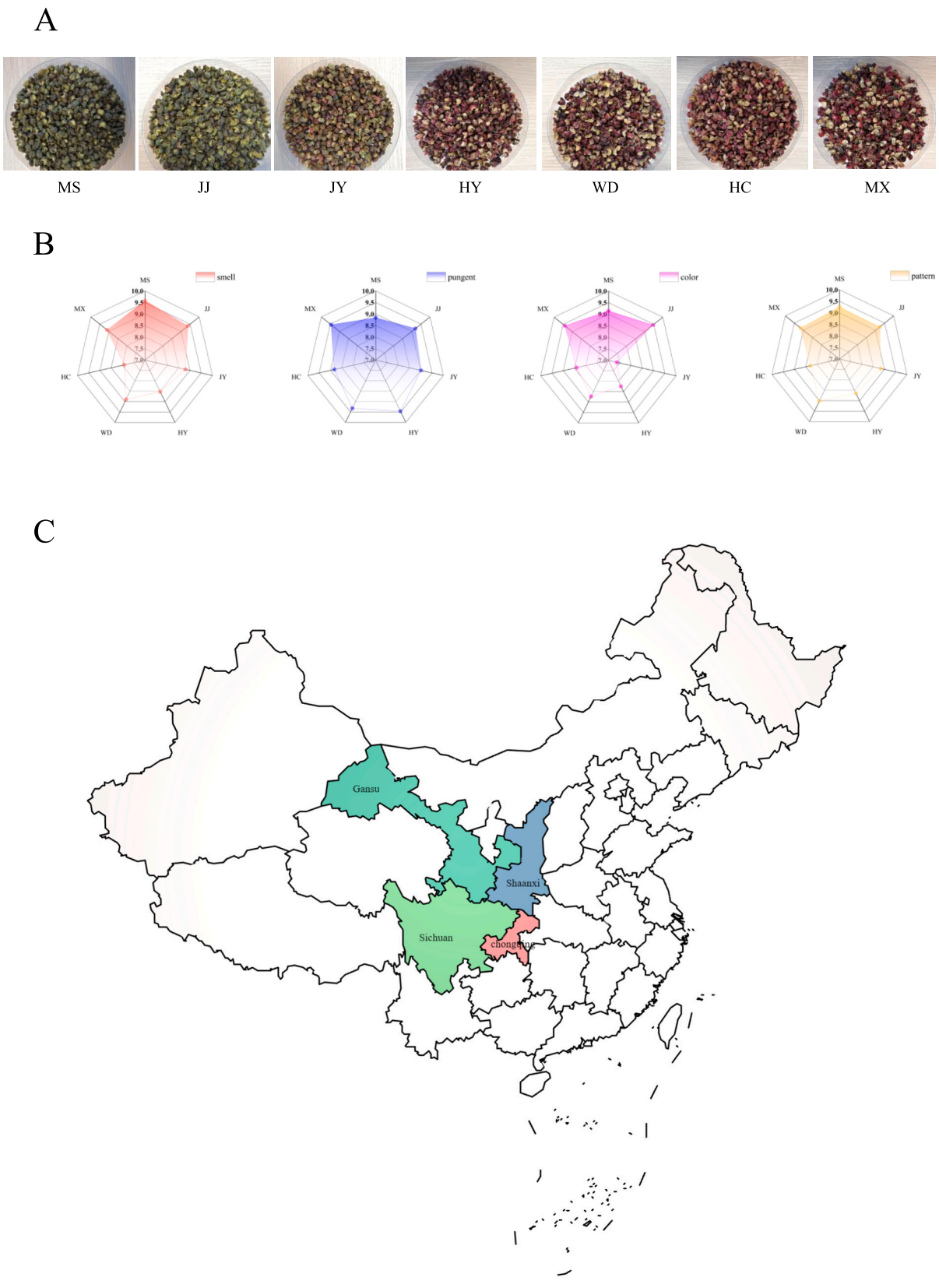

Fig. 1. Pictures of dried Zanthoxylum *bungeanum maxim* of different origins (A); results of sensory analysis of DRIED Zanthoxylum *bungeanum maxim* of different origins (B); sources of origin of dried Zanthoxylum *bungeanum maxim* of different origins (C).

2.4.5. Detection of heavy metals

This study referred to the determination of total arsenic and lead in food as per GB/T 5009.11–2014 and GB/T 5009.12–2023. The determination of Cadmium in food is in accordance with GB 5009.15–2023, the Food Safety National Standard for the Determination of Cadmium in Food. The sample was dried, ground, and then digested using a mixture of acids to convert all heavy metals into soluble forms. Graphite furnace atomic absorption spectrometry (GFAAS) was typically used for the quantification of heavy metals. The digested sample was introduced into the GFAAS instrument, and the heavy metal concentration was measured against standard calibration curves.

2.5. Analysis of volatile compounds from Zanthoxylum bungeanum maxim

2.5.1. Extraction of volatile oils from Zanthoxylum bungeanum maxim

Take 20 g of the sample and place it in a flask, adding an appropriate amount of distilled water. Heat the mixture to distill for 4 h. Once cooled to room temperature, measure the volume of the essential oil and collect the volatile oil, then seal and freeze it for storage. Prior to the experiment, remove the volatile oil from the refrigerator and allow it to reach room temperature. Using a pipette, accurately measure 0.2 mL of the volatile oil and transfer it to a 10 mL volumetric flask. Add anhydrous Na_2SO_4 to the flask and let it stand at −20 °C for 3 h. After this time, remove the samples and allow them to return to room temperature. Filter the samples through a 0.22 μm membrane, then take 20 μL of a cyclohexanone standard solution (1.333 mg/mL) and add it to the filtered sample solution in the flask. Shake the mixture thoroughly and proceed to test the samples.

2.5.2. Analysis of volatile oils from Zanthoxylum bungeanum maxim

The analysis was conducted using an HP-5MS flexible quartz capillary column (30 m × 0.25 μm × 0.25 μm). The temperature program for the column started at 50 °C, held for 2 min, then increased to 110 °C at a rate of 3 °C/min, held for 3 min, followed by an increase to 140 °C at 3 °C/min, held for 10 min, and finally increased to 220 °C at 6 °C/min, held for 2 min. The carrier gas was high-purity helium. The inlet temperature was set at 250 °C, the injection volume was 1 μL, the split ratio was 10:1, and the solvent delay time was 3 min. An EI ion source was used with an ion source temperature of 230 °C and a quadrupole temperature of 150 °C. The scanning range was set from *m/z* 40 to 550, and the NIST14.0 standard spectrum library was used for spectral searches. All samples were analyzed under the same experimental conditions, and the total ion chromatogram of the GC–MS analysis for each sample was recorded.

2.6. Analysis of pungent components from zanthoxylum bungeanum maxim by HPLC

Accurately weigh 5.0 g of crushed DZM, add 25 mL of chromatographic grade methanol, placed in ultrasonic extraction for 30 min filtration, filtrate transferred to a 50 mL brown volumetric flask for volume setting. The sample was diluted 10-fold with methanol before injection, and then filtered through 0.22 μm organic filtration membrane into the injection bottle, and then frozen at −20 °C for use.

High performance liquid chromatography (HPLC) conditions; Agilent 1260 HPLC: column: Eclipse Plus C18 (4.6 mm × 150 mm, 5 μm), detector: VWD, mobile phase: water (A)-acetonitrile (B), detection wavelength: 268 nm, column temperature: 35 °C, flow rate: 1.0 mL/min, injection volume: 10 μL. elution program: 0 min–5 min (35 % acetonitrile); 5 min–10 min (35 %–40 % acetonitrile); 10 min–30 min (40 %–50 % acetonitrile); 30 min–35 min (50 %–35 % acetonitrile).

2.7. Statistical analysis

All data were analyzed using SPSS Statistics software (version 13) and are presented as mean ± standard deviation (SD). Statistical analysis was conducted via one-way analysis of variance (ANOVA), followed by Duncan's test for post-hoc comparisons. A *p*-value of less than 0.05 was deemed statistically significant, while a *p*-value below 0.01 was considered highly significant. Volatile components in the dried DZM were identified by GC–MS and compared with mass spectrum data in the Agilent NIST14.0 L library. Partial least squares discriminant analysis (PLS-DA) and clustering heat map were performed using R software. The map was drawn in https://www.nbcharts.com.

3. Results and discussion

3.1. Sensory evaluation

The color, aroma, taste and shape of DZM were the important factors that determine consumer choice and economic value (Ji et al., 2019). The purpose of the sensory analysis was to directly analyze the flavor differences of DZM from other regions. Four attributes such as odor, pungency, color and pattern were selected for description and plotted on a radar chart as shown in Fig. 1B. The results of the dried DZM odor analysis revealed that DZM from MS and JJ had the highest scores, while DZM from HC had the lowest. In terms of the pungency taste feeling, DZM from HY and MX ranked highest, while DZM from HC and MS scored lower. DZM from JJ and MX scored higher in color, whereas DZM from JY scored the lowest. For shape, DZM from WD, JJ, MS, and MX ranked highest, followed by DZM from JY and HC, with DZM from HC scoring the lowest. Overall, the dried DZM from MX and JJ scored highly and average on all four indicators, indicating their superior quality. DZM from MS and WD ranked in the middle, while the dried peppercorns from HC had the lowest scores. From the color of the brightness, the softness of the smell, the stimulation of the hemp taste and the fullness of the shape all determine the flavor characteristics of DZM, meanwhile four attributes were also a direct expression of influencing consumer choice.

3.2. Color attributes

Peel color is a critical external economic characteristic of DZM productions (Jing et al., 2021). This study compared the color parameters (L value, a value, b value) of DZM from different regions. In Table 1, L represents lightness and darkness, a signifies redness and greenness, and b indicates yellowness and blueness, revealing the color characteristics of the DZM from various origins. From the color results, it can be observed that the colors of different origins are mainly categorized into two groups: green and red. MS, JJ, and JY were the primary regions for green DZM; HY, WD, HC, and MX are the main regions for red DZM.

