

Determination of essential oil in a traditional Chinese medicine, *Fructus amomi* by pressurized hot water extraction followed by liquid-phase microextraction and gas chromatography–mass spectrometry

Chunhui Deng¹, Ning Yao, Aiqin Wang, Xiangmin Zhang*

Department of Chemistry, Fudan University, Handan Road, No. 220, Shanghai 200433, China

Received 10 November 2004; received in revised form 16 December 2004; accepted 16 December 2004

Available online 2 February 2005

Abstract

In this paper, for the first time, two sample techniques of pressurized hot water extraction (PHWE) and liquid-phase microextraction (LPME) were combined and applied to the analysis of essential oil in a traditional Chinese medicine (TCM), *Fructus amomi*. Essential oil in *F. amomi* samples (50.0 mg) were extracted by PHWE equipment in dynamic mode, followed by extraction and concentration with headspace (HS)-LPME and detection by gas chromatography–mass spectrometry (GC–MS). The PHWE and HS-LPME parameters were optimized and the method repeatability was studied. The three active compounds of camphor, borneol and borneol acetate in the *F. amomi* sample from five different growing areas were quantitatively analyzed by internal standard method. Compared to steam distillation (SD), the proposed method required simple sample preparation, little sample mass and whole analysis time less than 40 min. The present method provided good repeatability (R.S.D. <12.0%). It has been demonstrated that PHWE–LPME–GC–MS is a simple, rapid and low-cost method for determination of essential oils in TCMs and is a potential tool for TCM quality assessment.

© 2004 Elsevier B.V. All rights reserved.

Keywords: Pressurized hot water extraction; Liquid-phase microextraction; Essential oils; Extraction method; Traditional Chinese medicine; *Fructus amomi*

1. Introduction

Traditional Chinese medicines (TCMs) have played an important role in clinical therapy because of their high pharmacological activity, low toxicity and rare complication [1]. In recent years, more and more interests have been re-attracted in this field and many studies focus on TCM modernization. Different natural conditions including soil and climate lead to discrepancy in TCM quality, therefore, quality assessment of TCM is an important subject. For many TCMs such as *Flos chrysanthemi indici* and *Acorus tatarinowii* Schot., there are active components in their essential oils. For these TCMs,

quality assessment can be performed by quantitative analysis of the active components. Several routine sample techniques such as solvent extraction and steam distillation combined with gas chromatography–mass spectrometry (GC–MS) were used for the analyses of TCM essential oils and used as the tools of TCM quality assessment. These conventional techniques have some shortcomings such as requirement of long preparation time and large amount of organic solvents. The disadvantages have led to the consideration of the use of supercritical fluids extraction. Supercritical CO₂ extraction technique was developed for plant essential oil extraction processes [2–7] and also used as a tool of TCM quality assessment [8,9]. However, it required expensive equipment. Moreover, the emissions of carbon dioxide are friendless to environment. In 1990, Pawliszyn introduced solid-phase microextraction (SPME) with the advantages of simplicity, rapidness and no need of solvent [10]. Recently,

* Corresponding author. Fax: +86 21 65641740.

E-mail addresses: dengch_fudan@126.com (C. Deng), xmzhang@fudan.edu.cn (X. Zhang).

¹ Tel.: +86 21 65643983.

it was developed for the extraction of essential oils in plant materials [11,12]. In our previous studies, SPME technique was developed for the determination of TCM essential oils [13–15]. As we know, it is very difficult to quantitatively analyze the volatile compounds in plant materials by direct SPME.

More recently, pressurized hot water extraction (PHWE), a sample extraction technique based on the use of water as an extractant in dynamic mode, at temperatures between 100 and 374 °C (critical point of water, 221 bar and 374 °C) and a pressure high enough to maintain the liquid state, is emerging as a powerful alternative for solid sample extraction [16,17]. The major advantages of PHWE are the low-cost and environmental friendliness of water. Furthermore, the solvating properties of water are easily altered through change in temperature and pressure. PHWE has been under intensive study for the analysis of solid samples. It has been applied to the analyses of alkylbenzenes, PAHs and PCBs residues in soils and environmental solids, pesticide residues in soils, plant material and food [18–21]. The technique has also been used for the analyses of essential oils in plant materials [22–25].

