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SHORT COMMUNICATION

Volatile components of *Rhizoma Alpiniae Officinarum* using three different extraction methods combined with gas chromatography–mass spectrometry

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KEYWORDS

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Volatile components;
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Headspace solid-phase microextraction;
Diethyl ether extraction;
Gas chromatography–mass spectrometry

Abstract Volatile components from *Rhizoma Alpiniae Officinarum* were respectively extracted by three methods including hydrodistillation, headspace solid-phase microextraction (HS-SPME) and diethyl ether extraction. A total of 40 (hydrodistillation), 32 (HS-SPME) and 37 (diethyl ether extraction) compounds were respectively identified by gas chromatography–mass spectrometry (GC/MS) and 22 compounds were overlapped, including α-farnesene, γ-muurolene, 2,6-dimethyl-6-(4-methyl-3-pentenyl)bicyclo[3.1.1]hept-2-ene, eucalyptol and cadina-1(10), 4-diene and so forth, varying in relative contents. HS-SPME is fast, sample saving and solvent-free and it also can achieve similar profiles as those from hydrodistillation and solvent extraction. Therefore, it can be the priority for extracting volatile components from medicinal plants.

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1. Introdution

Rhizoma Alpiniae Officinarum (RAO), the dry root and rhizome of *Alpinia officinarum* Hance, is a traditional Chinese medicine (TCM) mainly distributed in southern China [1]. RAO has long been used in practice for its antioxidation, antidiabetic, anti-ulcer, anti-diarrhea, antiemetic, analgesia, anti-inflammatory and anti-coagulation effects [2,3]. Flavonoids, volatile components and diarylheptanoids are reported as the main constituents of RAO and volatile components contribute a lot to those bioactivities

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[4,5]. Consequently, identifying and determining its volatile components make sense for quality control of the crude material.

Volatile components of TCMs can be extracted by many technologies including hydrodistillation, headspace solid-phase microextraction (HS-SPME) and solvent extraction [6]. Among of them, HS-SPME, a new sample pretreatment technique, was invented by Pawliszyn (University of Waterloo, Canada). Typically, the analytes are extracted from a sample adsorption on a thin polymer coating fiber inside an injection needle [7–9]. It is usually combined with gas chromatography–mass spectrometry (GC/MS) to analyze volatile components in natural products and foods [10].

In this paper, three sampling methods coupled with GC/MS, i.e., hydrodistillation, HS-SPME and diethyl ether extraction were compared and used for analysis of volatile components of RAO.

2. Experimental

2.1. Materials and chemicals

The dry RAO (Xuwen, Guangdong province, China) was purchased from Cai Zhi Lin pharmacy (Guangzhou, China) and authenticated by Dr. Xin-Jun Xu, Sun Yat-Sen University. It was ground to fine particles with the size about 40 mesh for follow-up pretreatments. Anhydrous sodium sulfate (Guangzhou Chemical Reagent Factory, Guangzhou, China), diethyl ether and *n*-hexane (Damao Chemical Reagents Works, Tianjin, China) were analytical pure.

2.2. Hydrodistillation procedure

About 15 g of the powder was weighed and suspended in 300 mL of water to collect the volatile oil by hydrodistillation for 4 h according to Appendix X D of Chinese Pharmacopoeia (2010, vol. 1) [11]. The volatile oil obtained was dried over anhydrous sodium sulfate and diluted with 4 mL of *n*-hexane. The solution was filtered through a 0.22 μm membrane filter and 1 μL was injected into the GC/MS port for analysis.

2.3. HS-SPME procedure

An HS-SPME holder and a coating fiber with a 50/30 μm layer of divinylbenzene/carboxen/polydimethylsiloxane (DVB/CAR/PDMS) were purchased from Supelco (Bellefonte, Pennsylvania, USA). The fiber was conditioned prior to use by insertion to the GC injection port at 250 °C for 0.5 h. 0.5 g of the powder was weighed and introduced into a 20 mL flat bottom headspace vial which was then sealed with gray butyl headspace stopper and 20 mm unlined crimp cap using a crimper. The needle of SPME holder was injected to the vial and the fiber was pushed out and exposed to the headspace for the absorption of volatile components, with the vial heated and maintained at 80 °C for 40 min. Finally, the fiber was removed from the vial and analytes were desorbed by exposing the fiber to the GC/MS injection port at 250 °C for 2 min.

