Journal of Pharmaceutical Analysis 2011;1(3):213–218

Xi'an Jiaotong University

Journal of Pharmaceutical Analysis

www.elsevier.com/locate/jpa
www.sciencedirect.com

ORIGINAL ARTICLE

Rapid determination of volatile constituents in safflower from Xinjiang and Henan by ultrasonic-assisted solvent extraction and GC–MS

Ling-Han Jia[a], **Yi Liu**[b,*], **Yu-Zhen Li**[b]

[a]*School of Pharmacy, China Pharmaceutical University, Nanjing 211198, China*
[b]*Department of Pharmacy, Peking University People's Hospital, Beijing 100044, China*

Received 5 March 2011; accepted 11 April 2011
Available online 22 July 2011

KEYWORDS

Safflower;
Ultrasonic solvent extraction;
Gas chromatography–mass spectrometry (GC–MS)

Abstract The total volatile components were extracted from safflower by ultrasonic-assisted solvent extraction (USE) and their chemical constituents were analyzed by gas chromatography–mass spectrometry (GC–MS) to provide scientific basis for the quality control of safflower. Five different solvents (diethyl ether, ethanol, ethyl acetate, dichloromethane and acetone) were used and compared in terms of number of volatile components extracted and the peak areas of these components in TIC. The results showed that USE could be used as an efficient and rapid method for extracting the volatile components from safflower. It also could be found that the number of components in the TIC of ethyl acetate extract was more than that in the TIC of other solvent ones. Meanwhile, the volatile components of safflower from Xinjiang Autonomous Region and Henan Province of China were different in chemical components and relative contents. It could be concluded that both the extraction solvents and geographical origin of safflower are responsible for these differences. The experimental results also indicated that USE/GC–MS is a simple, rapid and effective method to analyze the volatile oil components of safflower.

*Corresponding author.
E-mail address: liuyipku@126.com (Y. Liu).

Peer review under responsibility of Xi'an Jiaotong University.
doi:10.1016/j.jpha.2011.04.002

Production and hosting by Elsevier

1. Introduction

Safflower (*Carthamus tinctorius L.*) belongs to the family Compositae. It is a perennial, broad leaf oil seed and medicinal crop [1] and is widely cultivated in agricultural production system of Asia, Europe, Australia and America as a source of high-quality vegetable and industrial oil [2]. In many oriental countries, safflower is used as a food colorant, dye and herbal medicine all the time [3]. There is a great quantity of polyunsaturated fatty acids in safflower edible oil cultivars. Therefore, it is also used as a feed for livestock [4]. Safflower seeds have been clinically used in Korea as herbal medicine to promote bone formation and prevent osteoporosis for a long time. There are some studies on anti-oxidative compounds from safflower, describing their activity in scavenging free radical species [5], and some researches also indicated that the major safflower seed antioxidants, i.e. serotonin derivatives, could inhibit low-density lipoprotein (LDL) oxidation and atherosclerosis in apolipoprotein E-deficient mice [6–10].

Essential oils are one of the most important compounds in traditional Chinese medicines (TCMs). Many technologies can be used to extract volatile components of TCMs, such as hydro-distillation (HD), micro-simultaneous distillation extraction (MSDE) [11,12], vacuum headspace (VHS) [12], supercritical fluid extraction (SFE) [13,14], thermal desorption (TD) [15,16], extraction with organic solvents [17,18], solid-phase micro-extraction (SPME) [19] and ultrasonic-assisted solvent extraction (USE) [20]. Among these methods, USE has more advantages, for example, high extraction efficiency, low equipment cost and ease operation, little or no sample preparation and low extraction temperatures. GC–MS is one of the most widespread analytical techniques in many scientific fields owing to its high sensitivity, low detection limit, rapid identification and being capable of simultaneously analyzing a number of ingredients in analytes; therefore, it is the best appropriate to analyzing the volatile components of safflower.

