Valid evaluation of volatile flavor composition of fresh and dehydrated *Tuber indicum* with different drying methods

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ABSTRACT

The effects of three drying methods on volatile flavor components of *Tuber indicum* were studied. After hot air drying (AD), vacuum drying (VD), and vacuum-freeze drying (FD), flavor components were analyzed by headspace solid phase microextraction GC-MS and electronic nose (E-nose). The results from GC-MS showed that aldehydes (54.8%) and alcohols (31.4%) are the two dominant chemical species in fresh *T. indicum* and eight carbon (C8) compounds including 1-octen-3-ol, 3-octanol, n-octanol, 3-octanone. After dehydrating, C8 compounds, aldehyde, and ester components reduced, while alkanes, heterocyclic, and sulfur components were produced. Multivariate statistical analysis of the GC-MS revealed the components responsible for the chemical differences between fresh and three drying samples. In addition, E-nose could discriminate fresh and three drying samples. The result obtained by E-nose showed good identity compared with GC–MS. Therefore, FD was the optimal method to preserve *Tuber indicum* with the most retained fresh flavor.

1. Introduction

*Tuber indicum* (Chinese black truffle), edible fungi found majorly underground, is not only rich in nutrients and distinctive flavor, but also showing resistance to bacteria and virus (Dahham, Al-Rawi, Ibrahim, Majid, & Majid, 2016; Miao et al., 2014). It is a popular mushroom product among consumers. However, *Tuber indicum* is extremely susceptible to degradation, which brings difficulty to storage and transportation, and consequently results in great loss of food quality as well as economic value (Miao et al., 2014). Drying is proved to be an effective method in order to extend food shelf life, and has been widely used in the processing of edible fungi (Bhattacharya, Srivastav, & Mishra, 2015; Huang, Zhang, Wang, Mujumdar, & Sun, 2012), as the drying of mushroom (Guo, Xia, Tan, Chen, & Ming, 2014), *Agaricus bisporus* (Kumar, Singh, & Singh, 2013), and porcini (Aprea et al., 2015). There are three common drying methods currently used in massive production of dehydrated fruits and vegetables, including hot air drying (AD), vacuum drying (VD), and freeze-vacuum drying (FD) (Qiao, Fang, Huang, & Zhang, 2013; Saxena, Malty, Raju, & Bawa, 2012). Many studies have reported the changes in color, texture, rehydration capability, mechanical property, and flavor profile caused by different drying methods (Culleré, Ferreira, Venturini, Marco, & Blanco, 2013; Guine & Barroca, 2012; Pei et al., 2016; Vega-Gálvez et al., 2012). For instance, the hardness of pepper increases as the drying temperature increases, and low temperature drying with AD has little influence on the sample color (Guine & Barroca, 2012). The volatile flavor components of *T. indicum* are chiefly aldehydes and alcohols, and most of which are eight carbon chain (C8) compounds with distinct aroma (Culleré et al., 2013). A research studying the effects of FD and microwave vacuum drying (MD)
on the volatile components of *Agaricus bisporus* concluded that drying by MD had more similar volatile components to fresh *Agaricus bisporus* than the ones dried by FD (Pei et al., 2016). The effects of four drying methods (AD, VD, MD, and FD) on the flavor components of fresh ginger slices were compared and showed that the MD retained the most amount of flavor components, followed by VD and AD (Ding et al., 2012). Although there are studies focusing on the effects of different drying methods on flavor profiles, research specifically about *T. indicum* remains unexplored. Therefore, to explore the volatile profile change of *T. indicum* in different drying methods, we have designed and adopted three commonly used drying methods, including AD, VD, and FD, to dehydrate *T. indicum*. Hence, the impacts of drying methods on flavor components of *T. indicum* would be compared and investigated. Moreover, the results would be used to recommend the most suitable dehydration processing in order to achieve better preservation quality of natural fresh flavors of *T. indicum*.

2. Materials and methods

Ascorcaps of *Tuber indicum*, with original moisture content 76 ± 0.15% (wet base), was purchased from the local market in Kunming city, China and then refrigerated at 4°C until use. The samples were washed and cut to 5 mm slices prior to use.

