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Effect of hot air drying on volatile compounds of *Flammulina velutipes* detected by HS-SPME–GC–MS and electronic nose

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ABSTRACT

Volatile compounds are important factors that affect the flavor quality of *Flammulina velutipes*, but the changes occurring during hot air drying is still unclear. To clarify the dynamic changes of flavor components during hot air drying, comprehensive flavor characterization and volatile compounds of *F. velutipes* were evaluated using electronic nose technology and headspace solid phase micro-extraction combined with gas chromatography–mass spectrometry (HS-SPME–GC–MS), respectively. Results showed that volatile components in *F. velutipes* significantly changed during hot air drying according to the principal component analysis and radar fingerprint chart of electronic nose. Volatile compounds of fresh *F. velutipes* consisted mainly of ketones, aldehydes and alcohols, and 3-octanone was the dominant compound. Drying process could significantly decrease the relative content of ketones and promoted the generation of alcohols, acids, and esters, which became the main volatile compounds of dried *F. velutipes*. These may provide a theoretical basis for the formation mechanism of flavor substances in dried *F. velutipes*.

1. Introduction

Flammulina velutipes is a widely cultivated and commercially available mushroom in the world owing to desirable taste and aroma and high nutritional properties (Chang et al., 2013; Jing et al., 2014). With the rapid development of industrialized cultivation techniques of *F. velutipes*, it has become one of the fourth most popular edible mushrooms worldwide. Postharvest quality deterioration of *F. velutipes* such as loss of weight and firmness, browning, softening and rotting during storage results in its very short storage period, which severely reduces the value of the mushroom and restricts the development of *F. velutipes* industry (Wang et al., 2011). Although some measures such as low temperature, atmosphere packaging and chemical treatment were employed to prolong the storage period of *F. velutipes*, the storage time of *F. velutipes* was only prolonged from 4 days to about 20 days. Therefore, it is necessary to explore new technology for extending the preservation of *F. velutipes*.

Drying and dewatering technology plays a major role in food manufacturing or food processing activities worldwide, which is a cheap and convenient method to extend the shelf life of fruits and vegetables by reducing the moisture content. However, drying and dewatering impact the quality of food products, such as texture, taste, aroma and nutritional properties. The variety and relative content of volatile flavor compounds is an important factor that affects the aroma quality of dried fruits and vegetables. Some studies have shown that drying and thermal treatment could promote the production of some novel volatile substances and provide the formation of special characteristic flavor quality (Cho, Kim, Choi, & Kim, 2006; Misharina et al., 2010). However, flavor compounds changes of *F. velutipes* during drying process have not been reported.

In recent years, electronic nose technology (E-nose) and gas chromatography–mass spectrometry (GC–MS) were mainly used to analyze food flavor. E-nose, simulated sense of smell in mammals, has become an important method for detecting the characteristics changes of flavor profiles in food. E-nose analysis generally provided a comprehensive evaluation of the overall information scent used for quality control, fresh grading, the authenticity of identification and microbial testing of fruits and vegetables, meat, cereals, beverages and some other products (Baldwin, Bai, Plotto, & Dea, 2011; Berna, 2011; Papadopoulou, Panagou, Mohareb, & Nychas, 2013). E-nose technology has been used to identify the flavor differences among *F. velutipes*, *Lentinus*

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edodes, *Agaricus bisporus*, and *Pleurotus eryngii* Quel (Fujioka et al., 2013). GC–MS has the advantages of requiring less sample, high sensitivity, and rapid analysis and could clarify what the specific flavor compounds were, which was different from E-nose. However, less information is available on the application of E-nose technology and GC–MS in the flavor changes of *F. velutipes* during drying process.

In the present study, to clarify the evolution and formation of dried *F. velutipes* flavor during dehydration, air-drying treatment was used to dehydrate fresh *F. velutipes*. E-nose technology and headspace solid phase micro-extraction (HS-SPME) combined with GC–MS were used to evaluate the overall flavor quality changes and analyze dynamic changes of flavor ingredients of *F. velutipes* during air-drying treatment, respectively. These may provide a theoretical basis for clarifying the change mechanism of flavor compounds.

2. Materials and methods

2.1. Air drying of F. velutipes

Fresh *F. velutipes* was purchased from a local market (Nanjing, China). The sample was uniformly distributed in several trays (10 kg/m^2) and dehydrated at an air temperature of 60 °C and a constant relative humidity of ca. 20% in an experimental air dryer (DNF610, YAMATO Scientific Co. Ltd, Japan), which consists of two basic sections: air flow rate control and heat control (Marquez, Duenas, Serratosa, & Merida, 2012). Internal temperature of *F. velutipes* was monitored using a thermocouple probe. The measurements of moisture content and volatile compounds content under constant drying conditions were carried out every 2 h. Quantification of the volatile compounds was by comparison of peak-height with a known standard. A total of ten replicated experiments were performed.