Firstly, for the dried DZM from MS, JJ, and JY, their color characteristics displayed significant differences. The dried DZM from JY showed a relatively high a value and the lowest L value, indicating a darker color. In contrast, the dried DZM from MS displayed a bright green color with high glossiness, with a relatively high L value, which suggested that the DZM pigment was relatively stable and abundant. Second, the results revealed notable differences in the color characteristics of dried red DZM from HY, WD, HC, and MX. The dried DZM from HC and WD had higher L values, indicating a brighter color. Among them, the dried DZM from HC had the highest a value, suggesting a bright red color with high glossiness. Although the dried DZM from MX and HC had slightly lower L and a values compared to HC, they still exhibited good color characteristics, indicating that the environmental conditions in these regions were also conducive to the growth and pigmentation of DZM.

Based on the above differences in DZM color, many studies had also confirmed that the color of DZM was susceptible to different environmental factors, including varieties, altitude, climate, and soil conditions, which influenced the accumulation and distribution of pigment in DZM (Phuyal, Jha, Raturi, & Rajbhandary, 2020; Zheng, Zhang, Su, & Liu,

Table 1
Determination of basic indices, microorganisms and heavy metals for DZMs from different origins.

Sample	MS	JJ	JY	HY	WD	HC	MX
basic indices							
L value	39.06 ± 0.83^{c}	37.88 ± 1.13^{d}	30.70 ± 0.85^{e}	39.30 ± 0.85^{bc}	43.36 ± 0.23^{a}	44.30 ± 0.94^{a}	40.30 ± 0.64^{b}
a value	4.28 ± 0.08^{e}	3.84 ± 0.17^{f}	8.30 ± 0.10^{d}	12.08 ± 0.16^{c}	12.60 ± 0.21^{b}	13.22 ± 0.21^{a}	12.66 ± 0.06^{b}
b value	34.28 ± 0.51^{a}	34.14 ± 0.71^{a}	25.66 ± 0.45^{e}	28.00 ± 0.70^{d}	30.52 ± 0.34^{b}	29.78 ± 0.75^{c}	28.00 ± 0.36^{d}
water content/%	8.08 ± 0.05^{f}	8.43 ± 0.04^{e}	9.42 ± 0.06^{a}	8.70 ± 0.15^{d}	9.25 ± 0.01^{b}	8.92 ± 0.06^{c}	6.72 ± 0.06^{g}
ash/%	7.30 ± 0.00^{a}	5.51 ± 0.03^{d}	7.05 ± 0.07^{b}	4.15 ± 0.07^{g}	4.90 ± 0.00^{e}	4.71 ± 0.04^{f}	5.65 ± 0.04^{c}
aperture ratio/%	80.16 ± 0.566^{d}	92.65 ± 0.948^{a}	67.48 ± 0.028^{e}	90.51 ± 0.834^{b}	88.60 ± 0.318^{c}	92.71 ± 0.156^{a}	91.72 ± 1.414^{ab}
Microorganisms							
total number of bacterial colonies (log CFU/g)	5.52	3.58	5.64	3.28	5.32	5.45	4.57
Escherichia coli (log CFU/g)	4.89	<1	<1	<1	<1	<1	<1
Mold (log CFU/g)	4.23	4.15	4.98	3.45	4.95	4.82	4.30
Heavy Metals							
As mg/kg	0.02 ± 0.00^{d}	0.01 ± 0.00^{e}	0.03 ± 0.00^{c}	0.01 ± 0.00^{e}	0.11 ± 0.00^{a}	0.03 ± 0.00^{c}	0.05 ± 0.00^{b}
Pb mg/kg	0.22 ± 0.00^{c}	0.24 ± 0.01^{c}	0.47 ± 0.01^{b}	0.14 ± 0.01^{e}	0.18 ± 0.01^{d}	0.15 ± 0.01^{e}	1.06 ± 0.05^{a}
Cd mg/kg	0.15 ± 0.00^{b}	0.16 ± 0.01^{a}	0.09 ± 0.00^{d}	0.01 ± 0.00^{e}	0.00 ± 0.00^{f}	0.01 ± 0.00^{ef}	0.12 ± 0.00^{c}

Note: Number of experimental repetitions ($n = 3$).
The values of relative percentage contents are shown as the mean ± standard error. In the same rows, different letters behind values represent they have significant difference ($p < 0.05$), number of experimental repetitions ($n = 3$).

2020). High altitude regions, with a significant temperature difference between day and night, were most conducive to the accumulation of DZM epidermis pigment (Phuyal et al., 2020). in addition, the drying process can affect the color change of DZM (Feng et al., 2024). Through the review of the relevant literature, Pelargonin-O-hexoside-O-rham Noside-O-hexoside, pelargonidin 3,5-diglucoside, peonidin O-hexoside, cyanidin O-syringic acid and peonidin-3-O-glucoside were found to be the key anthocyanins in DZM, which gave DZM unique color characteristics. In addition, the cumulative of pel-rha, pel-3, 5-diglu, peo-O-hex, cya-O-sya, and peo-3-O-glu are the key reason in red peels at the mature periods (Zheng et al., 2020). Therefore, color was one of the important indicators for distinguishing DZM.

3.3. Water, ash and opening rate

The analysis of Table 1 provided insights into the quality attributes of dried DZM from different regions, focusing on key parameters essential for assessing their suitability and market value. Moisture content played a critical role in the storage and quality of dried DZM. According to the data, DZM from MX exhibited the lowest moisture content at 6.72 %, whereas DZM from JY had the highest at 9.42 %. The moisture levels of all samples meet the standard set by GB/T 30391 for premium-grade dried peppercorns (≤9.5 %). Higher moisture content, such as that found in JY, could potentially lead to microbial growth and affect the shelf life and overall quality (Tarlak, 2023). Ash content indicated the presence of inorganic impurities in dried DZM. DZM from MS exhibited the highest ash content at 34.28 %, whereas DZM from JY had the lowest at 25.66 %. GB/T 30391 specifies a maximum total ash content of 5.5 % for DZM. The higher ash content suggested a higher level of impurities, possibly from soil or processing residues, which can impact both safety and product purity (Ma, Wang, Huang, Tian, & Wei, 2022a).

The opening rate referred to the ratio at which the skin of DZM naturally split open after maturation, revealing the seeds inside. This indicator was commonly used to measure the maturity and quality of DZM, as well as the degree of shell breaking during processing. DZM with a high opening rate was easier to remove the skin from during picking and processing, thus obtaining pure seeds, which was important for enhancing its commercial value. Therefore, DZM with a high opening rate was usually of better quality. In the processing of DZM, the opening rate was an important production indicator. A high opening rate could reduce the effort required to remove the skin during processing and improve efficiency. Opening rate: as can be seen from Table 1, there were more unopened DZM and DZM seeds in the samples from JY and MS. The opening rate of DZM from other origins was relatively better.

3.4. Microbiological indicators

The microbial index was a critical indicator of food safety (Bevilacqua et al., 2023). This study evaluated the total bacterial colony counts, mold and *Escherichia coli* in dried DZM samples from seven distinct regions in China to assess their sanitary quality, guiding consumer choices and agricultural practices.

Table 1 presented a ranking of DZM samples by bacterial colony count, from the lowest to the highest. The samples from HY and JJ exhibited the lowest counts, with values in the thousands. In contrast, the samples from WD, HC, MS, and JY displayed significantly higher counts in the hundred-thousands, indicating better sanitary conditions in HY, JJ, and MX. These samples likely experience fewer microorganisms due to the presence of characteristic antibacterial volatile organic compounds, which also help preserve the original flavor of the DZM.

To assess *Escherichia coli*, a BGLB broth tube validation experiment was conducted, observing gas production during culture. The samples of HY, JJ, MX, WD, HC, and JY showed no *Escherichia coli* growth, indicating levels below the detection limit of 1 log CFU/g. In contrast, the sample of MS tested positive with a *Escherichia coli* count of 4.89 log CFU/g. The presence of *Escherichia coli* in food can lead to intestinal issues and, in severe cases, produce toxins causing health problems (Bevilacqua et al., 2023). The result suggested that DZM from MS may be subject to inadequate processing or storage, which could compromise its taste and safety.

Mould detection, as reported in Table 1, revealed varying levels of contamination across the seven varieties. DZM From HY complied with the GB/T 30391–2013 mold limit of ≤4 log CFU/g, with a count of 3.45 log CFU/g, reflecting effective microbial control. Conversely, DZM from JJ, MS, HC, WD, and JY exceeded the standard, with mold counts four to five orders of magnitude higher. DZM from MX also failed to meet the standard. These results underscored the necessity for enhanced mold control in these six regions to guarantee DZM safety and quality (Gupta, Bala, & Sharma, 2018). This study contributed to the understanding of the impact of geographical origin on the microbial quality of dried DZM, thereby informing consumer choices and agricultural practices.

3.5. Heavy metal indicators

In this paper, by referencing relevant literature reports, the main trace elements in DZM were ranked as follows: Ca > Mg > S > Fe > Al > Mn > Zn > B > Cu > Ni > Pb > Cr > Mo > As> Cd > Hg > Se, The heavy metals As and Cd pose a cancer risk (Ma, Wang, Huang, Tian, & Wei, 2022b); Referring to national standards and literature reports; Pb, Cd, and As were detected in various samples of dried DZM to assess their safety. Table 1 revealed that the combined levels of arsenic, lead, and cadmium in DZM from seven production regions were within permissible limits. DZM from HY demonstrated the lowest arsenic levels, while product from WD exhibited the highest. Regarding lead, DZM from HY, WD, and HC had low concentrations, as do their cadmium levels. In contrast, DZM from MX contained the highest lead content, and products from MS and JJ exhibited elevated cadmium levels, with products from MX and JY following closely. These variations in heavy metal content can be attributed to several factors, including soil and water pollution, the organic matter and pH of the soil, agricultural practices. Soil contamination was evident when DZM roots absorb arsenic, lead, and cadmium from the soil. Industrial waste, wastewater, and pesticide contamination can lead to pollutants accumulating in the soil and subsequently in the DZM. Additionally, the improper use of pesticides and fertilizers, coupled with inadequate soil management and environmental oversight, can result in excessive heavy metal residues in the plant (Ma et al., 2022a). In conclusion, the data suggested that DZMs from HY, HC, and JY may have the lowest levels of heavy metal contamination, indicating a potentially safer product for consumers.

