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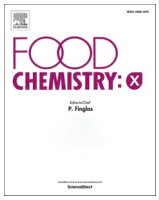

Insights into the key odorants in fresh and dried *Amomum tsaoko* using the sensomics approach

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ABSTRACT

To identify the key odorants in *Amomum tsaoko* (AT), volatiles in fresh AT (FAT) and dried AT (DAT) were investigated using molecular sensory science. In addition to this, the sensomics approach was used to confirm the presence of the compound in FAT that contributed the most to its aroma profile. A total of 49 odor-active compounds (43 in FAT and 42 in DAT) with flavor dilution (FD) factors ranging from 1 to 6561 were identified, with eucalyptol exhibiting the highest FD factor of 6561. Odorants with FD factors $\geq$ 27 were quantitated, and 23 and 20 compounds in FAT and DAT, respectively, with odor activity value $\geq$ 1 were determined as key odorants. Recombination and omission experiment further indicated that (*E*)-2-dodecenal, geranial, octanal, (*E*)-2-octenal, (*E*)-2-decenal, and eucalyptol contributed significantly to the overall aroma profile of FAT. After drying of FAT, the concentrations of aldehydes decreased significantly, whereas those of terpene hydrocarbons increased. Multivariate statistical analysis revealed that 26 FAT and 23 DAT odorants were biomarker compounds.

1. Introduction

Amomum tsaoko (AT), also called Chinese black cardamom, a perennial herb belonging to the Zingiberaceae family, is a cultivated medicinal and aromatic plant. Distinct from AT, green cardamom (*E. cardamomum*) and black cardamom (*A. subulatum*) are mainly found in India. While AT is found in the southwestern provinces of China, such as Yunnan and Guangxi, but also in the northern highlands of Vietnam and Laos (Sim, Tan, Kohlenberg, & Braun, 2019). Most of ATs grow at altitudes of 1000–2000 m on valley slopes with warm and humid climates (Yang, Xue, Chen, & Wang, 2022). Over the past decades, AT production has increased significantly in China. With a planting area of more than 78,050 acres, Nujiang Prefecture in Yunnan Province is the largest AT producer in China, and its annual output of fresh AT (FAT) has exceeded to 47,400 tons. As a spice with characteristic flavors, FAT has a short shelf life due to its high moisture content and susceptibility to deterioration. Currently, it is mainly used in jams, sauces or dips to accompany a variety of desserts and main dishes, or as a refreshing snack after marination to provide a refreshing taste and aroma. Dried AT (DAT) is widely used to recover the fishy smell of meat during cooking in Southeast Asia. AT is not only a perfect seasoning material, but also an excellent raw material for the formulation of Chinese patent medicine. AT has anti-inflammatory effects against insects (Dang, Aah, & Dat, 2020), antitumor effects in liver cancer cells (Zhang, Lu, & Jiang, 2015), and anti-angiogenesis efficacy in ovarian cancer (Chen, You, Wu, Luo, & Liu, 2020). Furthermore, AT can also inhibit sphingosine kinases 1 and 2 (Lee et al., 2019). Previous studies have shown that the essential oils of AT have bioactivities, including antimicrobial, antibacterial, insecticidal, antioxidant, etc. (Cui et al., 2017; Wang et al., 2014; Yang, Yang, Yan, & Zou, 2010). Recently, AT-containing prescriptions have been explored and used for treating hepatitis B, influenza, and coronavirus disease 2019 (COVID-19) (Yang et al., 2022).

Drying, which is a crucial step in the postharvest processing of spices, may change their flavor to some extent. Owing to its high moisture content and water activity, FAT is prone to rapid rotting and flavor loss; therefore, FAT is often dried to inhibit the growth of microorganisms and prolong its shelf life. Oven drying is the most commonly used method in the food industry mainly because of its ability to control the drying temperature and time (Thamkaew, Sjoholm, & Galindo, 2021). Qin et al. (2021) compared the effects of different drying methods on the quality and volatile oil content of AT; the results showed that AT dried in an oven contained a higher volatile oil content, did not cause mold or cracking, and required a shorter drying time, which is more conducive to the quality assurance of AT.

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To date, more than 600 volatile compounds have been detected. To isolate the volatiles in AT, some studies have used various extraction technologies such as steam distillation (Feng, Jiang, Wang, & Li, 2010), headspace solid-phase microextraction (Liang, Zhang, Wu, Wang, & Liu, 2023), and microwave hydrodistillation extraction (Yu et al., 2018). Wang et al. (2014) identified 43 components in AT by using hydrodistillation, of which eucalyptol, limonene, 2-isopropyltoluene, and undecane, were the major compounds, accounting for 23.87%, 22.77%, 6.66%, 5.74%, respectively. Sim et al. (2019) found that the main volatiles of AT were eucalyptol, geranial, geraniol, *trans*-2,3,3a,7a-tetrahydro-1*H*-indene-4-carbaldehyde, (*E*)-2-decenal, neral, and 4-indanecarbaldehyde after hydrodistillation. Using a combination of direct solvent extraction and solvent-assisted flavor evaporation (DSE-SAFE), a reliable flavor extract can be obtained that is almost the same as the original extract in the sensory sense (Xu, Fan, & Qian, 2007); however, this approach is rarely used for isolating volatiles in AT.

Most studies have only focused on the volatile components of AT, and volatiles have only been tentatively identified by mass spectrometry (MS) and retention index (RI). Odor is the most important index for evaluating the quality of AT; however, not all volatiles contribute to the overall aroma profile. To our knowledge, there are few reports on the odor-active components that contribute significantly to the odor profile of AT. The sensomics approach was first proposed by Peter Schieberle in 2006 to determine the key odorants in the overall aroma profile of food at the molecular level. This approach includes qualitative analysis of odor-active compounds, aroma extract dilution analysis (AEDA), quantitative analysis, calculation of odor activity value (OAV), aroma recombination, and omission experiments. At present, this approach is widely used in the aroma analysis of tea (Flaig, Qi, Wei, Yang, & Schieberle, 2020), spices (Dein & Munafo, 2022), wine (Marcq & Schieberle, 2021), and fruits (Tan, Wang, Zhan, & Tian, 2022). However, this method has yet not been used in characterizing the key odorants of FAT and DAT.

Currently, investigation on qualitative and quantitative analyses of the odor-active compounds of AT are insufficient. In addition, the key aroma components of AT have not been characterized, and a systematic comparison between the key odorants of FAT and DAT is lacking. Therefore, the present study aims at 1) comparing and identifying the volatiles present in FAT and DAT; 2) screening the odor-active components by gas chromatography–mass spectrometry combined with olfactometry (GC–MS–O); 3) performing AEDA and measuring flavor dilution (FD) factors to determine the important odorants; 4) quantitating important odorants in FAT and DAT and calculating their OAVs to find the key odorants; 5) preparing the odor simulation model to verify the key odorants identified in FAT; 6) determining the key odorants with the greatest impact on the overall aroma of FAT using omission experiments; and 7) establishing the correlation between the samples, key odorants, and sensory attributes using multivariate statistical analysis. The results obtained in this study could provide a scientifically sound basis for better exploitation of AT by the spice industry and offer new ideas for developing a series of AT derivative products.