2.2. Moisture content

Moisture content was determined by removing moisture at 105 °C and then calculated by the weight loss as a percentage of the initial weight (Li et al., 2015).

2.3. E-nose analysis

E-nose analysis of odors was carried out according to Lin et al. (2013). *F. velutipes* were placed in 20 ml sealed vials and heated at 50 °C for 10 min. an E-nose system (FOX 3000, Alpha MOS, Toulouse, France) equipped with 12 metal oxide gas sensors (MOS sensors) based on different sensing materials was used. During the measurement process the headspace gas was pumped into the sensor chamber at a constant rate of 100 ml/min via Teflon-tubing connected to a needle, using clean air as carrier gas. Same amount of sample (0.2 g dry weight) used each time was calculated according the moisture content of *F. velutipes*. W_t (g) = 0.2/(1−X_t), where W_t is the weight of *F. velutipes* after *t* hours drying and X_t is the moisture content of *F. velutipes* after *t* hours of drying.

2.4. HS-SPME–GC–MS analysis

HS-SPME–GC–MS analysis of volatile compounds in *F. velutipes* was performed as described by Laurienzo, Stasio, Malinconico, and Volpe (2010). *F. velutipes* was collected and ground in the presence of liquid nitrogen in a mortar. The powder of *F. velutipes* was put into a 100 ml glass vial. 2-Chlorobenzaldehyde (4 μl) was subsequently placed into the vial as internal standard (IS). The vials were sealed immediately with PTFE-silicone septa (Supelco, Bellefonte, PA, USA) and mixed by magnetic stirring (Laurienzo et al., 2010). Same amount of sample (0.2 g dry weight) was used each assay to keep a consistent sample-to-headspace ratio (8 ± 0.5%). To extract volatile compounds from *F. velutipes*, a 75 μm Carboxen/polydimethylsiloxane (CAR/PDMS) fiber was used and preconditioned at 250 °C for 20 min prior to analysis. The fibre was inserted into the sample vial through the septum and exposed to the HS for 35 min at 60 °C to collect the analytes. The distance between fiber tip and the sample bed was kept about 1 cm. Then the fiber was removed from the vial and inserted into the injection port of the GC–MS apparatus for analysis of volatile compounds. The volatile compounds were identified on the basis of matching the experimental mass spectra with a mass spectra library from NIST 98 data bank (NIST 08, Washington DC) and comparison of the fragmentation patterns with some reported in previous literatures. The relative contents presented here were calculated on the basis of peak area percentage, which expressed as % of total peak areas of all identified compounds. The peaks of 2-chlorobenzaldehyde in GC–MS total ion chromatogram of all samples were kept uniformity to ensure the instruments and the protocol are stable and the results are repeatable.

(1) *GC conditions:* The analysis of volatile compounds was performed on GC–MS apparatus (7890A/5975C, Agilent Technologies, Santa Clara, CA, USA). The analytes removal from the fiber was carried out by holding the injector temperature at 250 °C. Volatiles were separated using a 5% phenylmethyl silicone (HP-5) bounded phase fused silica capillary column (Hewlett–Packard, Palo Alto, CA, USA; 33 m × 250 μm i.d., film thickness 0.25 μm), operating at 80 kPa column heads pressure, resulting in a flow of 0.8 ml/min. The oven temperature program was set for 2 min at 45 °C, raised to 130 °C at a rate of 5 °C/min, ramped up to 200 °C at a rate of 8 °C/min, finally increased to 250 °C at a rate of 12 °C/min and maintained isothermal for 7 min (Cui, Wang, Yang, Wu, & Wang, 2015).

(2) *MS conditions:* The electron ionization source temperature was maintained at 200 °C and mass spectra were obtained by electronic impact at 70 eV. Temperature of interface and quadrupole were 280 °C and 150 °C, respectively. The data was collected at a rate of 1/scan over the range of 25–450.

2.5. Statistical analyses

The measured data were analyzed by principal component analysis (PCA) and radar fingerprint chart using AlphaSoft V9.1. Least significant differences (LSD) multiple comparison tests were then performed with a 95% confidence level.

3. Results and discussion

3.1. Moisture content

F. velutipes was dried at 60 °C and the moisture content in *F. velutipes* at different drying time was shown in Fig. 1. Within 12 h drying period, the moisture content of *F. velutipes* reduced from 90.8% to 13.1%, which was appropriate for long time storage of dried *F. velutipes*. Therefore, 12 h was considered as the termination of drying process. Interesting result was found that the internal temperature of *F. velutipes* steeply increased during 0–2 h drying at 60 °C, and then kept constant during 2–8 h as the moisture was evaporating at a fixed rate to dissipate thermal energy. As a result of the evaporation of most moisture in *F. velutipes*, the internal temperature of *F. velutipes* rose, and was higher than the environment temperature after 8 h of drying.