



Aromatic constituents and their changes of *Illicium verum* processed by different heating methods

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ABSTRACT

The aromatic constituents and their changes of *Illicium verum* during different heating processes and the practical applications were investigated in this study. Results of gas chromatography-mass spectrometry (GC-MS), gas chromatography olfactometry (GC-O) and corresponding sensory assessment showed that trans-anethole was the most representative and important component of aromatic volatiles in *Illicium verum*, which would cover up the scents of other ingredients. Furthermore, these results implied that the volatilization of trans-anethole reached the maximum at 100 °C during direct heating process and at 160 °C during frying heating process, which showed high similarity in sensory evaluation. The present study would provide the theory base for maximum utilizing the flavor of *Illicium verum* in food processing.

1. Introduction

Chinese traditional cooking, mainly including blanching, frying and stir-frying (Huan et al., 2015), makes food more delicious with better looking and smelling. Spices which imparts characteristic flavor to foods are extensively used in cooking industry in China. *Illicium verum* is a representative chinese spices of medicine food homology and an industrial crop in the tropic and subtropic areas of Asia (Scopel et al., 2016). The *Illicium verum* fruits are mainly consisted of bitter principle, tannins and essential oil (Chouksey et al., 2013), and recent researches discovered that the volatile essential oils exhibited significant anti-oxidant, antimicrobial and antifungal activities (Dzamic et al., 2009; Singh and Catalan, 2005).

Flavor substances of *Illicium verum* are volatile aromatic compounds emanated during cooking and processing (Reineccius and Peterson, 2013), which consist of many closely related isomers (Gu et al., 2016). The constituents of the flavor compounds would be influenced by the cooking temperature and time and determined mainly by gas chromatography-mass spectrometry (GC-MS) due to its high sensitivity and selectivity (Gu et al., 2016; Hudaib et al., 2002).

Recent researches mainly focused on the extraction, characteristics and bioactivities of volatile compounds from *Illicium verum* (Asif et al., 2016; Peng et al., 2016; Wang et al., 2011), while there were rare studies about the components changes during cooking and processing. The aim of this study was to investigate the changes of flavor substances

in *Illicium verum* during direct and frying heating processes, analyze the scores of corresponding sensory assessments and evaluate their possible applications in food industry. These results will provide a theoretical basis for further development of *Illicium verum*.

2. Materials and methods

2.1. Materials

Illicium verum was purchased from a local supermarket and identified by Professor Ping Xu of Xinxiang Medical University. A voucher specimen (No. ZWB-1) was deposited in the central laboratory of Xinxiang Medical University. The SPME manual fiber assembly (carboxen/polydimethylsiloxane, 75 μm) and SPME fibers (divinylbenzene/carboxen/polydimethylsiloxane, 50 μm) were purchased from Supelco Inc (Bellefonte, PA, USA). Other chemicals and agents were of analytical grade.

2.2. Aroma components preparation

Pulverized *Illicium verum* (5 g) was placed in a round-bottomed flask (100 mL) above a heating oven, and the temperature was controlled at 20, 40, 60, 80, 100, 120, 140, 160, 180 °C. Then, the aroma components during different direct heating temperatures were collected in a sample bottle (10 mL) via nitrogen (N₂) purging. The samples were gathered

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for 3 min and evaluated by gas chromatography–mass spectroscopy (GC–MS) and gas chromatography olfactometry (GC–O) analysis.

The aroma components during frying heating process were acquired by mixing 10 mL soybean oil with *Illicium verum* in that round-bottomed flask (100 mL) and controlling the temperature at 40, 60, 80, 100, 120, 140, 160 °C.

2.3. GC–MS and GC–O analyses

Aroma components of *Illicium verum* after heat treatment were detected on an Agilent 6890N GC-5973iMS GC–MS system (HP Inc, USA) equipped with a flame ionization detector and a fused silica capillary column HP-5MS (30 m × 0.25 mm ID, 0.25 μm film thickness). Helium was used as carrier gas at a flow rate of 1.0 mL/min. Injection port temperature was 250 °C. Column temperature program was as follow: 50 °C (2 min) isotherm, increased to 280 °C at a rate of 10 °C min⁻¹ and held at 280 °C for 30 min. The mass spectrometer was operated in the electron impact ionization mode (+EI, 70 eV). The Agilent 7980 gas chromatograph interfaced to an Alphamos sniffer device (Alpha-Mos Corporation, France) was employed for GC–O analysis. Aroma components of *Illicium verum*. The split ratio of the effluent into the Alpha-Mos sniffer was 10:1, and mass spectra was recorded in the range of *m/z* 30–500 amu under full scan acquisition mode. The collected compounds were identified by GC–MS comparing with retention index (RI) and aromaticity of standard compounds via searching NIST02.1 database of the MSDChem workstation.