3.6. Qualitative and quantitative analysis of pungent compounds

The results of the standard curves for hydroxy-*α*-sanshool, hydroxy-*β*-sanshool in DZMs of different origins were quantified and characterized by external standard method, and the results were shown in Table 2. The results of the standard curves for hydroxy-*α*-sanshool, hydroxy-*β*-sanshool showed that the fitting formula of hydroxy-*α*-sanshool was: y = 28.347×-185.72, R2 = 0.9992, applicable concentration range: 0.05–0.6 mg/mL; fitting formula of hydroxy-*β*-sanshool was: y = 28.347×-185.72, R^2 = 0.9992, applicable concentration range: 0.05–0.6 mg/mL; fitting formula of hydroxy-*β*-sanshool was: y = 71.352× + 17.05, R^2 = 0.9998, applicable concentration range: 0.001–0.01 mg/mL.

Comparing the differences of hydroxy-α-sanshool in DZMs from the seven origins, the numbness and characteristic sanshool content of DZMs from MX and WD were relatively high, which could reach 124.99 ± 2.30 mg/g and 121.62 ± 1.66 mg/g; followed by: HY, JY, JJ, HC; the hydroxy-α-sanshool content of DZM from MS was the lowest 69.83 ± 0.21 mg/g. Comparison of hydroxy-α-sanshool content of 7 origins. Comparing the differences in hydroxy-β-sanshool among the seven origins, the highest hydroxy-β-sanshool content was 11.34 ± 0.09 mg/g in DZM from JY, and the lowest hydroxy-β-sanshool content was 3.04 ± 0.02 mg/g in DZM from MS. Meanwhile, comparing the main differences between red and green DZM, it was found that the hydroxy-α-sanshool of red DZM was much higher than that of green DZM. In addition to the content of hydroxy-β-sanshool in DZM from JY was significantly higher than the other groups, the hydroxy-β-sanshool of red DZM was also finely higher than that of green DZM. Hydroxy-α-sanshool and hydroxy-β-sanshool were one of the main numbing components that had been found in DZM (Yang, 2008). In the contribution of the whole numbing system, the threshold value of hydroxy-α-sanshool was 6.8 nmol/cm^2 and hydroxy-β-sanshool was 8.3 nmol/cm^2, and the amount of hydroxy-α-sanshool was much larger than that of hydroxy-β-sanshool, which overall indicated that hydroxy-α-sanshool was the key numbing component (Bader et al., 2014).

Table 2
Analysis of numbing components in DZMs from different origin.

sample	Hydroxy-α-sanshool (mg/g)	Hydroxy-β-sanshool (mg/g)
MS	69.83 ± 0.21^{f}	3.04 ± 0.02^{f}
JJ	79.86 ± 0.40^{e}	3.30 ± 0.03^{f}
JY	84.01 ± 0.70^{d}	11.34 ± 0.09^{a}
MX	124.99 ± 2.30^{a}	5.30 ± 0.12^{d}
HC	69.90 ± 0.84^{f}	9.97 ± 0.15^{b}
HY	96.07 ± 0.52^{c}	4.81 ± 0.02^{e}
WD	121.62 ± 1.66^{b}	9.60 ± 0.26^{c}
standard curves	Y = 28.347×-185.72, R^2 = 0.9992	Y = 71.352× + 17.05 R^2 = 0.9998
applicable concentration range	0.05–0.6 mg/mL	0.001–0.01 mg/mL
oral sensation[A]	tingling, paresthetic	tingling, paresthetic
threshold concn (nmol/cm^2)[A]	6.8	8.3

Note: Number of experimental repetitions (n = 3).
A:Oral sensation and threshold were from (Bader et al., 2014).

3.7. Analysis of the VOCs of the zanthoxylum bungeanum maxim samples

In this study, steam distillation was employed to extract volatile organic oils from different DZMs, and the content of these oils were compared across various origins. The results indicated that the volatile oil content was highest in the JY variety, reaching 12.53 ± 1.58 mL/100 g, while the HC variety exhibited the lowest content at 3.05 ± 0.18 mL/100 g in Fig. 2B. Additionally, GC–MS was utilized to analyze the volatile organic compounds of DZM from seven primary origins, identifying a total of 173 volatile organic compounds, including 54 terpenes, 42 alcohols, 16 aldehydes, 31 esters, 8 ketones, and 22 other types of compounds in Table 3. The volatile organic compounds in DZM were predominantly alcohols and terpenes, with linalool being the primary compound, ranging from 6.65 to 46.66 mg/mL, and D-Limonene ranging from 11.43 to 23.40 mg/mL. The specific contents of other components were detailed in Table 3. A comparison of the percentage composition of different volatile organic compounds in dried green and red DZM revealed significant differences, with alcohols being the predominant component in green DZM and terpenes in red DZM. Furthermore, the content of esters in red DZM was relatively higher compared to green DZM in Fig. 2C.

Partial least squares discriminant analysis (PLS-DA) is a common supervised discriminant analysis statistical method that effectively differentiates between the variations in DZM origins (Feng, Wang, Wang, Huang, & Kan, 2022). By analyzing the VIP values and *p*-values, key biomarkers can be identified, and cross-validation can be used to determine the accuracy of the model's discrimination (Ni et al., 2022). In this study, the same processing model was applied to classify green and red DZM and to differentiate between DZM from various origins in Fig. 3. The PLS-DA score plot revealed a clear distinction between green and red DZM, with a total score of 66.45 % for the first and second principal components. Further classification of DZM from different origins indicated a degree of similarity, particularly among green DZM samples from the three main origins, which showed strong similarity. In contrast, red DZM varieties, such as WD and HC, exhibited stronger similarity, while other origins showed weaker similarity, with a total score of 65.89 % for the first and second principal components. In the VIP score plot, both linalool and 4-methylene-1-(1-methylethyl)bicyclo [3.1.0]hexane exhibited high VIP values, indicating that these volatile organic compounds can serve as key biomarkers not only for distinguishing between green and red DZM but also for differentiating between DZM from various origins. The results of this study generally agree with previous reports, and linalool was the key component for the identification of different origins (Feng et al., 2022)。.

Through a review of relevant literature and websites (Ji et al., 2019; Liu et al., 2023; Ni et al., 2022), this study identified 33 key aroma components from the volatile organic compounds, including eucalyptol (odor: cooling, fresh, medicinal); linalool oxide (odor: floral muguet,

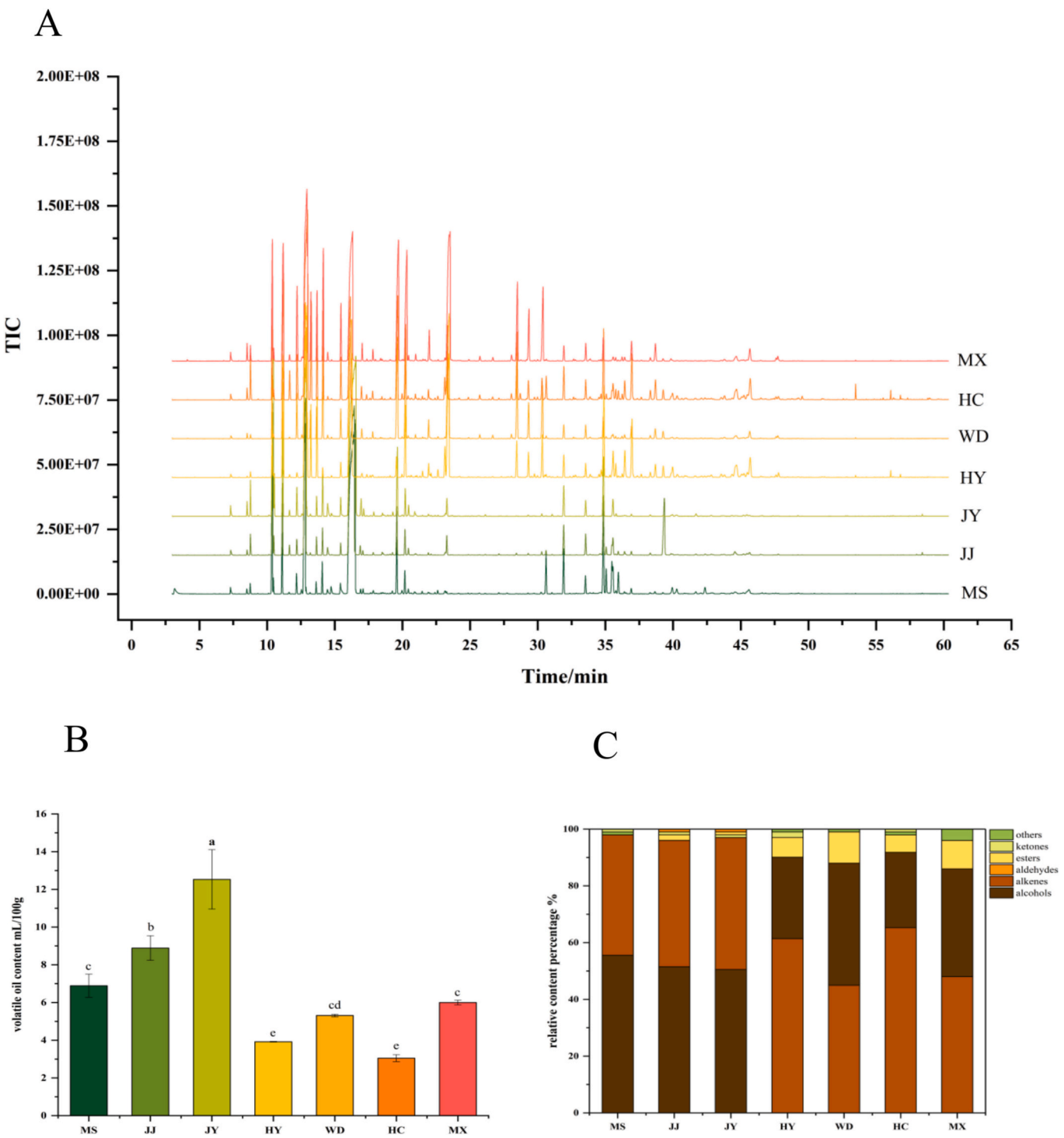

Fig. 2. GC-MS total ion chromatograms of Zanthoxylum *bungeanum maxim* under (A); comparison of essential oil content of different dried Zanthoxylum *bungeanum maxim* (B); percentage area of different volatile component species (C); heat map and clustering result analysis of key aroma compound (D).