2. Materials and methods

2.1. Materials

FATs used in this experiment were harvested in December 2021 from Yunnan Province, Gongshan County, China. Approximately 200 g of intact FAT sample were selected randomly, wiped off the surface dust and dried directly in an oven at 60 °C for 24 h to reach a constant weight, in triplicate. Complete dry samples of FAT are referred to as DAT. All samples (FAT and DAT) were stored at −40 °C before analysis.

2.2. Chemicals

The number of compounds is consistent with that in Supplementary Table 1. Compounds **1** (98%), **3** (≥ 95%), **4** (≥ 75%), **6** (80%), **10** (95%), **17** (96%), **20** (97%), **21** (99%), **25** (96%), **28** (95%), **61** (97%), **65** (99%), and **66** (96%) were gotten from Macklin Biochemical Co., Ltd., Shanghai, China. Compounds **7** (95%), **30** (95%), **36** (98%), **41** (98%), **59** (98%), **72** (98%), 2-octanol (98%), 2-isopropylphenol (98%), and citral (≥ 98%) were obtained from J&K Chemicals Ltd., Beijing, China. Compounds **22** (≥ 95%), **31** (> 90%), **46** (95%), **48** (90%) and **60** (95%) were purchased from TCI Chemical Ltd., shanghai, China. Compounds **5** (≥ 90%), **11** (98%), **35** (95%), **62** (90%), **67** (95%), and **70** (96%) were purchased from Aladdin Reagents Co., Ltd., Shanghai, China. Compounds **23** (95%), **32** (≥ 99%), **43** (98%), **45** (97%), **49** (92%), **50** (≥ 98%), **53** (98%), and **68** (98%) were obtained from Yuanye Bio-Technology Co., Ltd., Shanghai, China. C6–C28 *n*-alkanes were bought from Aldrich Chemical Co., Ltd., Shanghai, China, and dichloromethane, propylene glycol, anhydrous sodium sulfate, and sodium chloride were obtained from Sinopharm Chemical Reagent Co., Ltd.

2.3. Assessment of overall aroma profile of FAT and DAT

Quantitative descriptive analysis (QDA) is a sensory evaluation method in which assessors quantitatively describe and analyze the odor characteristics of products. Twelve trained panelists (five males and seven females) of ages between 23 and 53 years were recruited to evaluate the aroma of the AT samples in an odorless room under room temperature (25 °C ± 2 °C). First, they were asked to write down the odor descriptors they perceived after preliminary sniffing. Secondly, after discussion, seven odor descriptors with a higher frequency of occurrence, including citrus, herbaceous, fatty, spicy, woody, sweet, and floral were chosen to describe the overall aroma of FAT and DAT; seven attributes were defined as references to the following aromas: citral for "citrus" attribute, eucalyptol for "herbaceous" attribute, (*E*)-2-decenal for "fatty" attribute, geraniol for "floral" attribute, vanillin for "sweet" attribute, star anise for "spicy" attribute, and α-pinene for "woody" attribute. The concentrations of the aroma references were 100 times their respective odor thresholds in water. Finally, the coded AT samples were ranked randomly and evaluated by the sensory panelists using a ten-point interval scale (0–3 = weak, 4–6 = significant, 7–9 = strong). The average results were used to construct a spiderweb diagram.

2.4. Isolation of volatiles from AT using DSE-SAFE

The samples were frozen in liquid nitrogen for 180 s and then crushed in a pulverizer for 60s. For each trial, accurately 5.0 g of FAT (or 1.0 g of DAT) fine powder containing 100 μL each of internal standard 0.01 g/mL 2-octanol and 0.001 g/mL 2-isopropylphenol were extracted with dichloromethane (60 mL × 3) in a constant temperature incubator Shaker incubator shaker for 1 h at 25 °C and a speed of 200 rpm (Wu et al., 2023). The volatiles were isolated from the extract obtained through SAFE at a pressure of 2.5×10^{-5} mbar (Edwards TIC Pumping Station from BOC Edwards, England). The distillate obtained using SAFE was dehydrated over anhydrous Na_2SO_4 overnight and concentrated to approximately 5 mL via a glass Vigreux column (length = 50 cm, internal diameter = 2 cm) (Beijing Jing Shen Glassware Co., Ltd., Beijing, China) at 45 °C. In addition, the distillate was concentrated to approximately 1 mL using a nitrogen purge. The concentrated fraction was stored in a refrigerator at a temperature of −40 °C prior to GC–MS–O analysis.

2.5. GC–MS–O analysis

A gas chromatograph (Agilent model 7890B) equipped DB-WAX or HP-5MS capillary columns (30 m × 0.25 mm, 0.25 μm) was coupled with an Agilent 5975 mass spectrometer detector to isolate the volatiles. Helium (purity ≥99.999% purity) was used as the carrier gas with a flow rate of 1.7 mL/min. Splitless injection mode was adopted when inserting volatiles at 240 °C, and the injection volume was 1.0 μL. The oven

temperature and instrument parameters were programmed as described by Liang et al. (2023) with little modification. Using a Y-tape splitter, the GC effluent was split between the MS and sniffing port (ODP3; Gerstel GmbH & Co. KG) at a volume ratio of 1: 2. The sniffing port temperature was set at 120 °C. Three trained panelists recorded the odor characteristics and retention times of the odorants sniffed from the olfactory detector.

2.6. Aroma extract dilution analysis (AEDA)

Important odorants among the identified odor-active compounds were screened using the AEDA. Three trained panelists measured the FD factor for each odorant by sniffing the FAT and DAT SAFE distillates, which were diluted stepwise with dichloromethane in increments of 3^n (where $n = 0, 1, 2, 3\ldots$). One microliter of the diluted FAT or DAT isolate was placed on a chromatographic column in the splitless mode. The FD factor was defined as the highest dilution of the isolate in which the odorant could be perceived by at least two panelists.

2.7. Qualitative and quantitative analyses

The volatiles detected in AT were characterized positively by comparing their MS data, odor quality, and RI on the two columns (DB-WAX and HP-5MS) with those of authentic compounds, and RI was calculated for each volatile compound using the retention times of a homologous series of C6–C28 n-alkanes. If reference odorants were unavailable, the extracted volatiles were tentatively characterized by matching their mass fragmentation patterns with the MS library (NIST 2014) and comparing their RIs on the two columns. Aroma-active compounds were identified by comparing their odor quality on the website (http://www.thegoodscentscompany.com).

To compare the volatile contents of FAT and DAT, the volatiles were quantitated by the internal standard (IS) method. The relative concentration of each compound in FAT and DAT was calculated according to the following equation.

$$C_r = \frac{A_r \times C_{is}}{A_{is}} \quad (1)$$

Where C_r is the relative concentration of a volatile compound, A_r is the peak area of a volatile compound, C_{is} is the IS concentration, and A_{is} is the peak area of the IS. For comparison, the concentrations of the compounds in the dried sample were adjusted to match those in the fresh sample.