In the present study, USE was used to extract the volatile components from safflower using five different solvents (diethyl ether, ethanol, ethyl acetate, dichloromethane and acetone), and then these volatile components were identified by GC–MS technique. And also the differences in flavor composition among safflower samples from Xinjiang and Henan of China were investigated.

2. Experimental

2.1. Materials

Sample no. 1 was safflower from Xinjiang Autonomous Region and sample no. 2 was from Henan Province of China. The samples were ground and sieved into particles of 0.25 mm in diameter, and then they were dried at 60 °C for 6 h before use. The organic solvents used in the experiment included diethyl ether, ethanol, ethyl acetate, dichloromethane and acetone, which were extra pure and obtained from Merck Co. (Darmschadt, Germany).

2.2. Extraction procedure

The volatile components of safflower were extracted by USE in an ultrasound cleaning bath as described in literature [21]. Safflower samples were pulverized to uniform powder. The headspace vial was charged with 0.2 g of safflower. 2 mL of different solvents (diethyl ether, ethanol, ethyl acetate, dichloromethane and acetone) were used as the extraction solvent. Sonication was held for 120 min. After sonication, the whole organic extract was cooled at 4 °C. Then the organic layer was introduced in centrifuge tube, and centrifuged at 3500 rpm for 3 min. The resulting extract was dried, concentrated under the low temperature in the water bath and stored at 4 °C in dark place before GC–MS analysis. The obtained average volatile oil yield varied from 4.35% to 13.85%. The sample of 1 μL was used for GC–MS analysis.

2.3. GC–MS analysis

GC–MS (Finnigan TRACE MASS) was used. Volatiles were separated using a capillary column (DB-5MS quartz capillary chromatographic column, 30 m × 0.25 μm × 0.25 mm). The carrier gas was high-purity helium at a constant flow of 1.0 mL/min and the volume of injection was 2.0 μL. The injector temperature was 270 °C. Oven temperature programming was as follows: initiated at 80 °C, held for 1 min, and then rose at 5.0 °C/min to 250 °C and held for 20 min. Ionization source temperature was kept at 200 °C. Mass spectra were taken at 70 eV and solvent delay 1.5 min. Mass range was in a range of m/z 35–650 amu.

2.4. Statistical treatment of the essential oil

Typical GC–MS total ion chromatograms (TIC) of the volatile oil fraction extracted from safflower samples are shown in Fig. 1. Fig. 1 shows the results of samples from Xinjiang (No. 1) and Henan (No. 2) extracted using diethyl ether, ethanol, ethyl acetate, dichloromethane and acetone. MS data were compared with those in the Xcalibur NIST library to identify the peaks of TIC and determine compositions of the volatile components. Meanwhile, the relative content of each volatile compound was calculated by a ratio of the peak area of each component to total area of all peaks in TIC. And these results are listed in Table 1.

3. Results and discussion

In recent years, USE has been used to isolate bioactive compounds from plant materials using organic solvents, for example, aroma compounds from aromatic plants and foods at room temperature, and volatile compounds from TCMs [20,22–24]. The USE method has many advantages as mentioned above, and specially can make sample matrix efficiently contact with solvent. Mechanical action, thermal action and acoustic cavitations all have direct effect on the efficiency of ultrasonic extraction [25]. But acoustic cavitation is the most significant factor. Owing to the action of ultrasound irradiation, micro-bubbles will be formed when the negative pressure is high enough. Once the bubbles are generated, they will grow during the period of negative pressure and will be compressed during the period of positive pressure. The expansion and compression actions can cause constant pulsating or violent collapsing of micro-bubbles. When the collapse occurs near solid surface it can cause shockwave that passes through the solvent. Therefore, it can damage the cell walls to facilitate the release of contents [26]. The main advantages of USE method are high extraction efficiency, low equipmental cost, ease operation, little or no sample preparation and low extraction temperature. Hence, ultrasonic assistance is used more and more in analytical chemistry, and applied to different steps in the analytical process, particularly in sample preparation [27,28].

3.1. Comparison of the extraction solvents

In order to compare the extraction ability of different solvents, the number of components extracted from safflower and the