metallic); linalool (odor: floral muguet, citrus); 2,6-octadien-1-ol, 3,7-dimethyl-, (*Z*)- (odor: sweet, floral, fruity, rose); carveol (odor: caraway, solvent); *α*-pinene (odor: pine); camphene (odor: camphor, pungent); sabinene (odor: terpinenic, citrus, pine); *β*-pinene (odor: pine, resin); *α*-phellandrene (odor: terpenic, green, peppery); 3-carene (odor: lemon, resin); D-limonene (odor: lemon, citrus); *β*-phellandrene (odor: mint, terpentine); *γ*-terpinene (odor: terpenic); terpinolene (odor: citrus, lime); *β*-cubebene (odor: woody); caryophyllene (odor: spicy, clove); humulene (odor: spicy, woody); bicyclogermacrene (odor: fragrance); *α*-farnesene (odor: citrus, lavender herbal, bergamot, neroli, green); caryophyllene oxide (odor: sweet, fresh, dry woody, spicy); β-ocimene (odor: green, metallic); octanal (odor: aldehydic, citrus orange); nonanal (odor: green, cucumber); citronellal (odor: floral, rose, fatty, citrus); 2-nonenal, (*E*)- (odor: cucumber, melon); decanal (odor: aldehydic, waxy, orange, peel); undecanal (odor: fresh, fruity, orange peel); acetic acid, heptyl ester (odor: fruity, harsh); Linalyl acetate (odor: floral,

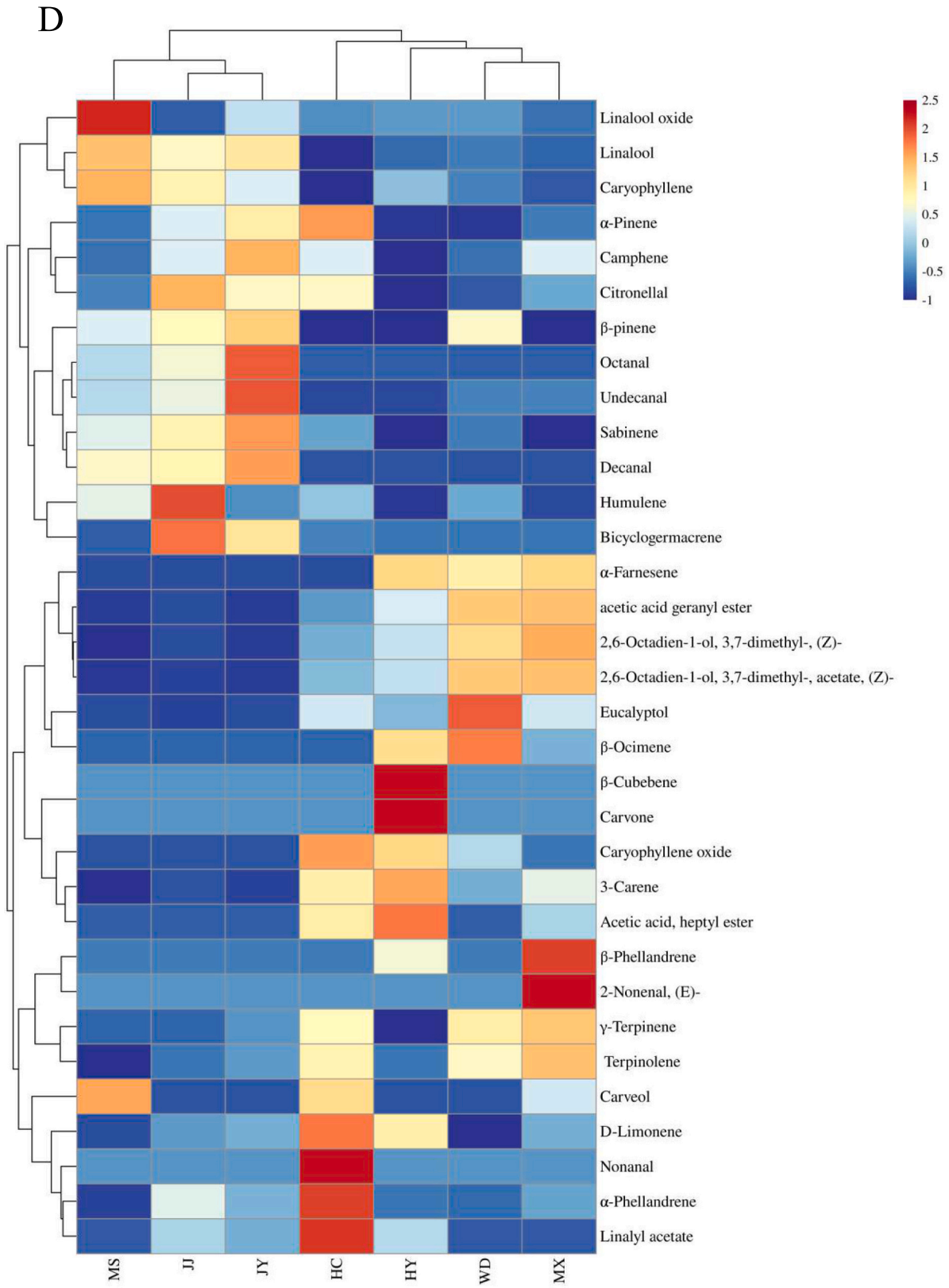

Fig. 2. (*continued*).

sweet); 2,6-octadien-1-ol, 3,7-dimethyl-, acetate, (Z)- (odor: floral, rose, soapy); acetic acid geranyl ester (odor: floral, rose); and carvone (odor: sweet, spearmint, herbal, minty).

The overall flavor profile of DZM was characterized by spicy, herbal, sweet, floral, and citrus notes. As depicted in the clustering heatmap in Fig. 2, green and red DZM were clearly distinguished. In green DZM, the

Table 3
Analysis of volatile components in DZMs from different origin by GC–MS.

No.	RI[A]	CAS	Compound	Formula[C]D	mg/mL[CD]							Idf[B]
					MS	JJ	JY	HY	WD	HC	MX	
alcohols												
1	1028	470–82-6	Eucalyptol	C10H18O	0.18 ± 0.01d	0.09 ± 0.01d	0.20 ± 0.01d	1.26 ± 0.09c	4.63 ± 0.23a	2.11 ± 0.11b	2.02 ± 0.10b	MS, RI
2	1033	546–79-2	4-Thujanol	C10H18O	0.27 ± 0.01c	0.45 ± 0.03b	0.67 ± 0.05a	0.03 ± 0.00f	0.17 ± 0.01d	0.10 ± 0.01e	0.24 ± 0.01c	MS, RI
3	1068	111–87-5	1-Octanol	C8H18O	0.01 ± 0.00a	–	0.01 ± 0.00a	–	–	–	–	MS, RI
4	1087	5989-33-3	Linalool oxide	C10H18O2	0.36 ± 0.04a	0.03 ± 0.00e	0.14 ± 0.01b	0.07 ± 0.00 cd	0.07 ± 0.00c	0.06 ± 0.00 cde	0.04 ± 0.00de	MS, RI
5	1101	78–70-6	Linalool	C15H26O	46.66 ± 1.50a	36.6 ± 3.67c	40.85 ± 2.82b	15.96 ± 1.1d	17.4 ± 0.88d	6.65 ± 0.34e	15.41 ± 0.79d	MS, RI
6	1128	29,803–82-5	2-Cyclohexen-1-ol, 1-methyl-4-(1-methylethyl)-,cis-	C10H18O	0.17 ± 0.01c	0.13 ± 0.01d	0.16 ± 0.01c	0.15 ± 0.01c	0.29 ± 0.01b	0.29 ± 0.02ab	0.31 ± 0.02a	MS, RI
7	1142	29,803–81-4	2-Cyclohexen-1-ol, 1-methyl-4-(1-methylethyl)-,trans-	C10H18O	0.2 ± 0.01d	0.18 ± 0.01de	0.24 ± 0.02c	0.16 ± 0.01e	0.41 ± 0.02b	0.43 ± 0.02ab	0.45 ± 0.02a	MS, RI
8	1148	89–79-2	Isopulegol	C10H18O	–	–	–	–	0.07 ± 0.00a	–	0.06 ± 0.00b	MS, RI
9	1148	7299-42-5	Cyclohexanemethanol, α,α-dimethyl-4-methylene-	C10H18O	–	–	–	–	–	0.09 ± 0.00a	–	MS, RI
10	1175	20,126–76-5	3-Cyclohexen-1-ol, 4-methyl-1-(1-methylethyl)-,(*R*)-	C10H18O	2.36 ± 0.04d	2.36 ± 0.16d	2.98 ± 0.21c	0.72 ± 0.05e	7.66 ± 0.39a	5.92 ± 0.30b	7.57 ± 0.39a	MS, RI
11	1187	10,482–56-1	L-α-Terpineol	C10H18O	0.88 ± 0.02d	1.14 ± 0.08d	0.99 ± 0.07d	3.36 ± 0.23c	5.65 ± 0.29b	3.28 ± 0.17c	6.08 ± 0.31a	MS, RI
12	1193	491–04-3	Piperitol	C10H18O	0.05 ± 0.01c	0.06 ± 0.00bc	0.07 ± 0.00b	0.05 ± 0.00c	0.18 ± 0.01a	0.19 ± 0.01a	0.19 ± 0.01a	MS, RI
13	1206	16,721–39-4	2-Cyclohexen-1-ol, 3-methyl-6-(1-methylethyl)-, trans-	C10H18O	–	–	–	0.04 ± 0.00c	0.12 ± 0.01b	0.11 ± 0.01b	0.13 ± 0.01a	MS, RI
14	1210	74,410–00-7	(−)-trans-Isopiperitenol	C10H16O	–	–	–	0.07 ± 0.00b	–	0.08 ± 0.00a	–	MS, RI
15	1228	106–25–2	2,6-Octadien-1-ol, 3,7-dimethyl-, (*Z*)-	C10H18O	0.05 ± 0.00f	0.11 ± 0.01e	0.08 ± 0.01e	0.55 ± 0.04c	0.88 ± 0.04b	0.35 ± 0.02d	1.02 ± 0.05a	MS, RI
16	1229	1197-06-4	2-Cyclohexen-1-ol, 2-methyl-5-(1-methylethenyl)-, cis-	C10H16O	0.11 ± 0.00b	–	0.04 ± 0.00e	0.24 ± 0.02a	0.07 ± 0.00c	0.11 ± 0.01b	0.06 ± 0.00d	MS, RI
17	1252	99–48-9	Carveol	C10H16O	0.06 ± 0.02a	–	–	–	–	0.05 ± 0.00a	0.03 ± 0.00b	MS, RI
18	1255	106–24-1	Geraniol	C10H18O	0.12 ± 0.01a	–	0.09 ± 0.01b	–	–	–	–	MS, RI
19	1273	18,409–18-2	2-Decen-1-ol, (*E*)-	C10H20O	0.02 ± 0.01b	–	–	0.06 ± 0.00a	–	–	–	MS, RI
20	1293	536–60-7	p-Cymen-7-ol	C10H14O	0.04 ± 0.01a	–	0.02 ± 0.00c	–	0.02 ± 0.00c	0.03 ± 0.00b	–	MS, RI
21	1473	69,064–37-5	trans-2-Dodecen-1-ol	C12H24O	–	0.02 ± 0.00b	0.15 ± 0.01a	–	–	–	–	MS, RI
22	1547	639–99-6	Elemol	C15H26O	0.05 ± 0.02e	0.15 ± 0.01d	0.07 ± 0.00e	0.26 ± 0.02b	0.20 ± 0.01c	0.45 ± 0.02a	0.13 ± 0.01d	MS, RI
23	1561	7212-44-4	1,6,10-Dodecatrien-3-ol, 3,7,11-trimethyl-	C15H26O	0.08 ± 0.00d	–	–	0.62 ± 0.04a	0.45 ± 0.02c	0.49 ± 0.03b	–	MS, RI
24	1565	40,716–66-3	1,6,10-Dodecatrien-3-ol, 3,7,11-trimethyl-, (E)-	C15H26O	–	4.92 ± 0.34a	–	–	–	–	0.1 ± 0.01b	MS, RI
25	1572	112–70-9	n-Tridecan-1-ol	C13H28O	0.01 ± 0.00e	0.05 ± 0.00b	–	0.15 ± 0.01a	0.02 ± 0.00d	0.03 ± 0.00c	–	MS, RI
26	1577	6750-60-3	Spatulenol	C15H24O	0.38 ± 0.04b	–	0.1 ± 0.01c	0.65 ± 0.04a	0.11 ± 0.01c	0.39 ± 0.02b	–	MS, RI
27	1582	77,171–55–2	(−)-Spathulenol	C15H24O	–	–	–	0.2 ± 0.01a	–	–	–	MS, RI
28	1591	552–02-3	Viridiflorol	C15H26O	0.05 ± 0.01b	–	–	0.09 ± 0.01a	–	–	–	MS, RI
29	1594	465–28-1	Carotol	C15H26O	–	0.07 ± 0.00c	–	0.24 ± 0.02a	–	0.17 ± 0.01b	–	MS, RI
30	1620	1460–73-7	Agarospirol	C15H26O	–	0.06 ± 0.00d	0.02 ± 0.00b	0.06 ± 0.00b	–	0.09 ± 0.00a	0.04 ± 0.00c	MS, RI
31	1630	19,912–67-5	1-epi-cubenol	C15H26O	–	–	–	0.23 ± 0.02a	0.03 ± 0.00c	0.14 ± 0.01b	–	MS, RI
32	1635	1209-71-8	γ-Eudesmol	C15H26O	–	0.08 ± 0.01d	0.07 ± 0.00d	0.21 ± 0.01b	0.11 ± 0.01c	0.27 ± 0.01a	0.1 ± 0.01c	MS, RI
33	1638	473–15-4	β-Eudesmol	C15H26O	0.07 ± 0.01bc	0.09 ± 0.01b	0.08 ± 0.01bc	0.21 ± 0.01a	0.07 ± 0.00 cd	–	0.06 ± 0.00d	MS, RI
34	1640	5937-11-1	τ-Cadinol	C15H26O	–	–	–	0.18 ± 0.01a	–	0.14 ± 0.01b	0.05 ± 0.00c	MS, RI
35	1652	473–16-5	α-eudesmol	C15H26O	–	0.13 ± 0.01b	0.10 ± 0.01c	0.24 ± 0.02a	–	–	0.05 ± 0.00d	MS, RI
36	1653	481–34-5	α-Cadinol	C15H26O	0.37 ± 0.01c	0.24 ± 0.02d	0.06 ± 0.00e	1.29 ± 0.09a	0.59 ± 0.03b	1.34 ± 0.07a	0.64 ± 0.03b	MS, RI