Compounds with FD factors > 27 were quantitatively analyzed by establishing internal standard curves, each calibration curve consisting of seven solutions of different concentrations, and the concentration gradients were formulated as follows: analyte concentration/internal standard concentration: 1:10, 1:5, 1:3, 1:1, 3:1, 5:1, 10:1. The calibration curves were obtained by plotting the analyte: IS concentration ratio (x) against the ratio of the peak areas of the authentic odorant and IS (y). The slopes of the calibration curves represented the response factors (R_f). Finally, the odorant concentrations were calculated using Eq. (2).

$$C = \frac{C_r}{R_f} \quad (2)$$

where C is the analyte concentration in mg/kg AT, C_r is the relative concentration of volatiles, and R_f is the response factor.

2.8. Determination of odor-activity values (OAVs)

Most odor thresholds were obtained from the literature (Gemert, 2011). The odor thresholds for odorants **11** and **43** were measured according to the method described by Czerny et al. (2008) with a minor modification. The odorant was diluted to a concentration where no odor could be smelled to obtain the estimated orthonasal detection threshold (EODT), and a series of solutions with concentrations ranging from the lower 50-fold EODT to the higher 50-fold EODT were prepared in equal proportions. The panelists evaluated these solutions using a series of triangle tests, and the odorant threshold was calculated according to the following equations:

$$OT_i = \sqrt{C_m \times C_{m+1}} \quad (3)$$

$$OT_p = \sqrt[n]{\prod_{n=1}^{n} OT_i} \quad (4)$$

where OT_i and OT_p represent the odor detection thresholds of each assessor and panelist, respectively, C_m and C_{m+1} are the lowest and highest concentrations of the odorants that were correctly and incorrectly selected by the panelist in a series, respectively, and n is the number of panelists.

The OAV is obtained by dividing the concentration of the odorant by its threshold value.

2.9. Recombination and omission experiments

The recombination model was prepared by mixing the odorants with OAV ≥ 1 at concentrations quantitated in FAT. The mixture was shaken evenly before evaluation. Twelve panelists were asked to compare the overall odor profiles of the recombination model and the FAT sample based on seven attributes using the QDA (0–3 = weak, 4–6 = significant, 7–9 = strong).

The omission model was designed to determine the contribution of single or grouped compounds in FAT to the overall odor profile using triangulation tests. The omission model was obtained by eliminating a key odorant or a group of key odorants and was compared with the complete recombinant models. All test samples were coded with three or four random numbers, and the panelists were asked to select one bottle out of the group that had a different odor characteristic from the other two after the sensory evaluation.

2.10. Statistical analysis

The mean and analysis of variance (ANOVA) of the volatiles were calculated by Microsoft Excel 2021. Origin 2021b (version 2021; Origin Lab Inc., Northampton, MA, USA) was used as a statistical analysis and graphing software. Heat maps were generated using the TB tools. One-way ANOVA and Duncan's multiple-range tests were conducted using the IBM SPSS Statistics 24 software, and $p \leq 0.05$ was considered statistically significant. Principal component analysis (PCA) and orthogonal partial least squares discrimination analysis (OPLS-DA) were performed using SIMCA14.1.

3. Result and discussion

3.1. Differences in the overall aroma profiles of FAT and DAT

The SAFE isolates and AT samples had the same odor characteristics as those agreed upon by the sensory panelists, proving that the aroma components in FAT and DAT were thoroughly extracted through DSE-SAFE.

The FAT and DAT scores were determined through the sensory evaluation of the overall aroma by the trained evaluators (Fig. 1a). The results indicated that the dominant aroma attributes of FAT and DAT were citrus, herbaceous, and fatty, and the citrus note was the strongest among the seven attributes, whereas the woody, floral, and sweet notes had lower scores. After drying, the intensities of the citrus, herbaceous, floral, and sweet attributes slightly decreased. However, the intensities of spicy and fatty attributes increased significantly ($p \leq 0.05$), while that of woody attributes increased slightly ($p \geq 0.05$). A previous study confirmed that heating can weaken the citrus and herbaceous notes (Li

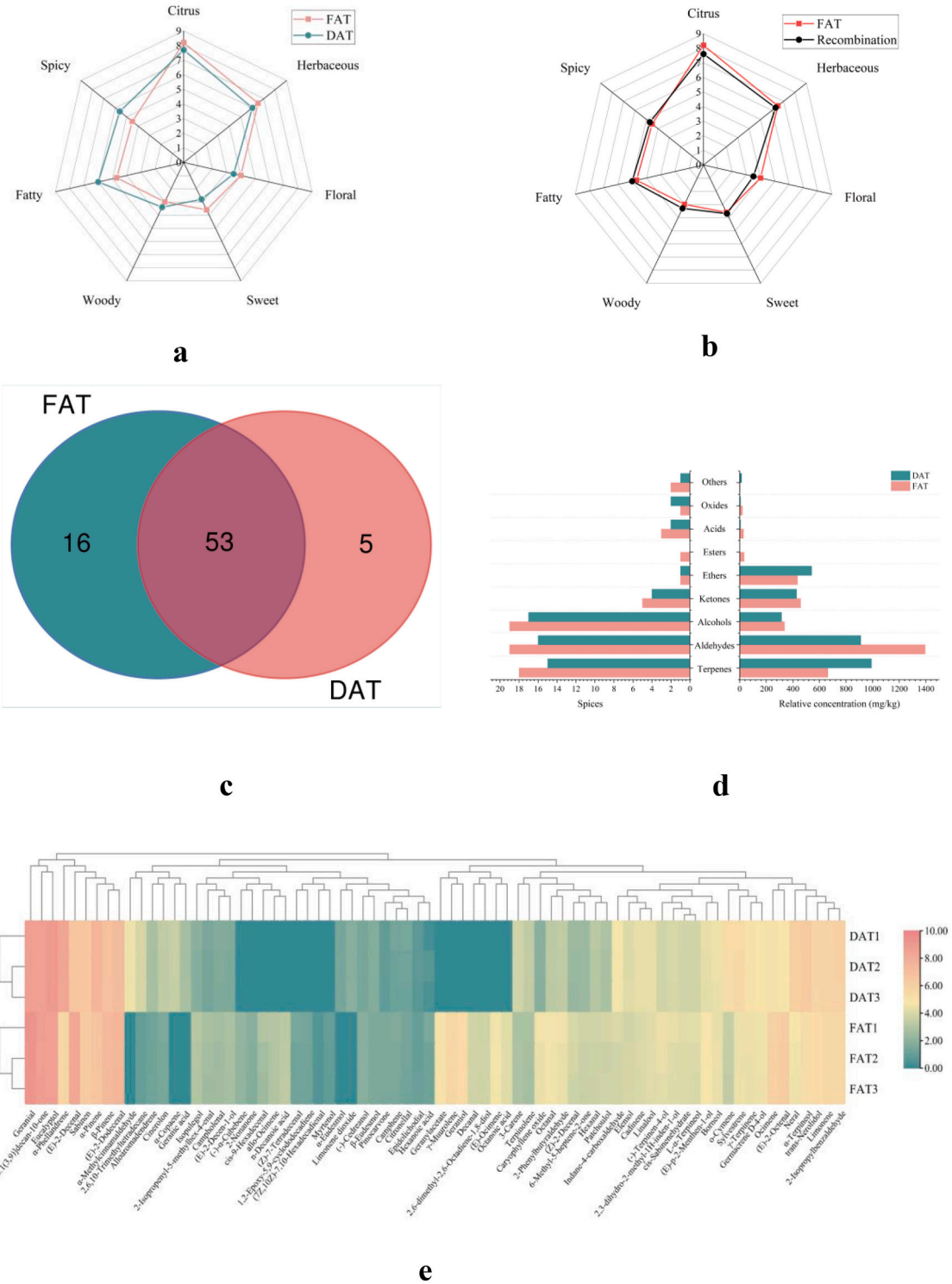


Fig. 1. Aroma profiles of FAT and DAT (a); aroma profiles of FAT and recombinant (b); Venn diagram of volatile compounds in FAT and DAT (c); accumulation diagram of volatile compounds in FAT and DAT (d); heatmap of volatile compounds identified in FAT and DAT (e).