2.1. Drying methods and process of *T. indicum*

2.1.1. Hot Air Drying

Hot air drying procedure used in this experiment was similar to that described by other researchers (Ferencz, Czukor & Cserhalmi, 2014; Kotwaliwale, Bakane & Verma, 2007; Li, Duan & Xu, 2014). Fresh evenly cut *T. indicum* slices were placed into an electronic heating air blowing dryer (Model 101-3A, Xinnuo Instrument Equipment Co. Ltd., Shanghai, China) and the dryer was heated to a temperature at 60°C and dried for 9 h.

2.1.2. Vacuum Drying

VD method was performed as described by Wu et al. with some modifications. Fresh evenly-cut *T. indicum* slices were placed into a vacuum dryer (Model DZF-6020, Shanghai Jinghong Equipment Co., Ltd, Shanghai, China) with a temperature at 50°C, under the vacuum pressure 90 kPa and dried for 12 h.

2.1.3. Vacuum Freeze-Drying

Fresh evenly cut *T. indicum* slices were frozen at a temperature at −24 ± 2°C for 24 h and the frozen samples were placed in the 12 L vacuum freeze dryer (Model, Labconco company, Kansas City, MS, USA) with heating plate temperature at 40°C, vacuum pressure 0.1 kPa and cold trap temperature at −83 ± 1°C (Pei et al., 2014).

2.1.4. Preparation of *T. indicum* powder

Upon drying to the moisture content of 5% by above mentioned three methods, that is AD, VD, and FD, the dried slices of *T. indicum* were stored in valve bags at room temperature. The slices were pulverized into powder by a super micro mill (Model FW100 Tianjin Taisme Equipment Co., Ltd., Tianjin, China), reaching the particle size of 200 mesh, and then were collected and used in GC-MS analysis and E-nose analysis.

2.2. Headspace solid-phase microextraction (HS-SPME) of volatile compounds

Dried samples and fresh samples were grinded adequately and weighed in a 20 ml vial with 0.05 g dry weight internal standard of 1-decanol solution (80 µg/mL) in methanol, and closed with a silicon cap. The cap was perforated for 40 min with a divinylbenzene/carboxen/polydimethylsiloxane (DVB/ CAR/PDMS, 50/30 m/m) fiber holder (Supelco Ltd., Bellefonte, PA, USA) after 40 min equilibrating in a 60°C water bath. The analytes were finally desorbed for 5 min at 250°C in the GC injector in splitless mode.

2.3. GC-MS analysis of volatile compounds

In the analysis, the instrument for GC-MS was 7890A-5975C GC-MS (Agilent company, Santa Clara, CA, USA) with settings: HP-5MS capillary column (30 m × 0.25 mm i.d. and 0.25 µm film thickness) from (J&W Scientific, Folsom, CA, USA). Column temperature was initially set and held at 40°C or 3 min, and then increased to 130°C at 5 min, held for 3 min. In the second ramp, temperature increased to 200°C from 8 min to 15 min, and finally reached 250°C at 20 min and held for 2 min. Helium was the carrier gas at a flow rate of 0.8 mL/min. Mass spectra were acquired in an electron impact mode. MS was taken at 70 eV ionization energy in the 25–450 amu mass range, and the ion source temperature was 200°C. The volatile compounds were tentatively identified by matching the mass spectra with the spectra of reference compounds in both the Wiley mass spectra library (6th edition) and the NIST/EPA/NIH mass spectra library (version 1.5a). The results from volatile analyses are provided in peak area counts of the compounds identified (Yang et al., 2016).

2.4. Electronic nose analysis

The flavor differences of fresh and dried powder samples of *T. indicum* were analyzed by an electronic nose detector (E-nose) equipped with a headspace auto-sampler (Fox 3000, Alpha M.O.S., Toulouse, France) comprising an array of 12 sensors (LY2/LG, LY2/G, LY2/AA, LY2/GH, LY2/gCT, LY2/gCT, T30/1, P10/1, P10/2, P40/1, T70/2, PA/2). Fresh *T. indicum* sample (1 g) and dried powder obtained by three drying methods (0.20 g per sample) were stored in the 20 mL of headspace vials from Supelco Inc. The E-nose sampling bottles were equilibrated under the temperature of 50°C for 10 min and then were introduced by the carrier gas, which was clean and dry air, with flow rate of 150 mL/min. The sampling gas volume was 2.5 mL. The sampling introduction was finished within 1s and the temperature of injection needle was 60°C. Parameter acquisition time was 120 s and lag time was 10 min. Sensor LY2/G sensitive to alcohols, ketones and aldehydes; LY2/AA sensitive to ketones; LY2/GH sensitive to alcohols; LY2/gCT sensitive to ethanol; T30/1 sensitive to acid compounds, PA/2 sensitive to ketones sulfur compounds and acids (Oupathumpanot & Suvonsichon, 2013; Yao et al., 2015).
2.5. Data analysis