(*continued on next page*)

Table 3 (*continued*)

No.	RI[A]	CAS	Compound	Formula[C]D	mg/mL[CD]							Idf[B]
					MS	JJ	JY	HY	WD	HC	MX	
37	1692	515–69-5	α-Bisabolol	C15H26O	–	–	–	–	0.13 ± 0.01a	–	0.12 ± 0.01a	MS, RI
38	1740	4602-84-0	2,6,10-Dodecatrien-1-ol, 3,7,11-trimethyl-	C15H26O	–	–	–	–	–	0.15 ± 0.01a	–	MS, RI
39	–	19,894–97-4	(−)-Myrtenol	C10H16O	0.23 ± 0.03a	–	–	–	–	–	–	MS
40	–	107–19-7	Propargyl alcohol	C3H4O	–	–	–	–	1.95 ± 0.10a	–	–	MS
41	–	56,298–90-9	2-Heptanol, 4-methyl-	C8H18O	–	–	–	0.02 ± 0.00a	–	–	–	MS
42	–	1724-39-6	Cyclododecanol	C12H24O	–	–	0.03 ± 0.00a	–	–	–	–	MS
43	922	80–56-8	α-Pinene	C10H16	0.37 ± 0.03d	0.85 ± 0.06c	1.09 ± 0.08b	0.19 ± 0.01e	0.18 ± 0.01e	1.41 ± 0.07a	0.38 ± 0.02d	MS, RI
44	930	2867–5-2	3-Thujene	C10H16	0.17 ± 0.01d	0.21 ± 0.01c	0.43 ± 0.03a	0.04 ± 0.00e	0.23 ± 0.01c	0.34 ± 0.02b	0.41 ± 0.02a	MS, RI
45	936	7785–26-4	(1*S*)-2,6,6-Trimethylbicyclo[3.1.1]hept-2-ene	C10H16	0.06 ± 0.00d	–	0.06 ± 0.00d	2.45 ± 0.17b	–	3.47 ± 0.18a	1.81 ± 0.09c	MS, RI
46	953	79–92-5	Camphene	C10H16	0.01 ± 0.00a	0.02 ± 0.00a	0.03 ± 0.00a	–	0.01 ± 0.00a	0.02 ± 0.00a	0.02 ± 0.00a	MS, RI
47	975	3387-41-5	Bicyclo[3.1.0]hexane, 4-methylene-1-(1-methylethyl)-	C10H16	6.91 ± 0.19c	8.66 ± 0.6b	11.93 ± 0.82a	–	2.57 ± 0.13e	3.54 ± 0.18d	0.29 ± 0.02f	MS, RI
48	986	127–91-3	β-pinene	C10H16	2.54 ± 0.09c	3.05 ± 0.21b	3.93 ± 0.27a	–	2.95 ± 0.15b	–	–	MS, RI
49	1002	99–83-2	α-Phellandrene	C10H16	–	0.43 ± 0.03b	0.23 ± 0.02c	0.11 ± 0.01d	0.08 ± 0.00d	0.93 ± 0.05a	0.19 ± 0.01c	MS, RI
50	1003	499–97-8	Cyclohexane, 1-methylene-4-(1-methylethenyl)-	C10H16	0.07 ± 0.00b	–	–	6.31 ± 0.44a	–	6.36 ± 0.33a	–	MS, RI
51	1012	13,466–78-9	3-Carene	C10H16	0.41 ± 0.01f	0.72 ± 0.05e	0.62 ± 0.04e	2.88 ± 0.20a	1.26 ± 0.06d	2.29 ± 0.12b	1.91 ± 0.10c	MS, RI
52	1020	99–86-5	1,3-Cyclohexadiene, 1-methyl-4-(1-methylethyl)-	C10H16	0.35 ± 0.03a	–	–	0.01 ± 0.00d	0.22 ± 0.01b	–	0.17 ± 0.01c	MS, RI
53	1022	29,050–33-7	(+)-4-Carene	C10H16	0.76 ± 0.01e	0.72 ± 0.05e	1.00 ± 0.07d	0.20 ± 0.01f	2.02 ± 0.10b	1.95 ± 0.10b	2.51 ± 0.13a	MS, RI
54	1032	138–86-3	D-Limonene	C10H16	12.74 ± 0.22d	14.62 ± 1.01c	15.27 ± 1.05c	19.79 ± 1.36b	11.43 ± 0.58d	23.40 ± 1.20a	15.17 ± 0.78c	MS, RI
55	1033	535–77-3	β-Cymene	C10H14	–	–	0.00 ± 0.00d	–	0.03 ± 0.00c	0.07 ± 0.00a	0.04 ± 0.00b	MS, RI
56	1034	555–10-2	β-Phellandrene	C10H16	–	–	–	1.62 ± 0.11b	–	–	3.65 ± 0.19a	MS, RI
57	1051	3779-61-1	trans-β-Ocimene	C10H16	–	0.09 ± 0.01b	–	–	1.05 ± 0.05a	–	–	MS, RI
58	1060	99–85-4	γ-Terpinene	C10H16	1.16 ± 0.03e	1.13 ± 0.08e	1.52 ± 0.1d	0.3 ± 0.02f	3.19 ± 0.16b	2.97 ± 0.15c	3.69 ± 0.19a	MS, RI
59	1081	586–62-9	Terpinolene	C10H16	–	0.50 ± 0.03d	0.63 ± 0.04c	0.50 ± 0.03d	1.22 ± 0.06b	1.30 ± 0.07b	1.57 ± 0.08a	MS, RI
60	1118	18,368–95-1	1,3,8-p-Menthatriene	C10H14	–	–	–	–	0.01 ± 0.00a	0.02 ± 0.00a	0.02 ± 0.00a	MS, RI
61	1131	7216–56-0	2,4,6-Octatriene, 2,6-dimethyl-, (E, Z)-	C10H16	–	–	–	0.11 ± 0.01b	0.05 ± 0.00c	0.16 ± 0.01a	0.06 ± 0.00c	MS, RI
62	1152	43,219–68-7	Ethanone, 1-(1,4-dimethyl-3-cyclohexen-1-yl)-	C10H16O	–	–	–	–	–	0.09 ± 0.00a	–	MS, RI
63	1390	13,744–15-5	β-Cubebene	C15H24	–	–	–	0.05 ± 0.00a	–	–	–	MS, RI
64	1392	515–13-9	β-Elemen	C15H24	1.79 ± 0.13a	0.25 ± 0.02c	0.03 ± 0.00f	0.14 ± 0.01d	0.14 ± 0.01d	0.90 ± 0.05b	0.06 ± 0.00d	MS, RI
65	1419	469–61-4	α-Cedrene	C15H24	–	–	–	–	0.18 ± 0.01b	–	0.21 ± 0.01a	MS, RI
66	1420	87–44-5	Caryophyllene	C15H24	2.05 ± 0.07a	1.63 ± 0.11b	1.32 ± 0.09c	0.98 ± 0.07d	0.68 ± 0.03e	–	0.52 ± 0.03f	MS, RI
67	1431	18,252–44-3	β-Copaene	C15H24	4.47 ± 0.12a	3.36 ± 0.23b	2.69 ± 0.19c	4.69 ± 0.32a	1.09 ± 0.06d	2.97 ± 0.15c	0.74 ± 0.04e	MS, RI
68	1433	29,873–99-2	γ-Elemene	C15H24	1.12 ± 0.02b	0.02 ± 0.00f	0.01 ± 0.00f	1.23 ± 0.09a	0.30 ± 0.02d	0.77 ± 0.04c	0.21 ± 0.01e	MS, RI
69	1440	654,486	α-Guaiene	C15H24	–	–	–	0.06 ± 0.00a	0.04 ± 0.00b	–	0.03 ± 0.00c	MS, RI
70	1461	25,246–27-9	Alloaromadendrene	C15H24	0.06 ± 0.00d	0.07 ± 0.00c	–	0.15 ± 0.01a	–	0.13 ± 0.01b	–	MS, RI
71	1466	30,021–74-0	γ-Muurolene		0.11 ± 0.01c	–	–	0.33 ± 0.02a	0.07 ± 0.00d	0.21 ± 0.01b	0.04 ± 0.00e	MS, RI
72	1470	22,567–17-5	γ-Gurjunene	C15H24	–	–	–	0.11 ± 0.01a	–	–	–	MS, RI
73	1475	17,066–67-0	β-Selinene	C15H24	0.96 ± 0.07a	0.4 ± 0.03b	–	0.03 ± 0.00e	0.10 ± 0.01d	0.20 ± 0.01c	0.01 ± 0.00e	MS, RI