2.4. Diethyl ether extraction

About 5 g of the powder was weighed and extracted with diethyl ether (1:8, w/v) for three times (15 min each time) using ultrasonic-assisted technology. The obtained turbid solution was filtrated and then the solvents in the filtrate were removed to obtain extractum by rotary evaporation under reduced pressure at 30 °C. Afterward, the extractum was diluted in *n*-hexane and filtered through a 0.22 μm membrane filter. One microliter of subsequent filtrate was analyzed by GC/MS.

2.5. GC/MS conditions

GC/MS (Thermo Electron Corporation, USA) instrument was Finnigan Trace DSQ with an electron impact (EI) ion source

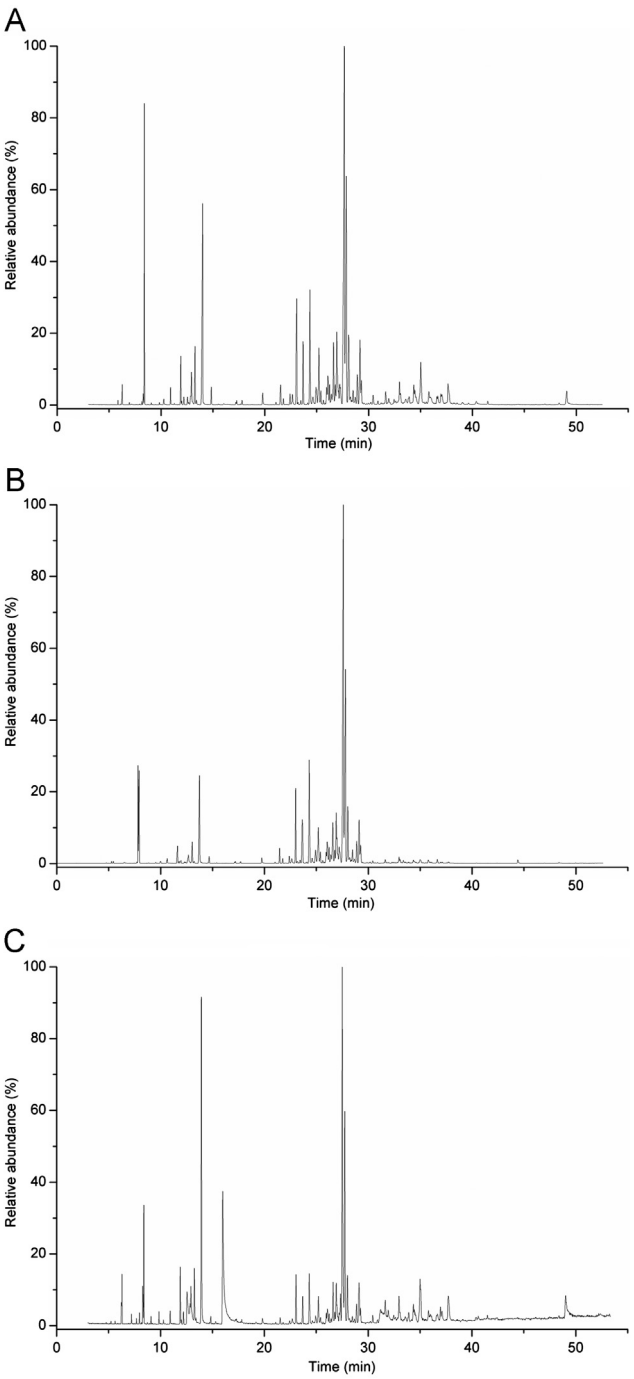

Fig. 1 GC/MS total ion chromatograms of *Rhizoma Alpiniae Officinarum* by (A) hydrodistillation, (B) HS-SPME and (C) diethyl ether extraction.

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