The data were analyzed by ANOVA with SPSS 18.0. The significant difference was defined as $p < 0.05$. Least significant difference-test (LSD) was used in multiple comparative analyses. In addition, multivariate statistical analysis was performed using simca software version 14.1. The output data from the E-nose was analyzed by using software (AlphaSoft version 3.0.0, Toulouse, France).

3. Results and discussion

HS-SPME-GC-MS analysis was performed to analyze the volatile flavor components of fresh and processed T. indicum by AD, VD and FD. From the results shown in Table 1, total of 45 volatile compounds were detected from the fresh T. indicum samples, among which 21 compounds with the AD samples, 20 compounds with the VD samples and 18 compounds with the FD samples.

3.1. Detection of volatile flavor components of fresh T. indicum

From the analysis of the volatile components in fresh T. indicum by HS-SPME-GC-MS, the results showed that the major chemical species were aldehydes (54.8%) and alcohols (31.4%). These results were illustrated in Figure 1 and they are similar to the study of Culleré et al. (2013). The major flavor compounds in fresh T. indicum were compounds with C8 compounds, including 1-octen-3-ol, 3-octanol, n-octanol, and 3-octanone. The production of C8 compounds has been attributed to enzymatic degradation of unsaturated fatty acids; for instance, lipogenase existed in situ produces C8 compounds. Fresh T. indicum had the highest amount of aldehydes with low odor threshold, including 3-methylbutanal, hexanal, benzaldehyde, benzeneacetaldehyde, and nonanal. Other volatile components were detected, including phenethyl alcohol, ethyl hexanoate, ethyl hexadecanoate, and 2-pentylfuran. In addition to aforementioned C8 compounds, a few acids constitute the flavor of fresh T. indicum, showed in Table 1.

3.2. Summary of volatile profiles of T. indicum from different drying methods

HS-SPME-GC-MS is one of the most commonly used techniques in analysis of volatile compounds because chemical profiles from the headspace extraction often represent flavor components. The volatile compounds from our dehydration study of T. indicum in three drying methods (FD, AD, and VD) were summarized in the following and in Figure 1.

3.2.1. Vacuum-Freeze Drying

There were 18 volatile components characterized in FD samples, which were mainly alcohols (37.1%), aldehydes (29.8%), and alkanes (18.7%), counting on more than 85% of the total flavor compounds (Figure 1). The major single compounds identified were phenethyl alcohol (26.1%), hexanal (15.3%), 2,2,4,6,6-pentamethylheptane (12.7%), and 1-octen-3-ol (10.1%).

3.2.2. Hot Air Drying

Heterocyclic volatiles (54.5%) were the major compounds identified in the AD, and they counted for more than half of the total 21 compounds (Figure 1). The others were alcohols (31.6%), aldehydes (11.1%). The major compounds were 2-furancarboxaldehyde (19.5%), 2-pyrollinone (17.7%), phenethyl alcohol (16.0%), 2,3-butanediol (12.7%), and 1-octen-3-ol (2.6%).

3.2.3. Vacuum Drying

There was total of 20 compounds identified, and the alcohol (43.2%) was the most abundant family, followed by volatile acids (16.5%) and aldehydes (15.6%). Similar to the results of FD sample, the major compounds were phenylethyl alcohol (41.5%) and the others included butanoic acid (16.5%), 1-buty1-1H-pyrrole (7.3%), hexanal (5.7%), and dodecanol (5.1%). There was no 1-octen-3-ol identified and also the contents of other C8 compounds were very low. Although the boiling point of “mushroom” flavor “1-octen-3-ol is 175°C at 101 kPa and 171°C at 90 kPa, it is a typical volatile compound particularly found in fungi. The possible explanation that 1-octen-3-ol was not in the profile of VD and that is it is a volatile compounds and easily vaporized. Hence it is evaporated in VD that was a prolonged process (12 h) at vacuum of 90 kPa and heated (50°C) temperature.