et al., 2022). Based on the above results, it was speculated that the change in aroma intensities in AT might be related to changes in the concentrations of the corresponding compounds (Suharta, Hunaefi, & Wijaya, 2021). Differences in the aroma characteristics of FAT and DAT are helpful in determining their application value in the food industry. Because of its relatively balanced and rich aroma, FAT has been used as a material for manufacturing food and cosmetics such as wine, jam, *tsaoko* Zhengqi tea, perfume, and masks (Liang et al., 2023). On the other hand, DAT is often used as a spice owing to its strong fatty and spicy aroma attributes.

3.2. *Effects of drying on the composition of volatiles in AT*

Chemical reactions triggered either by enzymes or chemicals, including oxidation, isomerization, cyclization, and dehydrogenation, induce changes in volatiles of AT (Turek & Stintzing, 2013). A total of 74

volatiles in FAT and DAT were identified using a combination of DSE-SAFE and GC–MS–O on both polar (DB-WAX) and non-polar (HP-5MS) capillary columns. As shown in Fig. 1c, 69 and 58 compounds were identified in FAT and DAT, respectively. Of these, 53 volatiles were common to both FAT and DAT, including 14 terpene hydrocarbons, 15 aldehydes, 16 alcohols, four ketones, one ether, one acid, one oxide, and one other compound (Table S1).

After drying, the number of volatiles in AT decreased; however, terpene hydrocarbons, aldehydes, and alcohols were the predominant compounds in both FAT and DAT (Fig. 1d). Converting FAT to DAT reduced the number of volatile compounds, including terpene hydrocarbons, aldehydes, alcohols, ketones, acids, and other compounds, to some extent. However, six unique constituents, including α-copaene (**16**), α-methyl cinnamaldehyde (**36**), α-eudesmol (**58**), geranylacetate (**66**), geranic acid (**70**), and limonene dioxide (**71**) were formed as a result of this conversion. This might be since some compounds have decomposed or interacted with other volatiles to produce new components in the process of heating (Li et al., 2022).

The loss or increase in the concentration of volatiles during drying is due to oxidation, esterification, glycoside hydrolysis, and other processes (Ghasemi Pirbalouti, Mahdad, & Craker, 2013). After drying, the total concentration of volatiles decreased, particularly that of aldehydes, whereas those of terpene hydrocarbons, ether, and other compounds increased. Even if the species of terpene hydrocarbons reduced after drying, the total relative content of the terpene hydrocarbons increased notably from 663.06 mg/kg to 991.46 mg/kg, accounting for 19.67% and 30.88% of FAT and DAT, respectively. This effect of drying on the concentrations of terpene hydrocarbon is in line with the results of Qin et al. (2021). Notably, Fig. 1e exhibited that the terpene hydrocarbons whose concentrations increased significantly were α-pinene (**1**) and α-phellandrene (**6**). One possible explanation for this phenomenon is that the heating process may cause the conversion of other terpene hydrocarbons. Another possible reason for the release of previously bound volatiles during drying. This is because volatile compounds may exist in free forms, or they can bound to other molecules such as sugar-forming glycosides as they are water-soluble and can accumulate in plant tissues (Sharma, Chatterjee, Kumar, Variyar, & Sharma, 2010; Thamkaew et al., 2021). The total relative content of aldehydes reduced from 1395.48 mg/kg to 911.20 mg/kg due to drying. In FAT, the level of aldehydes was the highest (41.39%), mainly due to the relatively high concentration of geranial (**30**) and unsaturated fatty aldehydes, including (*E*)-2-octenal (**22**), (*E*)-2-decenal (**28**), and (*E*)-2-dodecenal (**31**). However, the concentrations of all aldehydes decreased after drying, except for neral (**29**), 2-isopropylbenzaldehyde (**32**), and indane-4-carboxaldehyde (**35**); consequently, the relative content of aldehydes accounted for only 28.38% in DAT. Alcohol was less affected by drying, which may be due to the higher boiling points of alcohol compounds in FAT and DAT. Although ketones in FAT were few, their total content was higher than that of alcohols, mainly because of the relatively high concentration of tetracyclo[3.3.1.0.1(3,9)]decan-10-one (**63**), which has also been detected in linseed seeds (Sinha, Kumar, & Singh, 2013). Eucalyptol (**65**), with antibacterial effects, was supposed to be the main compound in AT by researchers (Wang et al., 2014; Wang et al., 2021).

3.3. Differentiating FAT and DAT based on odor-active compounds

Odor-active compounds contribute to overall odor quality. As summarized in Table 1, 49 odor-active compounds were determined (43 in FAT and 42 in DAT). Multivariate statistical analyses (PCA and OPLS-DA) were used for classification purposes and biomarker selection; therefore, PCA and OPLS-DA analyses were performed on 49 odorants to investigate the effect of drying on odor-active components by reducing the dimension. As illustrated in Fig. 2a and b, PCA was employed to evaluate the stability and consistency of the odor-active compounds in FAT and DAT, and the cumulative contribution of the first two principal

Table 1
Odor-active compounds determined by GC–MS–O in both FAT and DAT.