(*continued on next page*)

Table 3 (*continued*)

No.	RI[A]	CAS	Compound	Formula[C]D	mg/mL[CD]							Idf[B]
					MS	JJ	JY	HY	WD	HC	MX	
74	1481	6753-98-6	Humulene	C15H24	0.84 ± 0.01b	1.11 ± 0.08a	0.67 ± 0.05c	0.57 ± 0.04d	0.7 ± 0.04c	0.74 ± 0.04c	0.59 ± 0.03d	MS, RI
75	1483	515–17-3	γ-Selinene	C15H24	1.65 ± 0.06a	–	–	–	–	–	–	MS, RI
76	1489	54,324–03-7	Bicyclosesquiphellandrene	C15H24	–	–	–	0.09 ± 0.01a	0.02 ± 0.00c	0.05 ± 0.00b	0.02 ± 0.00c	MS, RI
77	1496	24,703–35-3	Bicyclogermacrene	C15H24	–	1.06 ± 0.07a	0.74 ± 0.05b	0.07 ± 0.00c	0.07 ± 0.00c	0.09 ± 0.00c	0.06 ± 0.00c	MS, RI
78	1497	41,702–63-0	Epizonarene	C15H24	0.24 ± 0.02b	0.39 ± 0.03a	0.06 ± 0.00c	0.04 ± 0.00 cd	0.01 ± 0.00e	–	0.02 ± 0.00de	MS, RI
79	1497	31,983–22-9	α-Muurolene	C15H24	–	–	–	0.59 ± 0.04a	0.13 ± 0.01c	0.41 ± 0.02b	0.12 ± 0.01c	MS, RI
80	1508	3691-11-0	δ-Guaijene	C15H24	0.02 ± 0.00a	–	–	–	–	–	–	MS, RI
81	1508	502–61-4	α-Farnesene	C15H24	–	–	–	0.14 ± 0.01c	0.12 ± 0.01b	–	0.14 ± 0.01a	MS, RI
82	1513	29,837–12-5	Cubenene	C15H24	0.03 ± 0.00c	–	–	0.09 ± 0.01a	–	0.05 ± 0.00b	–	MS, RI
83	1515	39,029–41-9	γ-Cadinene	C15H24	0.10 ± 0.01ef	0.19 ± 0.01 cd	0.04 ± 0.00f	1.36 ± 0.09a	0.24 ± 0.01c	0.84 ± 0.04b	0.15 ± 0.01de	MS, RI
84	1524	483–76-1	δ-Cadinene	C15H24	0.26 ± 0.02c	0.23 ± 0.02 cd	0.10 ± 0.01d	2.92 ± 0.20a	0.76 ± 0.04b	–	0.73 ± 0.04b	MS, RI
85	1535	24,406–05-1	α-Cadinene	C15H24	–	–	–	0.17 ± 0.01a	0.05 ± 0.00c	0.11 ± 0.01b	0.04 ± 0.00c	MS, RI
86	1540	21,391–99-1	α-Calacorene	C15H20	–	–	–	0.04 ± 0.00a	–	0.02 ± 0.00a	–	MS, RI
87	1557	15,423–57-1	Germacrene B	C15H24	0.13 ± 0.01d	0.03 ± 0.00e	–	0.73 ± 0.05b	0.65 ± 0.03c	1.00 ± 0.05a	0.76 ± 0.04b	MS, RI
88	1583	1139–30-6	Caryophyllene oxide	C15H24O	0.33 ± 0.02a	–	–	0.19 ± 0.01c	0.09 ± 0.00d	0.23 ± 0.01b	0.02 ± 0.00e	MS, RI
89	1608	19,888–34-7	(1R,3E,7E,11R)-1,5,5,8-Tetramethyl-12-oxabicyclo[9.1.0] dodeca-3,7-diene	C15H24O	0.06 ± 0.00a	–	–	–	0.05 ± 0.00b	–	–	MS, RI
90	1960	20,016–73-3	m-Camphorene	C20H32	–	–	0.01 ± 0.00c	0.16 ± 0.01b	0.01 ± 0.00c	0.22 ± 0.01a	0.01 ± 0.00c	MS, RI
91	1994	20,016–72-2	p-Camphorene	C20H32	–	–	–	0.08 ± 0.01b	–	0.11 ± 0.01a	–	MS, RI
92	–	18,172–67-3	Bicyclo[3.1.1]heptane, 6,6-dimethyl-2-methylene-, (1*S*)-	C10H16	0.51 ± 0.04c	0.63 ± 0.04c	1.02 ± 0.07b	0.16 ± 0.01d	0.18 ± 0.01d	0.31 ± 0.02d	4.60 ± 0.24a	MS
93	–	3856-25-5	Copaene	C15H24	0.05 ± 0.00c	0.03 ± 0.00d	–	0.15 ± 0.01a	0.03 ± 0.00d	0.10 ± 0.01b	0.03 ± 0.00d	MS
94	–	20,307–84-0	δ-Elemene	C15H24	0.04 ± 0.00a	–	–	–	–	–	–	MS
95	–	13,877–91-3	β-Ocimene	C10H16	–	–	–	8.26 ± 0.57b	11.18 ± 0.57a	–	2.23 ± 0.11c	MS
96	–	75,023–40-4	1,5-Cyclodecadiene, 1,5-dimethyl-8-(1-methylethenyl)-, [S-(Z,E)]-		–	–	0.01 ± 0.00a	–	–	–	–	MS
aldehydes												
97	916	142–83-6	2,4-Hexadienal, (E,*E*)-	C6H8O	–	–	–	–	–	–	0.01 ± 0.00a	MS, RI
98	1007	124–13-0	Octanal	C8H16O	0.02 ± 0.00c	0.03 ± 0.00b	0.06 ± 0.00a	–	–	–	–	MS, RI
99	1102	124–19-6	Nonanal	C9H18O	–	–	–	–	–	0.05 ± 0.00a	–	MS, RI
100	1153	53,447–45-3	Lilac aldehyde B	C10H16O2	0.04 ± 0.00c	–	–	–	0.06 ± 0.00b	–	0.07 ± 0.00a	MS, RI
101	1154	106–23-0	Citronellal	C10H18O	0.04 ± 0.00e	0.12 ± 0.01a	0.09 ± 0.01c	–	0.03 ± 0.00f	0.09 ± 0.00b	0.05 ± 0.00d	MS, RI
102	1164	18,829–56-6	2-Nonenal, (E)-	C9H16O	–	–	–	–	–	–	0.01 ± 0.00	MS, RI
103	1204	112–31-2	Decanal	C10H20O	0.10 ± 0.01b	0.11 ± 0.01b	0.16 ± 0.01a	–	–	–	–	MS, RI
104	1264	3913-81-3	2-Decenal, (E)-	C10H18O	–	–	–	–	–	–	0.02 ± 0.00	MS, RI
105	1270	2111-75-3	1-Cyclohexene-1-carboxaldehyde, 4-(1-methylethenyl)-	C10H14O	–	0.01 ± 0.00a	–	0.05 ± 0.00a	–	–	–	MS, RI
106	1283	1197-15-5	4-Isopropylcyclohexa-1,3-dienecarbaldehyde	C10H14O	–	0.01 ± 0.00a	0.01 ± 0.00a	–	–	–	–	MS, RI
107	1303	112–44-7	Undecanal	C11H22O	0.03 ± 0.00c	0.04 ± 0.00b	0.08 ± 0.01a	–	0.01 ± 0.00d	–	0.01 ± 0.00d	MS, RI
108	1405	112–54-9	Dodecanal	C12H24O	0.01 ± 0.00a	–	0.01 ± 0.00a	–	–	–	–	MS, RI

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Table 3 (*continued*)