3.3. Influence of drying methods on the volatile flavor components of T. indicum

From Table 1, it could be concluded that the three drying methods had significant influences on the flavor of T. indicum. Chemical analysis showed that after drying, most contents of alcohols, aldehydes, ketones, and esters were reduced with different extent.

There was no significant difference between fresh T. indicum and FD samples of 1-octen-3-ol concentration ($p > 0.05$), but significantly higher than that of AD samples ($p < 0.05$), and there was no 1-octen-3-ol detected from FD samples, suggesting that the FD had the most advantage among three drying methods in the retention characteristic flavor compound of T. indicum. Because the major flavor compounds in fresh T. indicum are C8 compounds, such as 1-octen-3-ol, 3-octanol, 1-octanol, and also 3-octanone, it is important to identify and profile these C8 compounds for flavor analyses. The HS-SPME-GC-MS data also showed the overall content of other C8 compounds, including alcohols, such as 3-octanol, (E)-2-octenal, 3-octanone, and compounds with short chain such as 1-hexanol and 1-heptanol were greatly decreased, even reached the detection limit of the instrument. Hence, the drying process had a direct impact on the loss of C8 volatile molecules by evaporation or reaction with other components. However, phenethyl alcohol content in drying samples was significantly higher than that of fresh samples ($p < 0.05$). The unusual increase could be explained that phenethyl alcohol was the thermal Strecker degradation product of phenylalanine, which was enhanced by the heat of drying process (Cosmai, Summo, Caponio, Paradiso, & Gomes, 2013). The drying process also resulted in the loss of aldehydes. The only aldehyde survived the AD samples is hexanal, whereas there were five aldehydes in the VD samples, including hexanal, 2-phenyl-2-butenal, nonenal, benzaldehyde, and benzeneacetaldehyde, and there were six aldehydes characterized in the FD samples, 3-methylbutanal, hexanal, 2-phenyl-2-butenal, nonenal, benzaldehyde, and benzeneacetaldehyde. The aldehyde content in the FD samples was the most similar to fresh T. indicum, indicating a better retention quality of the fresh flavors. This phenomenon could be explained that the high drying temperature of AD facilitated the loss of large amount of...
| Retention time (min) | Volatile compounds | Fresh (%) | AD (%) | VD (%) | FD (%) | Odour description |
|----------------------|--------------------|-----------|--------|--------|--------|--------------------|
| 2.92                 | 2,3-Butanediol     | nd        | 12.692 ± 1.021 | nd     | nd     | Cheesy caramel     |
| 6.72                 | 1-Octanediol       | 2.720 ± 0.248 | nd     | nd     | nd     |                    |
| 7.10                 | 1-Heptanediol      | 1.978 ± 0.161 a | 12.692 ± 1.021 | nd     | nd     |                    |
| 10.71                | 1-Octen-3-ol       | 5.095 ± 0.757 a | 0.298 ± 0.025 d | nd     | 10.126 ± 1.157 a | Mushroom           |
| 11.32                | 3-Octanol          | 4.947 ± 0.484 a | nd     | nd     | nd     | Mushroom           |
| 14.16                | 1-Octanol          | 5.175 ± 0.542 a | 1.061 ± 0.099 b | nd     | nd     |                    |
| 15.72                | Phenethyl alcohol  | 7.642 ± 0.659 d | 16.002 ± 1.766 c | 41.475 ± 4.335 a | 26.060 ± 2.265 b | Honey, spice, rose |
| 1.75                 | 3-Methylbutanal    | 17.623 ± 1.680 a | nd     | nd     | nd     | Cocoa, almond      |
| 3.25                 | Hexanal            | 8.519 ± 0.803 b | 1.071 ± 0.124 d | 5.667 ± 0.533 c | 15.317 ± 1.376 a | Grass, tallow, fatty |