Nos.	Compounds	RI		Odor quality[a]	FD[b]	
		DB-WAX	HP-5MS		FAT	DAT
	Terpenes (11)					
1	α-Pinene	1027	932	pine, earthy	243	243
3	β-Pinene	1106	977	pine, woody	81	27
4	Sabinen	1115	973	woody, spicy, camphor	3	9
5	3-Carene	1142	1016	citrus, pine, herbal	/[c]	1
6	α-Phellandrene	1151	/	citrus, herbal	3	27
7	Limonene	1191	1030	citrus, herbal, terpene	27	27
11	Ocimene	1242	1048	citrus, green	3	3
14	allo-Ocimene	1358	/	green, floral, citrus	9	3
16	α-Copaene	1484	/	woody	/	3
17	Alloaromadendrene	1657	/	orange-like	/	1
19	Cadinene	1751	/	green, woody	27	27
	Aldehydes (14)					
20	Hexanal	1077	802	aldehydic, grass	1	1
21	Octanal	1278	1005	aldehydic, herbal, orange peel	81	27
22	(*E*)-2-Octenal	1421	1060	fatty, grass, nutty	729	729
23	Citronellal	1463	1152	floral, green	1	1
25	Decanal	1483	1203	soap, orange peel, fatty	27	/
27	(*Z*)-2-Decenal	1598	/	fatty, tallow	81	9
28	(*E*)-2-Decenal	1651	1267	fatty, waxy, earthy	729	243
29	Neral	1674	1243	citrus, lemon	9	27
30	Geranial	1735	1285	citrus, lemon, sweet	243	243
31	(*E*)-2-Dodecenal	1864	1303	citrus, waxy, fatty	2187	729
32	2-Isopropylbenzaldehyde	1873	1470	fatty, green	27	9
33	2-Phenylbutyraldehyde	1906	1316	smoky, roast-like	81	27
34	*cis*-9-Hexadecenal	1956	/	burned-like, almond	1	/
35	Indane-4-carboxaldehyde	1978	/	caramel-like, smoky	9	81
	Alcohols (15)					
41	Linalool	1528	1098	floral, pepper	81	243
42	(*E*)-p-2-Menthen-1-ol	1535	1123	mint, green	81	3
43	(−−)-Terpinen-4-ol	1590	1180	woody	3	3
44	L-α-Terpineol	1663	1169	lilac, floral, terpenic	3	3
45	α-Terpineol	1688	1194	woody, pine	3	3
46	Borneol	1693	/	earthy, mouldy	/	1
48	Isopulegol	1787	/	minty, cooling, woody	1	/
50	Geraniol	1837	/	rose, geranium	2187	/
51	Germacrene D-4-ol	1932	/	almond	81	81
52	2,3-Dihydro-2-methyl-1*H*-inden-1-ol	2007	1352	bitter	1	/

(*continued on next page*)

Table 1 (*continued*)

Nos.	Compounds	RI		Odor quality[a]	FD[b]	
		DB-WAX	HP-5MS		FAT	DAT
53	*trans*-Nerolidol	2023	/	floral, green, citrus	81	243
54	Elemol	2069	/	green, spicy, rose	9	9
56	Patchoulol	2150	/	patchouli, earthy, camphor	1	1
57	(−)-Cedreanol	2191	/	herbal, honey	1	/
59	*β*-Eudesmol	2228	/	woody, green	1	1
	Ketones (5)					
60	6-Methyl-5-heptene-2-one	1324	986	floral, citrus, green	3	/
61	2-Nonanone	1375	/	green, floral	81	9
62	Pinocarvone	1560	1166	minty	/	1
63	Tetracyclo[3.3.1.0.1 (3,9)]decan-10-one	1849	/	floral	6561	243
64	Cinerolone	2603	1645	fruity, earthy	1	3
	Ether (1)					
65	Eucalyptol	1219	1039	eucalyptus-like	6561	6561
	Acids (3)					
67	Hexanoic acid	1803		sour, fatty, sweat	1	/
68	Octanoic acid	2041	1569	fatty, waxy	1	/
70	Geranic acid	2258	/	dry, acidic	/	3

[a] Odor quality perceived at the sniffing port.
[b] Flavor dilution factor determined by AEDA on capillary DB-WAX.
[c] Not detected at sniff port.

variables were 0.979. Based on the biplot analysis, the odor-active components associated with FAT were three terpene hydrocarbons (**3**, **11**, and **14**), 10 aldehydes (**20**, **21**, **22**, **25**, **27**, **28**, **30**, **31**, **33**, and **34**), seven alcohols (**42**, **43**, **44**, **48**, **52**, **56**, and **57**), four ketones (**60**, **61**, **62**, and **63**), two acids (**67** and **68**). These odorants primarily presented "green", "citrus/lemon-like", "fatty", "sweet", "floral", "minty", and "herbal-like" aroma characteristics and contributed to the herbaceous, fatty, citrus, floral, and sweet flavors of FAT. The odor-active components facilitating the generation of the unique aroma of DAT were seven terpene hydrocarbons (**1**, **4**, **5**, **6**, **7**, **16**, **17**, and **19**), three aldehydes (**29**, **32,** and **35**), seven alcohols (**41**, **45**, **46**, **51**, **53**, **54**, and **59**), one ketone (**64**), one ether (**65**), and one acid (**70**). These compounds mainly provided the "pine", "woody", "herbal-like", "citrus/orange-like", "pepper-like", "floral", "spicy", "earthy", and "eucalyptus-like" scents to DAT, corresponding to the woody, spicy, fatty, citrus, and herbaceous attributes. Consequently, the PCA results agreed with the aroma profiles of FAT and DAT.

The OPLS-DA was used to investigate the influence of drying on the odor-active compounds of FAT. The Variable Importance for the Projection (VIP) plot was acquired according to the OPLS-DA model, and variable X was markedly different between the groups when the VIP value >1. As shown in Fig. 2c, 39 odor-active compounds labeled with orange color, including *α*-copaene, *α*-terpineol, *α*-phellandrene, octanoic acid, decanal et al. were the main compounds in charge of the differences in the odor profiles of FAT and DAT. These odorants included 10 terpene hydrocarbons (**16**, **6**, **1**, **14**, **11**, **5**, **3**, **17**, **4**, and **7**), 13 aldehydes (**25**, **30**, **28**, **21**, **31**, **22**, **29**, **32**, **33**, **35**, **27**, **20**, and **34**), eight alcohols (**45**, **51**, **57**, **53**, **48**, **42**, **56**, and **59**), five ketones (**64**, **61**, **62**, **60**, and **63**), one ether (**65**), and two acids (**68** and **70**) (ranked according to their VIP values). Therefore, these compounds were named as differential odorants. This result also indicated that the difference in aroma profiles between FAT and DAT mainly originates from the distinct levels of aldehydes and terpene hydrocarbons. In this study, FAT and DAT were discriminated based on the content of the odor-active compounds. However, 10 compounds that were marked in green, including **19**, **30**, **43**, **67**, **41**, **44**, **54**, **52**, **46**, and **23**, had VIP values < 1, indicating that these odorants did not undergo significant changes before and after drying of AT.

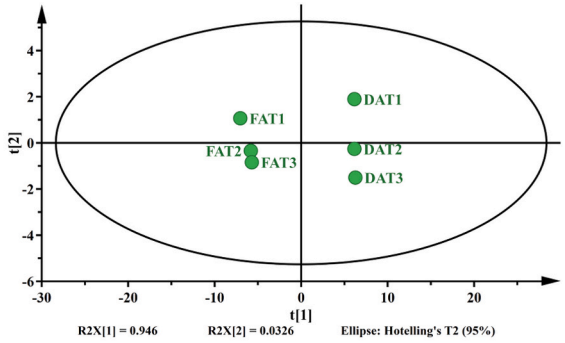
3.4. Aroma extract dilution analysis

Using AEDA to investigate the impact of drying on the odor-active compounds of AT, three experienced panelists perform GC–MS–O analysis of SAFE distillates on DB-WAX capillary, and the results were averaged (Table 1). The most intensive odorants were eucalyptol (FD = 6561 in FAT/6561 in DAT, eucalyptus-like), tetracyclo[3.3.1.0.1(3,9)] decan-10-one (FD = 6561/243, floral), (*E*)-2-dodecenal (FD = 2187/729, citrus, waxy, fatty), geraniol (FD = 2187/0, rose, geranium), followed by (*E*)-2-octenal (FD = 729/729, fatty, grass, nutty), (*E*)-2-decenal (FD = 729/243, fatty, waxy, earthy), *α*-pinene (FD = 243/243, pine, earthy), geranial ((FD = 243/243, citrus, lemon, sweet), linalool (FD = 81/243, floral, pepper), and *trans*-nerolidol (FD = 81/243, floral, green, citrus). To our best knowledge, the aroma of tetracyclo [3.3.1.0.1 (3, 9)] decan-10-one was detected for the first time.

