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Flash Evaporation and Headspace Solid-phase Microextraction for the Analysis of the Essential Oils in Traditional Chinese Medicine, *Houttuynia Cordata Thunb*

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Abstract: We have investigated the use of flash evaporation, headspace solid-phase microextraction (HS-SPME) and steam distillation (SD) as sample concentration and preparation techniques for the analysis of volatile constituents present in *Houttuynia cordata Thunb*. The samples were analyzed by gas chromatography (GC) and identified by mass spectrometry (MS). Comparison studies were performed. It was found that the results obtained between Headspace solid-phase microextraction HS-SPME and SD techniques were in good agreement. Seventy-nine compounds in *Houttuynia cordata Thunb* were identified by MS. In flash evaporation, thirty-nine compounds were identified. Discrimination in the response for many constituents studied was not observed, which can be clearly observed in SD and HS-SPME techniques. As a conclusion, HS-SPME is a powerful tool for determining the volatile constitutes present in the *Houttuynia cordata.*

Keywords: Headspace solid-phase microextraction, flash evaporation, essential oils, GC-MS.

Houttuynia cordata Thunb is a traditional Chinese medicinal herb, which has been used as a disinfector, an antipyrotic and a diuretic. Stream distillation combined with GC-MS are used as the routine methods for the analysis of the volatile essential oils[1]. However, they are complex and time-consuming and require a relatively large amount of sample. In this work, headspace SPME and flash evaporation with GC-MS[2] were applied to the separation and identification of the essential oils in *Houttuynia cordata Thunb*. To demonstrate the validation of the two techniques, the results were compared with the SD results. It was found that the results obtained between HS-SPME and SD techniques were in good agreement. In flash evaporation, discrimination in the response for many constituents studied was not observed, which can be clearly observed in SD and HS-SPME techniques. As a conclusion, HS-SPME is suitable for the analysis of volatile constitutes in *Houttuynia cordata Thunb*.

Experimental

Samples of *Houttuynia cordata Thunb* were obtained from Jiangxi Province, China. The manual SPME holder was used with a 100 μm polydimethylsiloxane fibre assembly

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(Supelco, Bellefonte, USA). HS-SPME sampling was as follows: *Houttuynia cordata Thunb* samples were first ground to powder and was sealed in a vial, then SPME fibre was suspended in the HS and equilibrated for 30 min at 100℃ before injection.

GC–MS analysis was carried out on a GCMS-QP5050 (Shimazhu, Japan). After sampling, the HS-SPME device was inserted into the GC injector, chromatographic conditions: injector temperature: 240℃; column temperature programme: from 80℃ to 240℃ at 3℃/min; carrier gas: helium at a flow-rate of 1 mL/min. The electron impact ionization conditions were: ion energy 70 eV, and the mass range scanned was 50-400 a.m.u. in the full-scan acquisition mode.

The flash evaporation experiments were conducted in a micro furnace pyrolyzer (CZ-100, BIT Analytical Instrumental Factory, Beijing, China). About 0.5 mg of a powdered *Houttuynia cordata Thunb* sample taken in a platinum sample cup was introduced into the heated center of the pyrolyzer, and pyrolyzed for 5 s at 250℃ under the flow of helium carrier gas. The relatively low temperature of 250℃ was used to avoid evaporation of the sample at higher temperatures. GC-MS conditions were the same as described above.

The essential oil was prepared according to the Chinese Pharmacopoeia[3], the obtained essential oil was dried over anhydrous sodium sulfate and stored at 4℃ until analysis. GC-MS analysis was carried out under the same conditions as described above.

Results and Discussion

In HS-SPME, the volatile compounds of the *Houttuynia cordata Thunb* were extracted under different conditions (temperature and extraction time). The sum of the peak areas under different conditions was used for the determination of the optimum extraction conditions. The result indicated that 100℃ and 30 min were the optimum conditions. In flash evaporation, the experiments were carried out between 100 and 900℃. The sum of the peak areas at different temperatures was used for the determination of the optimum extraction conditions. The results showed that 250℃ was the optimum temperature.

The chromatogram obtained by HS-SPME-GC-MS is shown in **Figure 1**. The relative contents were calculated by the peak area ratio. Seventy-nine compounds were identified. The main compounds included 2-undecanone, *n*-hexadecanoic acid, dodecanoic acid, caryophyllene, caryophyllene oxide, phytol, and decanal. In flash evaporation GC-MS, thirty-nine compounds were identified. The chromatogram of the sample is shown in **Figure 2**. Two principal compounds, houttuynum and 2-undecanone, were detected at concentrations of 7.23% and 22.21% in the HS-SPME extracts, of 3.60%, 3.59% in flash evaporation extracts, and 6.60%, 25.93% in SD extracts. The concentration of houttuynum was higher than that in SD and flash evaporation.