| 7.83                 | Heptanal           | 2.606 ± 0.265 b | nd     | nd     | nd     | Fat, citrus, rancid |
| 9.93                 | Benzenaldialdehyde | 7.027 ± 0.719 a | nd     | nd     | 2.504 ± 0.260 c | 4.714 ± 0.411 b |
| 13.11                | Benzenesulfide     | 5.502 ± 0.541 a | nd     | 0.863 ± 0.074 c | 1.344 ± 0.146 b | Almond, burnt sugar |
| 13.66                | (E)-2-Octenal      | 8.870 ± 0.772 a | nd     | nd     | nd     |                    |
| 15.42                | Nonanal            | 3.669 ± 0.344 a | nd     | 3.163 ± 0.316 a | 2.564 ± 0.297 b | Fat, citrus, green |
| 21.48                | 2-Phenyl-2-butenal | 1.000 ± 0.125 b | nd     | 3.425 ± 0.312 a | 0.902 ± 0.094 b |                    |
| 10.91                | 2-Methyl-3-oxanone | 0.311 ± 0.035 a | nd     | nd     | nd     | Mushroom           |
| 10.98                | 3-Octanone         | 6.064 ± 0.558 a | nd     | nd     | nd     | Nut, crushed bug   |
| 12.71                | 3-Octen-2-one      | nd        | 2.533 ± 0.216 a | 1.836 ± 0.133 b | 1.373 ± 0.118 b |                    |
| 22.27                | 2-Undecanol        | 1.318 ± 0.105 b | 1.039 ± 0.14 c | 1.704 ± 0.166 a | 1.373 ± 0.118 b |                    |
| 5.07                 | 3-Methylbutanoic acid | 0.500 ± 0.052 c | nd     | 16.493 ± 1.523 a | 5.752 ± 0.514 b | Apple              |
| 21.37                | Nonanoic acid      | 0.444 ± 0.046 a | nd     | nd     | nd     | Green, fatty       |
| 6.22                 | Ethyl 3-methylbutanoate | 1.403 ± 0.155 a | nd     | nd     | nd     |                    |
| 11.49                | Ethyl hexanoate    | 0.563 ± 0.053 a | nd     | nd     | nd     |                    |
| 18.84                | Ethyl octanoate    | 0.379 ± 0.037 a | nd     | nd     | 0.427 ± 0.043 a |                    |
| 33.87                | Isobutyl octyl phthalate | 1.325 ± 0.104 a | nd     | nd     | nd     |                    |
| 35.11                | Ethyl hexadecanoate | 0.482 ± 0.041 a | nd     | nd     | nd     |                    |
| 7.46                 | Styrene            | 0.430 ± 0.043 a | nd     | nd     | nd     | Balsamic, gasoline |
| 10.69                | 2,2,4,6,8-Pentamethyl-heptane | 6.092 ± 0.559 b | 1.748 ± 0.163 c | 12.670 ± 1.071 a |                    |
| 17.76                | 2,8-Dimethyl-decane | nd     | 1.268 ± 0.131 a | 0.978 ± 0.089 b | nd     |                    |
| 18.85                | Dodecane           | 1.644 ± 0.136 c | 4.179 ± 0.435 b | 5.101 ± 0.531 a | 1.273 ± 0.104 b | Alkane             |
| 26.33                | Tetradecane        | nd        | 2.107 ± 0.201 a | 1.273 ± 0.104 b |                    |
| 5.29                 | 2-Furanmethanol    | nd        | 19.514 ± 1.839 a | nd     | nd     |                    |
| 7.59                 | 2(5H)-Furanone     | nd        | 2.379 ± 0.28 a | 1.412 ± 0.173 b | nd     |                    |
| 7.70                 | 2,3-Dimethylpyrazine | nd     | 0.840 ± 0.084 a | nd     | nd     |                    |
| 7.47                 | 2-acetylfluran     | nd        | 1.847 ± 0.18 a | nd     | nd     |                    |
| 10.80                | 2-Pentylfluran     | 0.545 ± 0.05 c | 0.799 ± 0.082 b | nd     | 1.690 ± 0.167 a |                    |
| 11.13                | Trimethylpyrazine   | nd        | 3.325 ± 0.346 a | nd     | nd     | Roast, potato, must |
| 11.70                | 2-Ethyl-6-methylpyrazine | nd     | 2.526 ± 0.229 b | nd     | nd     |                    |
| 13.42                | 1-Butyl-1H-pyrorle | nd        | 7.316 ± 0.727 a | nd     | nd     |                    |