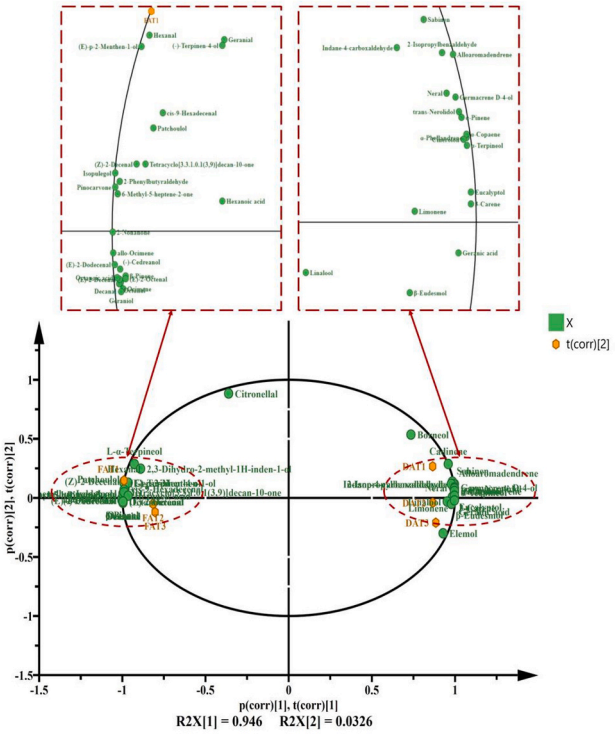
The FD factors of sabinene (woody, spicy, camphor), *α*-phellandrene (citrus, herbal), neral (citrus, lemon), indane-4-carboxaldehyde (caramel-like, smoky), linalool (floral, pepper), and *trans*-nerolidol (floral, green, citrus) increased after drying. This implied that drying could significantly strengthen the spicy, citrus, and woody attributes. The FD factors of odorants, including *β*-pinene (pine, woody), allo-ocimene (green, floral, citrus), octanal (aldehydic, herbal, orange peel), (*Z*)-2-decenal (fatty, tallow), (*E*)-2-decenal (fatty, waxy, earthy), (*E*)-2-dodecenal (citrus, waxy, fatty), 2-isopropylbenzaldehyde (fatty, green), 2-phenylbutyraldehyde (smoky, roast-like), (*E*)-*p*-2-menthen-1-ol (mint, green), 2-nonanone (green, floral), and tetracyclo[3.3.1.0.1 (3,9)]decan-10-one (floral), decreased after drying. This could be associated with the loss of odorants with water evaporation during the drying process. The FD factors of *α*-pinene, limonene, ocimene, cadinene, (*E*)-2-octenal, geranial, (−)-terpinen-4-ol, L-*α*-terpineol, *α*-terpineol, germacrene D-4-ol, elemol, patchoulol, and eucalyptol were unchanged. This meant that heating had less effect on these odor-active compounds, and they mainly contributed pleasant odor to FAT and DAT, such as floral, fruity, and green notes. Notably, decanal (FD = 27, soap, orange peel, fatty), geraniol (FD = 2187, rose, geranium), and 6-methyl-5-heptene-2-one (FD = 3, floral, citrus, green), which contributed richer aroma to FAT, were not perceived after drying.

3.5. Quantitation and odor activity value calculation

The important odorants of FAT and DAT with FD ≥ 27 were quantitated by IS curves, and their OAVs were calculated. Odorants with their OAVs >1, as listed in Table 2, were identified as key odorants. Geranial had the highest content (1846.73 mg/kg) in FAT, which decreased to 1227.69 mg/kg after drying. Geranial and neral are known as the *trans* and *cis* isomers of citral, respectively. Upon drying, the level of geranial declined, while that of neral increased. This tendency was also observed by Wang et al. during FAT drying (Wang et al., 2021). In addition, previous researches have demonstrated that this isomerization happens during the drying (Suharta et al., 2021), and that amino acids can catalyze this isomerization (Wolken, ten Have, & van Der Werf, 2000). The content of eucalyptol in AT increased from 1146.48 mg/kg to 1428.34 mg/kg after heating. Wang et al. suggested that the stress response of the fruit might have occurred during the heating process, which activated the synthesis pathway of eucalyptol and led to its accumulation (Wang et al., 2021). The concentrations of other odorants, including linalool, *α*-pinene, *α*-phellandrene, sabinene, neral, *trans*-nerolidol, limonene, elemol, and *α*-terpineol, also increased after drying. Two hypotheses had been proposed as the possible reason for this



a



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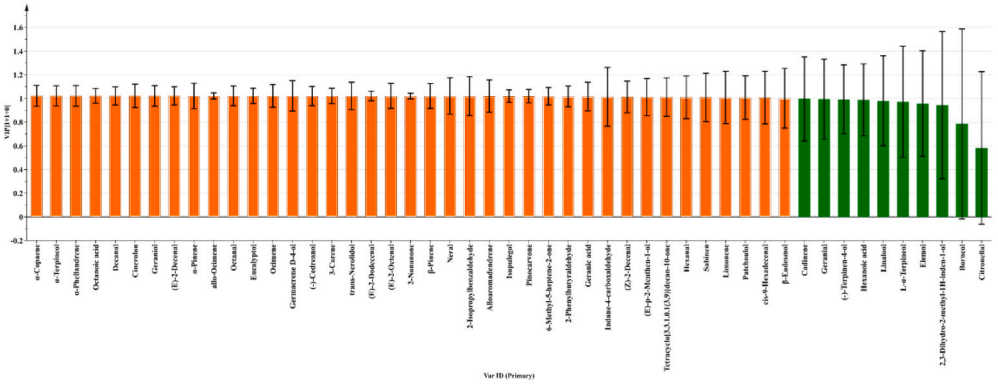

c

Fig. 2. PCA score plot (a), biplot (b), and VIP analysis (c) of FAT and DAT.

Table 2
Concentrations, odor thresholds, and odor activity values of odorants in FAT and DAT.