Figure1 The chromatogram of the volatile constituents of *Houttuynia cordata Thunb* by SPME-GC-MS under the extraction condition of 100℃and 30 min

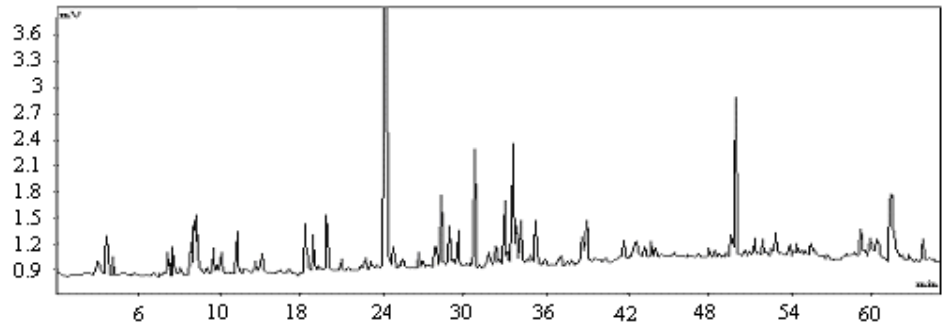


Figure 2 The chromatogram of the volatile constituents of *Houttuynia cordata Thunb* by FE-GC-MS under the extraction condition of 250℃ and 5s

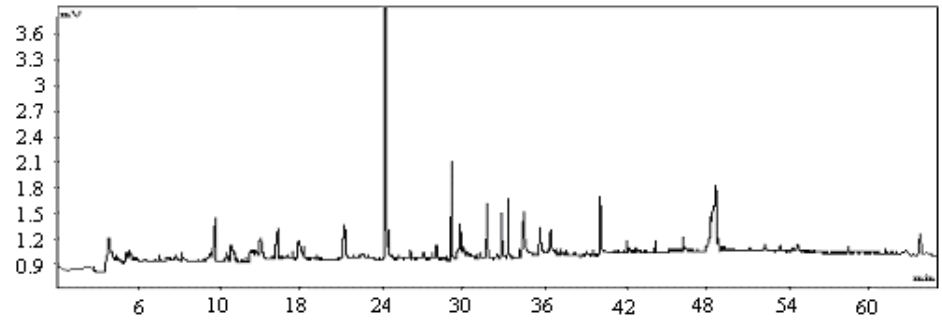
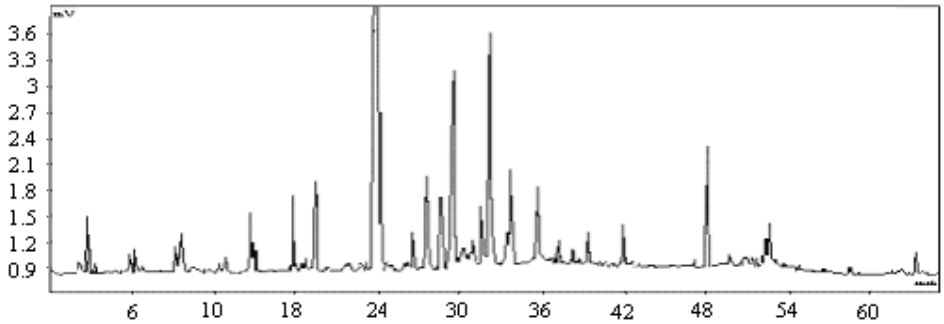


Figure 3 The chromatogram of the volatile constituents of *Houttuynia cordata Thunb* by SD/GC-MS



To demonstrate its feasibility, HS-SPME was compared to SD for determination of the volatile compounds present in *Houttuynia cordata Thunb*. **Figure 3** is the chromatogram of the volatile oil of *Houttuynia cordata Thunb* obtained by SD method. The results in **Figure 3** and **Figure 1** showed that the results detected by SD method were in good agreement with that of HS-SPME method. So the latter method is a very suitable one for the determination of volatile constituents in the TCMs. A series of five

consecutive extractions were performed in order to evaluate the repeatability of the HS-SPME method. The precision of the HS-SPME method was very good and the relative standard deviations (%RSD) were below 5% for all the compounds.

Conclusion

HS-SPME has been shown to be a very suitable methodology for the determination of volatile constituents present in *Houttuynia cordata Thunb*. RSD values were less than 5%, showing the method has a very good repeatability, and sample preparation is simple for this method.

Acknowledgments

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