(Continued)
| Retention time (min) | Volatile compounds | Fresh (%) | AD (%)         | VD (%)         | FD (%)         | Odour description |
|----------------------|--------------------|-----------|----------------|----------------|----------------|------------------|
| 13.51                | 2-Acetylpyrrole    | nd        | 4.62 ± 0.47    | nd             | nd             |                   |
| 13.60                | 2-Pyrrolidinone    | nd        | 17.74 ± 1.66   | nd             | nd             |                   |
| 25.14                | Dihydro-2-pentyl-2(3H)-furanone | nd       | 0.69 ± 0.06 b   | 1.18 ± 0.115 a | nd             |                   |
| 7.87                 | Dimethyl sulfone   | nd        | 1.10 ± 0.108 b  | 1.02 ± 0.131 b | 2.96 ± 0.237 a | Sulfur, burnt     |
| 18.71                | 2,3-Dihydrothiophene | nd      | 1.71 ± 0.173   | nd             | nd             |                   |
| 36.24                | 2(5H)-Thiophenone  | nd        | nd             | 0.50 ± 0.04 b  | nd             |                   |

Note: The measurement is at dry base except fresh *T. indicum* samples. Each value is expressed as mean ± SD (n = 3). Different lowercase letters in a row means significantly different t (p < 0.05). nd: not detected. 

References for odor description: Culleré et al. (2013); Maga (1981); Sucan and Weerasinghe (2005).
fresh flavors, comparing to the low temperature drying process as in the FD. Therefore, the most important factor in preserving the fresh flavor of *T. indicum* is low temperature. Similar results were reported in the research of drying methods impacting of perfume profiles.

The content of long chain alkanes and heterocyclic compounds containing nitrogen increased significantly. Alkanes were majorly produced by lipid oxidation (Sucan & Weerasinghe, 2005). Heterocyclic compounds were formed by Maillard reaction in the drying process, which could produce oxygen-, nitrogen-, and sulfur-containing heterocyclic species (Sucan & Weerasinghe, 2005). There were ten major heterocyclic compounds generated in AD processing such as 2-furanmethanol (19.5%), 2-pyrrolidinone (17.7%), 2-acetylpyrrolole (4.6%), 2-(5H)-furanone (2.6%), trimethylpyrazine (3.3%), 2-ethyl-6-methylpyrazine (2.5%), 2-acetylfuran (1.8%), 2,3-dimethylpyrazine (0.8%), and dihydro-5-pentyl-2-(3H)-furanone (0.7%). In the VD samples, three major heterocyclic compounds were detected, including 1-butyl-1H-pyrrole (7.3%) and two minor ones, 2-(5H)-furanone (1.4%) and dihydro-5-pentyl-2-(3H)-furanone (1.2%). There was only one compound, 2-pentylfuran, which was a lipid oxidation product generated in the FD process with small content (1.7%) compared to other flavor compounds in the FD samples.
High temperature in the AD process facilitated Maillard reaction, Strecker degradation and aldol condensation, which generated heterocyclic aroma compounds. In other words, the generation of some volatile compounds, such as pyrazine molecules, requires high temperature, and the drying temperature in the methods of VD and FD was too low to form these pyrazine products.

In summary, with HS-SPME-GC-MS technique, 45 volatile compounds were identified from fresh *T. indicum*. The effects of three different drying methods on these fresh volatile compounds were investigated. The flavor profile of drying *T. indicum* from FD processing was the closest to the fresh *T. indicum*, compared to the large amount of pyrazine compounds formed by AD processing and the great loss of aldehyde species in VD processing. Therefore, the FD is the most proper way among the three evaluated processes to preserve the original fresh flavor of *T. indium* in the presented study.
3.4. Principal component analysis of flavor components