Nos.	Compounds	Quantification ions[a]	Calibration equations	R^2	OT (mg/kg)[b]	Concentration (mg/kg)		OAV[c]	
						FAT	DAT	FAT	DAT
31	(*E*)-2-Dodecenal	70	y = 0.2675x	0.9968	0.0014	677.83 ± 11.25	530.39 ± 1.83	484,166	378,852
22	(*E*)-2-Octenal	70	y = 0.1613x	0.9973	0.003	447.61 ± 4.28	170.86 ± 4.34	149,204	56,954
30	Geranial	69	y = 0.3503x	0.997	0.032	1846.73 ± 149.73	1227.69 ± 21.81	57,710	38,365
21	Octanal	84	y = 0.0988x	0.9998	0.0069	241.80 ± 1.72	85.12 ± 1.11	35,044	12,336
65	Eucalyptol	108	y = 0.3790x	0.9978	0.064	1146.68 ± 5.41	1428.34 ± 2.69	17,917	22,318
28	(*E*)-2-Decenal	98	y = 0.3110x	0.9985	0.077	981.29 ± 3.18	320.51 ± 3.18	12,744	4163
41	Linalool	93	y = 0.3118x	0.9995	0.006	55.42 ± 1.73	63.37 ± 1.28	9237	10,562
50	Geraniol	69	y = 1.4029x	0.9949	0.0066	29.99 ± 0.34	–	4544	–
1	*α*-Pinene	93	y = 0.9080x	0.9963	0.041	113.08 ± 0.80	202.97 ± 2.92	2758	4951
6	*α*-Phellandrene	93	y = 0.6357x	0.9996	0.04	77.87 ± 3.98	519.71 ± 9.83	1947	12,993
3	*β*-Pinene	93	y = 0.7728x	0.9946	0.14	196.45 ± 1.42	155.43 ± 1.00	1403	1110
5	Sabinen	69	y = 0.0747x	0.9996	0.98	1102.41 ± 29.05	1213.52 ± 18.34	1125	1238
27	(*Z*)-2-Decenal	83	y = 0.3110x	0.9985	0.05	44.31 ± 3.79	13.09 ± 0.58	886	262
29	Neral	69	y = 0.6551x	0.997	0.053	36.47 ± 2.89	100.72 ± 1.30	688	1900
53	*trans*-Nerolidol	93	y = 0.2490x	0.9956	0.25	166.02 ± 2.17	215.94 ± 2.17	664	864
7	Limonene	93	y = 0.4109x	0.9949	0.2	109.91 ± 3.82	141.59 ± 3.38	550	708
25	Decanal	161	y = 0.4945x	0.9953	0.063	25.06 ± 1.01	–	398	–
11	Ocimene	93	y = 0.7346x	0.9998	0.27	84.99 ± 0.64	43.62 ± 0.26	315	162
54	Elemol	161	y = 1.2786x	0.9974	0.068	10.98 ± 0.49	13.17 ± 0.75	161	194
45	*α*-Terpineol	121	y = 0.4209x	0.9976	1.2	92.33 ± 2.04	194.42 ± 1.71	77	162
43	(−)-Terpinen-4-ol	154	y = 0.4209x	0.9976	0.696	45.21 ± 6.25	31.08 ± 0.45	65	45
61	2-Nonanone	142	y = 3.1335x	0.9957	0.082	0.90 ± 0.19	–	11	–
44	L-*α*-Terpineol	59	y = 0.4209x	0.9976	9.18	37.68 ± 4.42	31.03 ± 1.14	4	3

[a] The ions selected for quantitative analysis.
[b] Odor detection threshold in water according to reference (van Gemert, 2011), and the OT of compounds **11** and **43** were determined by triangle tests according to Czerny et al. (2008).
[c] Odor activity value (ratio of the concentration to the odor threshold).

drying-induced increase in their concentrations. First, the cells and organelles containing volatiles were damaged during drying, thus increasing the liberation of the volatiles (de Torres, Díaz-Maroto, Hermosín-Gutiérrez, & Pérez-Coello, 2010); Second, drying could release the glycosidically bound volatiles by cracking the glycosidic bounds, possibly resulting in a better release of these terpenoids (Jonas & Schieberle, 2021). Generally, glucosides are hydrolyzed by enzymes, acids, and heat (Dziadas & Jeleń, 2016; Sarry & Günata, 2004). Therefore, it was postulated that the native enzymes within AT hydrolyzed the glycosidically bound volatiles. Fatty aldehydes, including (*E*)-2-octenal, (*E*)-2-dodecenal, octanal, (*E*)-2-decenal, (*Z*)-2-decenal, and decenal, had higher concentrations in FAT, which were reduced upon drying. This observation is in line with Jonas et al., who concluded that aldehydes were unstable even in aqueous solutions (Jonas et al., 2021). Geraniol (29.99 mg/kg in FAT) was completely lost during drying.

OAVs of odorants were measured, ranked, and used to describe the contribution of odors to the DAT and FAT. Twenty-three odorants with OAV > 1 (Table 2), and these odorants were considered key odorants. Aldehydes were the major contributors to the citrus and fatty aromas of AT owing to their low olfactory threshold and unique odor characteristics. The highest OAVs were calculated for (*E*)-2-dodecenal with 484,166 and 378,852 owing to its low odor threshold in FAT and DAT, respectively. In addition, (*E*)-2-octenal (OAV 149204), geranial (OAV 57710), octanal (OAV 35044), eucalyptol (OAV 17917), and (*E*)-2-decenal (OAV 12744) in FAT had higher OAVs. On the other hand, in DAT, higher OAVs were observed for (*E*)-2-octenal (OAV 56954), geranial (OAV 38365), eucalyptol (OAV 22318), *α*-phellandrene (OAV 12993), octanal (OAV 12336), and linalool (OAV 10562). In contrast, the OAVs of *α*-phellandrene, linalool, and eucalyptol in DAT were significantly higher than those in FAT.

Comparison with literature data concluded that 11 of the 23 key odorants were reported for the first time in the FAT, which could be attributed to different origins of samples or different extraction methods (Liang et al., 2023). Up to now, few reports on the key odorants in DAT have been published.

3.6. Correlation between samples, key odorants, and sensory attributes

The O2PLS model was employed to explore the relationship between the chemometric and sensory properties of FAT and DAT based on 23 key odorants and seven attributes. As shown in Fig. 3, DAT was positively correlated with spicy, fatty, and woody notes, which was attributed to the contributions of linalool (**41**), neral (**29**), *trans*-nerolidol (**53**), *α*-pinene (**1**), sabinene (**4**), *α*-phellandrene (**6**), limonene (**7**), *α*-terpineol (**45**), and elemol (**54**), which is consistent with the PCA results. FAT showed a positive correlation and weak correlation with floral and herbaceous notes, respectively; and connected to the compounds of (−)-terpinen-4-ol (**43**), L-*α*-terpineol (**44**), geranial (**30**), decanal (**25**), (*Z*)-2-decenal (**27**), (*E*)-2-decenal (**28**), geraniol (**50**), octanal (**21**), (*E*)-2-dodecenal (**31**), *β*-pinene (**3**), ocimene (**11**), and 2-nonanone (**61**). The weak correlation between the sweet note and DAT was due to its low sensory score. Because six odorants (**6**, **7**, **11**, **29**, **30**, and **31**) out of the 23 key odorants exhibited strong citrus notes, which had relatively high scores in both FAT and DAT, there was no obvious correlation.