No.	RI[A]	CAS	Compound	Formula[CD]	mg/mL[CD]							Idf[B]
					MS	JJ	JY	HY	WD	HC	MX	
109	1518	10,486–19-8	Tridecanal	C13H26O	0.01 ± 0.00a	–	0.02 ± 0.00a	–	–	–	–	MS, RI
110	1817	629–80-1	Hexadecanal	C16H32O	–	–	0.01 ± 0.00a	–	–	–	–	MS, RI
111	–	34,246–57-6	3-Isopropylbenzaldehyde	C10H12O	0.06 ± 0.00a	0.02 ± 0.00c	0.03 ± 0.00b	0.02 ± 0.00c	0.02 ± 0.00c	0.06 ± 0.00a	0.01 ± 0.00d	MS
112	–	18,486–69-6	(1*R*)-(−)-Myrtenal	C10H14O	–	0.31 ± 0.02b	0.39 ± 0.03a	–	–	–	–	MS
esters												
113	938	539–90-2	Butanoic acid, 2-methylpropyl ester	C8H16O2	–	–	–	0.01 ± 0.00a	–	0.01 ± 0.00a	–	MS, RI
114	1115	112–06-1	Acetic acid, heptyl ester	C9H18O2	–	–	–	0.03 ± 0.00a	–	0.02 ± 0.00a	0.01 ± 0.00a	MS, RI
115	1148	105–79-3	Hexanoic acid, 2-methylpropyl ester	C10H20O2	–	–	–	0.01 ± 0.00	–	–	–	MS, RI
116	1177	101–41-7	Benzeneacetic acid, methyl ester	C9H10O2	–	–	–	0.04 ± 0.00a	–	–	–	MS, RI
117	1187	16,491–36-4	Butanoic acid, 3-hexenyl ester, (*Z*)-	C10H18O2	–	–	–	–	–	0.01 ± 0.00a	0.01 ± 0.00a	MS, RI
118	1211	112–14-1	Acetic acid, octyl ester	C10H20O2	–	–	–	–	–	0.04 ± 0.00a	–	MS, RI
119	1256	115–95-7	Linalyl acetate	C12H20O2	–	0.93 ± 0.06b	0.60 ± 0.04c	1.04 ± 0.07b	–	3.16 ± 0.16a	–	MS, RI
120	1261	2270–60-2	6-Octenoic acid, 3,7-dimethyl-, methyl ester	C11H20O2	–	0.01 ± 0.00a	0.02 ± 0.00a	–	–	–	–	MS, RI
121	1282	5655-61-8	Bicyclo[2.2.1]heptan-2-ol, 1,7,7-trimethyl-, acetate, (1S-endo)-	C12H20O2	0.05 ± 0.00b	0.03 ± 0.00c	0.01 ± 0.00d	0.03 ± 0.00c	0.06 ± 0.00a	–	0.06 ± 0.00a	MS, RI
122	1290	20,777–39-3	lavandulyl acetate	C12H20O2	–	–	0.10 ± 0.01a	–	–	–	–	MS, RI
123	1295	1686-15-3	trans-Pinocarvyl acetate	C12H18O2	0.05 ± 0.00a	0.03 ± 0.00a	–	–	–	–	–	MS, RI
124	1316	93,836–50-1	δ-Terpineol, acetate	C12H20O2	–	–	–	0.05 ± 0.00d	0.19 ± 0.01a	0.12 ± 0.01c	0.13 ± 0.01b	MS, RI
125	1333	120–50-3	Benzoic acid, 2-methylpropyl ester	C11H14O2	–	0.01 ± 0.00a	0.01 ± 0.00a	–	–	–	–	MS, RI
126	1350	80–26-2	α-Terpinyl acetate	C12H20O2	–	0.1 ± 0.01d	0.02 ± 0.00d	1.55 ± 0.11c	4.57 ± 0.23a	–	3.36 ± 0.17b	MS, RI
127	1354	150–84-5	6-Octen-1-ol, 3,7-dimethyl-, acetate	C12H22O2	0.01 ± 0.00c	0.01 ± 0.00c	–	–	–	0.24 ± 0.01a	0.03 ± 0.00b	MS, RI
128	1360	1205-42-1	2-Cyclohexen-1-ol, 2-methyl-5-(1-methylethenyl)-, acetate, cis-	C12H18O2	–	–	–	0.02 ± 0.00a	–	–	0.04 ± 0.00a	MS, RI
129	1362	141–12-8	2,6-Octadien-1-ol, 3,7-dimethyl-, acetate, (Z)-	C12H20O2	0.03 ± 0.00d	0.09 ± 0.01d	0.05 ± 0.00d	1.04 ± 0.07b	1.93 ± 0.10a	0.74 ± 0.04c	1.98 ± 0.10a	MS, RI
130	1436	15,111–96-3	p-Mentha-1,8-dien-7-yl acetate	C12H18O2	±	–	–	0.11 ± 0.01a	0.04 ± 0.00d	0.05 ± 0.00b	0.04 ± 0.00c	MS, RI
131	1444	103–52-6	β-Phenylethyl butyrate	C12H16O2	–	–	–	0.03 ± 0.00a	–	–	–	MS, RI
132	1480	5451-67-2	Thymol isobutyrate	C14H20O2	–	–	–	–	–	0.02 ± 0.00a	–	MS, RI
133	1490	140–26-1	Butanoic acid, 3-methyl-, 2-phenylethyl ester	C13H18O2	–	–	–	0.02 ± 0.00a	–	–	–	MS, RI
134	1727	134–28-1	Guaiol acetate	C17H28O2	–	0.07 ± 0.00b	–	0.15 ± 0.01a	0.04 ± 0.00c	–	–	MS, RI
135	1839	4128–17-0	2,6,10-Dodecatrien-1-ol, 3,7,11-trimethyl-, acetate, (E,E)-	C17H28O2	0.01 ± 0.00d	0.02 ± 0.00 cd	–	0.05 ± 0.00b	0.03 ± 0.00c	0.38 ± 0.02a	0.02 ± 0.00 cd	MS, RI
136	1841	94–47-3	Benzoic acid, 2-phenylethyl ester	C15H14O2	–	0.01 ± 0.00a	0.01 ± 0.00a	–	–	–	–	MS, RI
137	1924	102–20-5	Phenethyl phenylacetate	C16H16O2	0.04 ± 0.00a	–	–	–	–	–	–	MS, RI
138	1926	112–39-0	Hexadecanoic acid, methyl ester	C17H34O2	–	–	–	–	–	0.05 ± 0.00a	–	MS, RI
139	–	1079-01-2	Myrtenyl acetate	C12H18O2	0.06 ± 0.00b	0.08 ± 0.01a	0.01 ± 0.00c	–	–	0.06 ± 0.00b	–	MS
140	–	3886-78-0	cis-p-Mentha-2,8-dien-1-ol	C10H16O	0.05 ± 0.00b	–	0.04 ± 0.00c	0.07 ± 0.00a	0.03 ± 0.00d	–	–	MS
141	–	105–87-3	acetic acid geranyl ester	C12H20O2	–	0.18 ± 0.01d	–	1.91 ± 0.13b	3.18 ± 0.16a	0.82 ± 0.04c	3.28 ± 0.17a	MS
142	–	600–22-6	Propanoic acid, 2-oxo-, methyl ester	C4H6O3	–	–	–	–	–	–	0.03 ± 0.00a	MS
143	–	2258-65-3	3-Pentenoic acid, 4-methyl-, methyl ester	C7H12O2	–	–	0.01 ± 0.00a	–	–	–	–	MS

(*continued on next page*)

Table 3 (*continued*)

No.	RI[A]	CAS	Compound	Formula[C]	mg/mL[CD]							Idf[B]
					MS	JJ	JY	HY	WD	HC	MX	
ketones												
144	1105	546–80-5	Thujone	C10H16O	–	–	–	–	–	–	0.01 ± 0.00a	MS, RI
145	1116	471–15-8	β-Thujone	C10H16O	0.19 ± 0.01c	0.45 ± 0.03b	0.73 ± 0.05a	0.03 ± 0.00d	0.01 ± 0.00d	–	–	MS, RI
146	1183	500–02-7	2-Cyclohexen-1-one, 4-(1-methylethyl)-	C9H14O	0.03 ± 0.00d	–	0.05 ± 0.00b	–	0.03 ± 0.00d	0.12 ± 0.01a	0.04 ± 0.00c	MS, RI
147	1189	122–00-9	p-Acetyltoluene	C9H10O	–	–	–	0.01 ± 0.00a	–	–	–	MS, RI
148	1234	2244-16-8	D-Carvone	C10H14O	0.15 ± 0.01b	–	0.03 ± 0.00e	0.31 ± 0.02a	0.10 ± 0.01d	0.12 ± 0.01c	0.04 ± 0.00e	MS, RI
149	1243	99–49-0	Carvone	C10H14O	–	–	–	0.04 ± 0.00a	–	–	–	MS, RI
150	1253	89–81-6	Piperitone	C10H16O	0.23 ± 0.02c	0.06 ± 0.00d	0.05 ± 0.00d	1.35 ± 0.09a	0.07 ± 0.00d	0.83 ± 0.04b	0.1 ± 0.01d	MS, RI
151	1296	112–12-9	2-Undecanone	C11H22O	0.01 ± 0.00a	–	–	–	–	–	–	MS, RI
other												
152	693	3208-16-0	Furan, 2-ethyl-	C6H8O	–	–	0.01 ± 0.00a	0.01 ± 0.00a	0.01 ± 0.00a	0.01 ± 0.00a	–	MS, RI
153	792	4229-91-8	Furan, 2-propyl-	C7H10O	–	–	–	–	–	–	0.00 ± 0.00a	MS, RI
154	1014	527–84-4	o-Cymene	C10H14	0.20 ± 0.01c	0.08 ± 0.01e	0.13 ± 0.01d	0.04 ± 0.00f	0.22 ± 0.01b	0.00 ± 0.00 g	0.26 ± 0.01a	MS, RI
155	1027	99–87-6	p-Cymene	C10H14	–	0.01 ± 0.00a	0.00 ± 0.00a	–	–	–	–	MS, RI
156	1056	105–05-5	Benzene, 1,4-diethyl-	C10H14	–	–	–	0.01 ± 0.00a	–	–	–	MS, RI
157	1085	1758-88-9	Benzene, 2-ethyl-1,4-dimethyl-	C10H14	0.01 ± 0.00a	–	–	–	–	–	–	MS, RI
158	1156	6931-54-0	β-Pinene oxide	C10H16O	–	–	–	–	–	0.03 ± 0.00a	–	MS, RI
159	1295	585–34-2	Phenol, m-tert-butyl-	C10H14O	–	–	–	0.02 ± 0.00a	–	–	–	MS, RI
160	1295	120–72-9	Indole	C8H7N	–	0.01 ± 0.00a	–	–	–	–	–	MS, RI
161	1296	89–83-8	Thymol	C10H14O	0.12 ± 0.01a	–	–	–	–	0.05 ± 0.00b	–	MS, RI
162	1328	717–74-8	Benzene, 1,3,5-tris(1-methylethyl)-	C15H24	–	–	–	–	–	0.08 ± 0.00a	–	MS, RI
163	1354	72,257–53-5	2-acetoxy-1,8-cineole	C12H20O3	–	–	–	0.05 ± 0.00d	0.23 ± 0.01b	0.24 ± 0.01a	0.21 ± 0.01c	MS, RI
164	1454	1560-95-8	Tetradecane, 2-methyl-	C15H32	0.03 ± 0.00a	0.01 ± 0.00c	0.02 ± 0.00b	–	–	–	–	MS, RI
165	1700	473–04-1	Juniper camphor	C15H26O	0.46 ± 0.03a	–	–	–	–	–	0.02 ± 0.00b	MS, RI
166	–	16,052–42-9	(−)-Menthyl chloride	C10H19Cl	–	–	–	–	0.04 ± 0.00a	–	–	MS
167	–	927–80-0	Ethoxyacetylene	C4H6O	–	–	–	0.67 ± 0.05b	–	0.71 ± 0.04a	–	MS
168	–	288–36-8	1H-1,2,3-Triazole	C2H3N3	–	–	–	–	–	0.19 ± 0.01b	0.94 ± 0.05a	MS
169	–	2562-38-1	Cyclopentane, nitro-	C5H9NO2	–	–	–	–	–	0.01 ± 0.00b	1.88 ± 0.10a	MS
170	–	54,060–30-9	*m*-Aminophenylacetylene	C8H7N	0.01 ± 0.00a	–	0.01 ± 0.00a	0.00 ± 0.00a	–	–	–	MS
171	–	464–15-3	Bicyclo[2.2.1]heptane, 1,7,7-trimethyl-	C10H18	–	–	–	0.01 ± 0.00a	–	–	–	MS
172	–	53,091–80-8	1H-Pyrazole, 5-methoxy-1,3-dimethyl-	C6H10N2O	–	–	–	–	–	–	0.04 ± 0.00a	MS
172	–	613–94-5	Benzoic acid, hydrazide	C7H8N2O	–	–	0.01 ± 0.00a	–	–	–	–	MS