2.4. Sensory assessment of *Illicium verum*

Several characteristic aromas were exhibited to assess the aroma components of heat-treated *Illicium verum* which was confirmed by Professor Xu and a panel of inspectors in our group by visual and chemical identification according to Chinese Pharmacopoeia. Sweet aroma (30%), alcoholic aroma (20%), fruity aroma (20%), woody aroma (20%) and caramelized aroma (10%) were chosen as standards for grading and evaluating *Illicium verum* (Zhang et al., 2015). Total score of samples achieving 80 or more was qualified. According to the quantitative description analysis (QDA) sensory assessment rules, five or more inspectors were involved in the evaluation (Table 1).

2.5. Statistical analysis

Results were analyzed using analysis of variance, and standard error was calculated accordingly.

3. Results and discussion

3.1. Changes of aroma components during direct heating process

The strength of a substance’s odour depends on the threshold and absolute mass of the material, and stronger smells would appear when lower threshold value exists under a certain absolute mass. As shown in Fig. 1, the total amounts of volatile components in *Illicium verum* were increased with the rise of direct heating temperature, which was consistent with the daily observation.

Trans-anethole is the major flavor compound in *Illicium verum* and

Table 1
Quantitative criteria of aroma components of *Illicium verum*.

Overall normalized score	Poor	Fair	Good	Standard samples
Sweet aroma	0–9	10–19	20–29	30
Alcoholic aroma	0–11	12–15	16–19	20
Fruity aroma	0–11	12–15	16–19	20
Woody aroma	0–11	12–15	16–19	20
Caramelized aroma	0–5	6–7	8–9	10

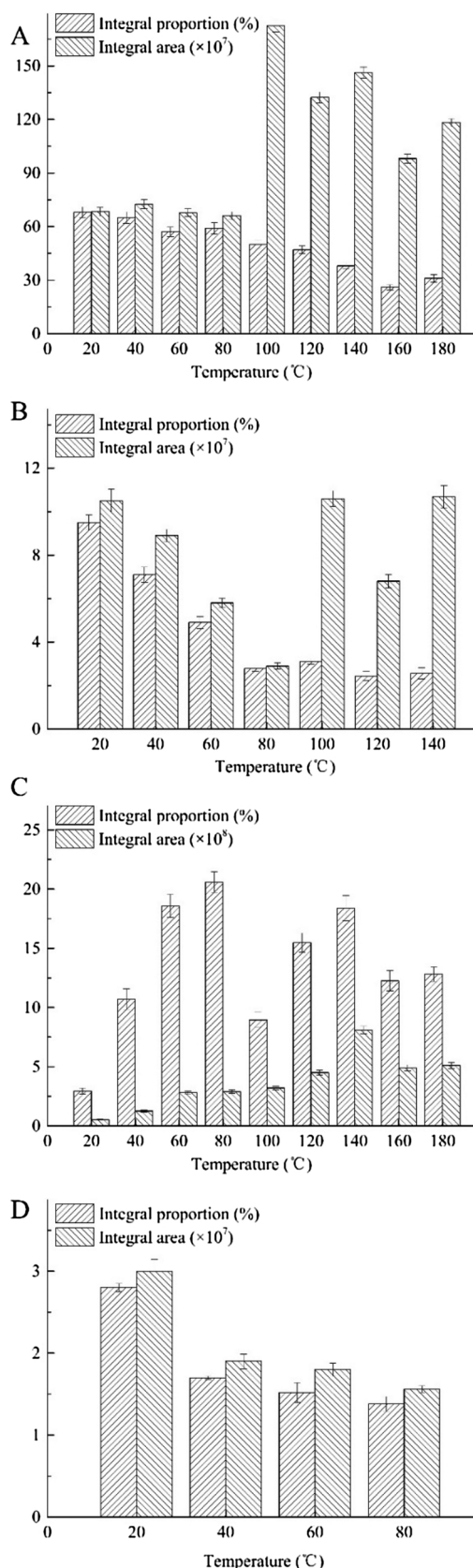


Fig. 1. Changes of aroma components during direct heating process. A: trans-anethole; B: estragole; C: allyl phenol isoprene; D: linalool.

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