3.7. Recombination and omission experiments

Chromatographic processes separate olfactory stimuli the isolates each other, resulting in the deficiencies of sensory interactions, such as synergistic and inhibitory effects (Baldovini & Chaintreau, 2020). Consequently, to further validate the key odorants of FAT, it was necessary to perform aroma recombination and omission experiments. The aroma recombination model was established according to 23 odorants with OAV > 1 in their quantitated concentrations in FAT. Twelve panelists were asked to compare and score the odor attributes of the recombinant and FAT SAFE isolates using QDA. From Fig. 1b, it can be seen that the aroma profile of recombinant was similar to those of the FAT isolate and there were no significant differences between them ($p > 0.05$). Other five attributes in the isolate were the same as those in the recombinant. This indicated that the typical aroma profile of FAT could be successfully simulated by the combination of these 23 compounds, which was further corroborated the accuracy of the identification and quantitation results.

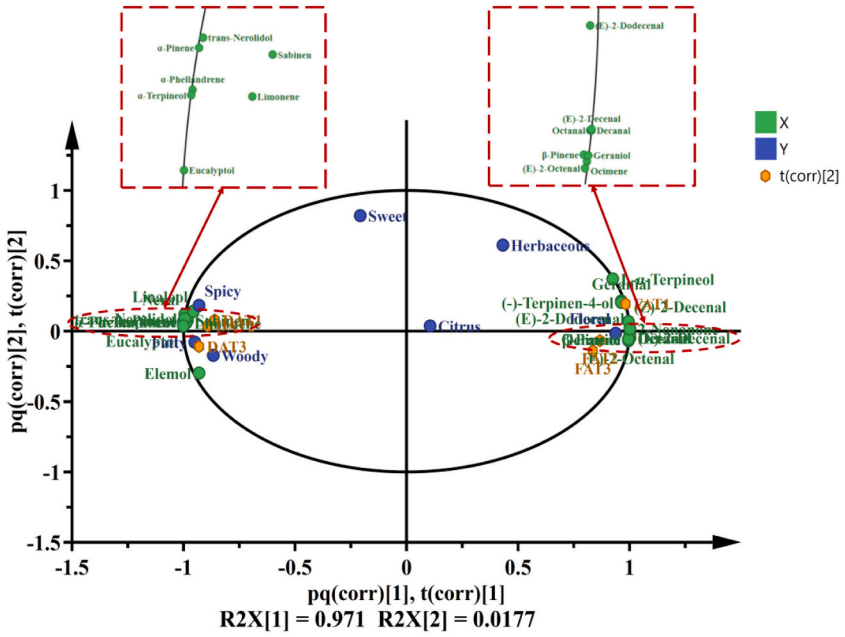

Fig. 3. The correlation between sensory properties and key odorants by O2PLS modeling of FAT and DAT.

To determine whether odorants with higher OAVs contributed greatly to the FAT aroma, 18 omission models, in which individual compounds or groups of compounds were omitted, were prepared, and evaluated by triangle tests. According to Table 3, M1, M6, and M7 resulted in the most noticeable differences in aroma ($p \leq 0.001$). The results for M1 indicated that aldehydes strongly influenced the odor of FAT. (*E*)-2-Dodecenal (M4, $p \leq 0.01$), geranial (M6, $p \leq 0.001$), octanal (M7, $p \leq 0.001$), and eucalyptol (M8, $p \leq 0.01$) were responsible for the fatty, citrus, and herbaceous attributes in FAT, respectively. Therefore, the omission experiments demonstrated the importance of fatty, citrus, and herbaceous odors in the overall aroma profile of FAT and showed that these four compounds contributed greatly to the overall aroma of FAT. The omission of alcohols and terpene hydrocarbons was also perceived by 8 of 12 panelists (M2 & M3, $p \leq 0.05$), suggesting that the floral, sweet, and woody aroma also obviously influenced the overall odor profile of FAT. The omission of (*E*)-2-octenal and (*E*)-2-decenal (M5 and M9, $p \leq 0.05$) also led to significant differences in aroma, indicating that these odorants made an essential aroma contributed to the overall aroma of FAT. However, nine compounds exhibited no significant differences in the omission experiment. The omission test further confirmed that six odorants were identified as the most key odorants among the 23 potent compounds. In conclusion, these key odorants were the main contributors to the typical overall aroma of FAT.

Table 3
Omission experiments from the recombination model.

Model	Odorants omitted from therecombination model	Correct judgmentsfrom 12 panelists[a]	Significance[b]
M1	All aldehydes	12	***
M2	All terpenes	8	*
M3	All alcohols	8	*
M4	(*E*)-2-Dodecenal	9	**
M5	(*E*)-2-Octenal	8	*
M6	Geranial	11	***
M7	Octanal	10	***
M8	Eucalyptol	9	**
M9	(*E*)-2-Decenal	8	*
M10	Linalool	5	–
M11	Geraniol	6	–
M12	*α*-Pinene	7	–
M13	*α*-Phellandrene	4	–
M14	*β*-Pinene	4	–
M15	Sabinene	6	–
M16	*trans*-Nerolidol	3	–
M17	Limonene	6	–
M18	Ocimene	5	–

[a] Number of correct judgments from 12 panelists.
[b] * Significant ($p \leq 0.05$); ** highly significant ($p \leq 0.01$); *** very highly significant ($p \leq 0.001$).

4. Conclusion

In summary, 74 volatiles were identified in FAT and DAT. Out of these, 49 compounds (43 and 42 in FAT and DAT, respectively) were screened as odor-active compounds using GC–MS–O, and their FD factors were measured using AEDA. The FD factors of the odor-active compounds ranged from 1 to 6561. Odorants with FD factors $\geq$ 27 were quantitated, and out of them, 23 odorants were identified as key odorants. Recombination and omission experiment further verified the determined key odorants, and the aldehydes contributed the most to the overall aroma profile of AT. The concentration of aldehydes decreased significantly after drying, whereas that of terpene hydrocarbons increased. In generally, AT mainly exhibits the citrus, herbaceous, and fatty attributes. Upon drying, the floral and sweet notes of FAT were weakened, while the woody and spicy attributes were strengthened. The results of PCA and OPLS-DA indicated that 20 compounds, including *α*-copaene, *α*-phellandrene, decanal et al. were significantly affected by drying, and the O2PLSDA results revealed that FAT exhibited a positive correlation with floral and herbaceous notes, while DAT exhibited a significant positive correlation with spicy, fatty, and wood aroma. The present study insights into the key odorants of FAT and DAT and provides a foundation for the reasonable application of FAT and DAT in the food industry. Because certain odor-active compounds are currently not available, future investigations will involve synthesizing them in a laboratory or extracting them from natural sources. Moreover, their contributions to the overall odor profiles of FAT and DAT will be investigated in the future.

CRediT authorship contribution statement

Miao Liang: Formal analysis, Investigation, Visualization, Writing – original draft, Methodology. **Yajian Wu:** Data curation, Investigation.

Rui Wang: Validation. **Zhimin Zhang:** Formal analysis, Software. **Runhu Xin:** Formal analysis. **Yuping Liu:** Project administration, Resources, Funding acquisition.

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

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

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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.2024.101344.

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