Abbreviations: MS – mass spectrum; RI – retention indices;

[A] : Retention indices determined by GC–MS on stationary phases (HP-5).

[B] : Identification: RI – compound confirmed by retention index; MS: compound identified by comparison with the NIST14.L mass spectral database.

[C] : "-" means compound was not detected.

[D] : The values of relative percentage contents are shown as the mean ± standard error. In the same rows, different letters behind values represent they have significant difference ($p < 0.05$), number of experimental repetitions (n = 3).

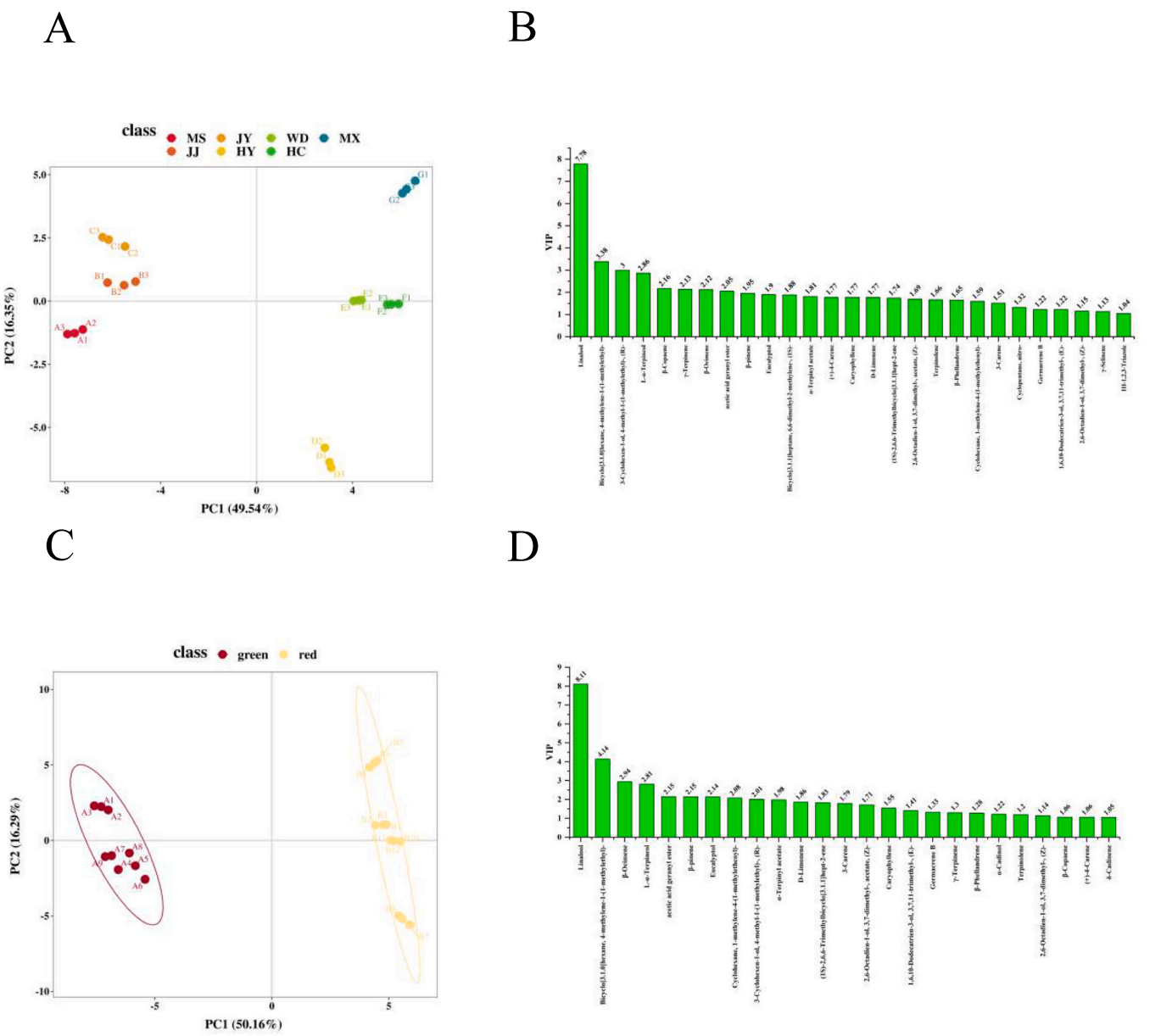

Fig. 3. The partial least squares analysis of volatile organic compounds in huajiao from different origins (A); variable importance in projection (VIP>1) scores of each variable in the analysis of huajiao from different origins (B); the partial least squares analysis of volatile organic compounds in green and red huajiao (C); variable importance in projection (VIP>1) scores of each variable from the analysis of green and red huajiao (D).

contents of linalool oxide, linalool, caryophyllene, α-pinene, camphene, citronellal, β-pinene, octanal, undecanal, sabinene, decanal, humulene, and bicyclogermacrene were relatively higher, whereas other components were more abundant in red DZM, reflecting a more pronounced fresh aroma in green DZM and a distinct spicy aroma in red DZM. The results of this study were consistent with the previous study results, which further supported the flavor differences between green DZM and red DZM, and clarifies the key flavor characteristics of green and red DZM (Yang, 2008).

In comparing the differences among DZM from various origins, the study selected potential marker compounds based on significant differences in the contents of key aroma components: DZM from MS, with potential markers linalool oxide, linalool, and caryophyllene; DZM from JJ with humulene, bicyclogermacrene; DZM from JY, with octanal and undecanal; DZM from HC, with nonanal, α-phellandrene, and linalyl acetate; DZM from HY, with β-cubebene and carvone; DZM from WD, with eucalyptol and β-ocimene; and DZM from MX, with β-phellandrene and 2-nonenal, (E). These components serve as indicative markers for distinguishing DZM from different origins.

4. Conclusion

This study comprehensively evaluated the flavor and quality of DZM. from seven major producing regions in China, shedding light on the influence of origin and processing methods. Sensory analysis revealed significant differences in aroma, pungency, color, and shape, with MX and JY samples scoring highly across all attributes. Color analysis demonstrated the impact of origin and processing, with MS green peppercorns exhibiting a bright, glossy green, and HC red peppercorns showing a bright red color. Physical and chemical analysis highlighted the need for standardized processing and cleaning practices. HPLC analysis identified MX and WD samples as containing the highest levels of pungent compounds. GC–MS analysis revealed a complex volatile oil profile with 173 compounds identified, with linalool and D-limonene

being the most abundant. PLS-DA analysis differentiated volatile oil compositions between green and red peppercorns and identified origin-specific markers, providing a valuable tool for origin identification and quality assessment. Microbial analysis indicated that HY and JJ samples had the lowest levels of bacterial and mold contamination, emphasizing the importance of proper processing and storage practices for ensuring food safety. Heavy metal analysis revealed that HY, HC, and JY samples contained the lowest levels of arsenic, lead, and cadmium, suggesting better soil and environmental conditions in these regions. The findings of this study provide valuable insights for producers and consumers in selecting the most suitable DZM varieties for specific applications, ensuring food safety, and guiding the development of standardized processing and storage methods to preserve the unique flavor and quality of this spice.

CRediT authorship contribution statement

Xiang Zhu: Writing – review & editing, Writing – original draft, Conceptualization. **Di Wu:** Resources, Methodology, Data curation. **Lin Zhao:** Investigation, Funding acquisition, Formal analysis. **Chenggang Wen:** Software, Project administration, Investigation, Funding acquisition. **Cao Yong:** Writing – review & editing, Writing – original draft, Formal analysis, Conceptualization. **Qixin Kan:** Writing – review & editing, Writing – original draft, Methodology, Formal analysis, Data curation, Conceptualization.

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.2024.102017.

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