Since PHWE is a dynamic extraction method, the aqueous extract (about 5–50 ml) has to be extracted and concentrated before analysis. Liquid–liquid extraction (LLE) [26], solid-phase extraction (SPE) [27,28] and SPME [29,30] were developed for this purpose. To collect the analytes in PHWE extract, LLE requires large amounts of solvents and SPE needs expensive adsorbents. Moreover, both LLE and SPE are time-consuming procedures. Due to SPME with the advantages of extraction and simultaneous concentration, SPME has been shown to be a better approach to the determination of the analytes in PHWE extracts than LLE and SFE [29–31]. However, the disadvantages are that SPME fiber is still relatively expensive and polymer coating is fragile and easily broken. Furthermore, sample carryover is sometimes difficult or impossible to be eliminated. Therefore, a simple, rapid and low-cost sample concentration technique for extraction and concentration of PHWE aqueous extracts is desired.

In 1996, liquid-phase microextraction (LPME) was introduced by Jeannot et al. [32–34]. This relatively new technique is performed by suspending 1 µl drop of organic solvent on the tip of either a Teflon rod or the needle tip of a microsyringe immersed in the stirred aqueous solution. The analytes partition between the bulk aqueous phase and the organic solvent microdrop. The application of LPME to environmental analysis and drug analysis has been described in several papers [35–39]. Jeannot and Cantwell introduced headspace liquid-phase microextraction (HS-LPME) and demonstrated the feasibility of the application of microdrop headspace mode to preconcentration of volatile organic compounds (VOCs) in an aqueous matrix [40]. In the HS-LPME mode, extraction solvent with high-boiling point and low vapor pressure was required. In order to overcome this limit, Shen and Lee developed a novel HS-LPME mode, where the thin organic sol-

vent film (OSF) formed in a microsyringe barrel through the movement of the plunger was used as an extraction interface [41].

In this work, PHWE combined with HS-LPME was developed for GC–MS analysis of TCM essential oil. *Fructus amomi*, the dried ripe fruit of *Amomum villosum* Lour. is a common TCM. Its main active ingredient—essential oil is often used to eliminate damp and improve appetite, to warm the spleen and check diarrhea, and to prevent abortion [42]. In the present work, the essential oil in *F. amomi* was extracted by PHWE, followed by extraction and concentration with HS-LPME and detection by GC–MS. The PHWE and LPME parameters were studied. The proposed method was applied to quantitative analysis of three active compounds (camphor, borneol, borneol acetate) in *F. amomi* samples from different growing areas.

2. Experimental

2.1. PHWE instruments

PHWE was performed using the following assembly (Fig. 1): a Shimadzu LC10AD pump was used to propel the water used as extractant through the system. An extractor (a prototype designed and patented by Salvador and Merchan [43]), consisting of a stainless steel cylindrical extraction chamber (8 cm × 3 mm i.d.), closed with screws at either end that permit the circulation of the leaching fluid through them, was used. The screw caps also contain stainless steel filter plates (2 µm in thickness and 0.635 cm i.d.) to ensure that the plant material remains in the extraction chamber. This chamber, together with a stainless steel pre-heater, is located in an oven, designed to work up to 300 °C and controlled using a Toho TC-22 temperature controller. A cooler system (consisting of a coil coupled to an Ultraterm 6000383 P-Selecta recirculation bath) was used to cool the fluid from the oven to a constant temperature close to 25 °C, thus avoiding the losses of volatiles caused by the hot water. The outlet of this coil was coupled to a stainless steel variable restrictor that was used to control the pressure in the system in order to maintain the extractant water in liquid state.

2.2. Materials

The dried ripe fruit of *A. villosum* Lour., *F. amomi* from five growing areas (Guangdong, Yunnan, Hainan, Jiangxi and Fujian, China) was purchased from a Chinese herb shop, Shanghai, China. *F. amomi* samples were stored in the dark at 4 °C till used. The samples were ground to fine powder. About 0.050 g *F. amomi* was used for pressurized hot water extraction, and 50 g was used for steam distillation.

Camphor, borneol, borneol acetate and menthol standards were all provided by the National Institute for the Control of Pharmaceuticals and Biological Products, Beijing, China.

Download English Version:

<https://daneshyari.com/en/article/9743845>

Download Persian Version:

<https://daneshyari.com/article/9743845>

[Daneshyari.com](https://daneshyari.com)