Principal component analysis (PCA) is a multivariate statistical analysis method that transfers multiple indicators to a few comprehensive indicators by reducing dimensions. The intention of this method is data simplification and a clear demonstration of the relationships among variables. PCA is adopted to analyze the volatile composition. The object of data analysis is the volatile components of four samples mentioned above (samples from fresh \textit{T. indicum}, and AD, VD and FD). The analysis results are illustrated in Figure 2. The first three principal components accounted for 98.7% of the total variance, with values for PC1, PC2 and PC3 of 49.8%, 31.1% and 17.8%, respectively. Fresh \textit{T. indicum}, AD, VD, and FD samples formed four clusters. Fresh \textit{T. indicum} and three drying samples separate along PC1 where fresh \textit{T. indicum} are located on negative PC1 while three drying samples occupy positive PC1 (Figure 2(a)). The three drying samples were similar along PC1, but there was clear separation on the PC2 or PC3 axis (Figure 2(a,b)). Moreover, AD samples were obvious difference from VD, and FD samples. To analyze the variables responsible for the clustering, the loading plot of PCA was performed (Figure 2(c,d)). The results showed chemical composition contributing to the separation of drying samples were 2,3-Butanediol, 2-Furanmethanol, 2,3-Dimethylpyrazine, 2-acetylfuran, Trimethylpyrazine, 2-Ethenyl-6-methylpyrazine, 2-Acetylpyrrole, 2-Pyrollidinone and 2,3-Dihydrothiophene.

3.5. \textit{E-nose} analysis

\textit{E-nose} technology provides comprehensive information about volatile profiles, which is commonly used to differentiate and analyze complicated flavor components. The volatile profile of samples subjected to the three drying methods and fresh samples were also performed by Fox 3000 E-nose. The original response data generated by 12 sensors of \textit{E-nose} were collected and transformed to radar graphs, as shown in Figure 3. Generally, the similar shape of these radar graphs implied some similarities among these samples. The response values ofLY2/LG, T30/1, P10/1, P10/2, P40/1, T70/2 and PA/2 sensors to fresh \textit{T. indicum} and three drying samples were positive, LY2/G, LY2/AA, LY2/Gh, LY2/gCT, LY2/gCT were negative. The tends of connect shapes were different between fresh sample and three drying samples, and three drying samples were in substantial agreement.

Compared with the fresh \textit{T. indicum}, the response values of sensor decreased of the three drying samples, indicating that the drying caused the loss of fresh \textit{T. indicum} flavor. The response values of LY2/G, LY2/AA, LY2/Gh, LY2/gCT of FD samples was higher than AD samples, those sensors (LY2/G, LY2/AA, LY2/Gh, LY2/gCT) sensitive with ethanol, aldehydes and ketone, so the result indicated that content of ethanol, aldehydes, ketones of FD samples higher than AD samples. The flavor profiles generated by \textit{E-nose} were in accordance with the experimental data detected by GC-MS.

3.6. Principle component analysis from \textit{E-nose} data

The PCA from \textit{E-nose} data of the flavor compounds of fresh and three different drying processed samples of \textit{T. indicum} was conducted on a \textit{E-nose} software and the volatile flavor component analysis of the original response values was performed for principle component analysis. As illustrated in Figure 4, the cumulative variance contribution rates of PC1 and PC2 of \textit{T. indicum} samples were 99.505% and 0.466%, respectively, which indicated that PC1 and PC2 had a large amount of information and could reflect the whole information of the samples. The discriminate index (DI) is the representation value of the degree of sample discrimination provided by data statistics software of \textit{E-nose}. The DI value of Figure 4 is 98, indicating that the electronic nose can significantly differentiate samples of different drying methods. The volatile odor difference from fresh samples and samples of three drying methods is apparent, and this difference can be displayed on the platform of PC1 and PC2. The flavor of the fresh \textit{T. indicum} is the closest to that of the FD samples, which indicates proximity of flavor similarity between the two samples and is also consistent with the results detected by HS-SPME-GC-MS.

4. Conclusions

This study compared the volatile components of fresh, VD, AD and FD samples by using HS-SPME-GC-MS. In fresh \textit{T. indicum}, 45 volatile components were detected, among which aldehydes and alcohols were prevalent in fresh \textit{T. indicum}. The main flavor components included C8 compounds (1-octen-3-ol, 3-octanol, n-octanol, 3-octanone), 2-Octenal, 3-Octanone, 3-Octen-2-one, 2-Undecanone, Ethyl 3-methylbutanoate, Ethyl hexanoate, Isobutyl octyl phthalate, Ethyl hexadecanoate, Styrene.

Acknowledgments

The authors acknowledge financial support from China Agriculture Research System (CARS-20) and the Priority Academic Program Development of Jiangsu Higher Education Institutions (PAPD).
Disclosure statement

No potential conflict of interest was reported by the authors.

Funding

This work was supported by the China Agriculture Research System [CARS-20] and the Priority Academic Program Development of Jiangsu Higher Education Institutions (PAPD).

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