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Ultrasonic extraction of ferulic acid from *Ligusticum chuanxiong* ☆

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Abstract

The extraction of ferulic acid, a pharmacologically active ingredient from the root of *Ligusticum chuanxiong*, with ultrasonic extraction was investigated. Percolation and supercritical fluid extraction (SFE) were employed to make comparisons with ultrasonic extraction. Three variables, which included the concentration of solvent, the ratio of solvent volume/sample (mL/g), and extraction time, were found to have a great influence on ultrasonic extraction. The optimum extraction was with pure ethanol with a solvent volume/sample ratio 8:1 (mL/g) and extraction time of 30 min. Under the optimum extraction conditions, the extraction yield could reach 8.8% which was higher than that using SFE with ethanol as co-solvent and a content of ferulic acid of 0.7%; both the yield and the content were higher than those obtained by percolation. Ultrasonic extraction significantly shortened the time required for the extraction process. Overall, ultrasonic extraction was shown to be highly efficient in the extraction of ferulic acid from *Ligusticum chuanxiong*.

Keywords: Ultrasonic extraction; *Ligusticum chuanxiong*; Ferulic acid; Percolation; Supercritical fluid extraction

1. Introduction

With better understanding of natural products, an increasing number of people have become interested in studying active natural products for use as medicines or food additives.

The root of *Ligusticum chuanxiong* Hort. has been used as a traditional Chinese medicine for thousands of years. It is mainly used for the treatment of headaches, theumatic arthralgia, menstrual disorders, swelling and pain due to traumatic injury, pricking pain in the chest and costal region, and coronary heart disease. The essential biologically active components of *Ligusticum chuanxiong* are volatile oil, cnidiumlactone, tetramethylpyrazine, and ferulic acid. Ferulic acid (4-hydroxy-3-methoxycinnamic acid) is one of the most important medical components possessing anti-oxidative properties by virtue of the phenolic hydroxyl group in its structure. In 1986, Yin *et al.* (1986) found that ferulic acid could inhibit malondialdehyde (MDA) produced from platelets, erythrocyte lyses induced by MDA, and hydroxyl radical and lipid peroxidation induced by H_2O_2 and $O_2^{\bullet-}$.

The conventional technique for extracting pharmacologically active ingredients including ferulic acid is the percolation method. Unfortunately, the percolation method has a number of shortcomings, including long extraction time, great consumption of solvents, and lowered extraction efficiency. Therefore, developing a fast and efficient extraction method has become an issue of concern in the pharmaceutical and food industries.

Ultrasound, the term used to describe sounds ranging from 20 kHz to 1 GHz, is usually generated by a transducer which converts mechanical or electrical energy into high frequency vibrations. Vinatoru (2001) and Romdhane and Gourdon (2002) reported that the enhancement of extraction efficiency of organic compounds using ultrasound is attributed to a phenomenon called cavitation produced in the solvent by the passage of an ultrasonic wave. They found that cavitation bubbles are produced and compressed during the application of ultrasound, allowing higher penetration of the solvent into the raw plant materials and intracellular products released by disrupting the cell walls. Albu *et al.* (2004) and Rostagno *et al.* (2003) reported that ultrasound has been shown to aid extraction in a number of plant materials by significantly reducing extraction time and increasing maximum extraction yield, respectively.

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Sun and Li (2006) studied the extraction of ferulic acid from *Ligusticum chuanxiong* by the conventional methods and by another technology, supercritical fluid extraction. In contrast, the extraction of ferulic acid from *Ligusticum chuanxiong* with ultrasonic solvent has never been reported. The purpose of the present study was to examine the extraction process of ferulic acid from *Ligusticum chuanxiong* with an ultrasonic method, and to compare the effects with percolation and SFE. Both the content of ferulic acid in extracts and the extraction yield were used as quality control indices, with the former being the major determinate.

2. Experimental

2.1. Materials and chemicals

Carbon dioxide (with purity of 99.9%) was used as a supercritical fluid. Methanol (HPLC grade), ethanol (AC grade), and glacial acetic acid (AC grade) were purchased from Tianjin Chemical Reagent Factory (Tianjin, China). Ferulic acid (standard sample) was obtained from the National Detection Institute of Pharmaceutical and Biological Products of China. All chemicals were used without further purification.

The root of *Ligusticum chuanxiong* was provided and identified by Tianjin Zhongxin Pharmaceutical Group Corporation Limited. *Ligusticum chuanxiong* was ground, and material of powder size between 20 and 80 mesh was used in this study.

2.2. Ultrasonic extraction

Ultrasonic extraction experiments were performed in an ultrasonic bath (23.5 cm × 13.3 cm × 10.2 cm) bought from Kunshan Ultrasound Co. Ltd. (Kunshan, China). The working frequency was 50 kHz and the bath power rating was 400 W on the scale of 0–10. The experiment temperature was 25 °C at the beginning. The extraction of ferulic acid was performed by adding powdered material (about 10 g) into selected volume solvent in a 150 mL tube. The tube was then partially immersed into the ultrasonic bath. The liquid in the tube was kept at the level of the water in the bath, and the latter was circulated and regulated at constant desired temperatures to avoid water temperature increase caused by ultrasonic exposure. After extraction, the extracts were filtered through filter paper and then the solvent was vaporized from the extracts using a vacuum rotary evaporator.

2.3. Supercritical fluid extraction

Supercritical CO_2 extraction was experimentally performed using the Speed SFE instrument (Applied Separations Inc., Allenton, PA, USA). Liquid CO_2 was pressurized with a high-pressure pump and then charged into the extraction column to the desired pressure. The extraction column was 32 mL with 14.40 mm inner diameter and 195 mm length, being packed with powdered sample (about 10 g) and glass beads. The extraction column was heated by an oven and its temperature was indicated and controlled by the thermocouple. The supercritical CO_2 with dissolved compounds passed through a heated micrometer valve and was subsequently expanded to ambient pressure. The flow rate was controlled at 3.0 mL/min (ambient temperature and pressure) in this study; the extracts were precipitated in a collecting vial at ambient pressure and temperature. When co-solvent was used, it was directly added into the powered materials and soaked for four hours before extracting. After the SFE process, the solvent was vaporized from the extracts using the rotary evaporator.

2.4. Percolation extraction

The percolation method described by Chinese Pharmacopoeia Commission of People's Republic of China (2005) was employed. Ethanol–water mixture was used as a solvent. Before percolation, the powdered *Ligusticum chuanxiong* was soaked with solvent for 24 h. The percolation flow rate was 15 drops/min and the percolation time was about eight hours. After percolation, the extracts were filtered through filter paper and the solvent was vaporized from the extracts using the rotary evaporator.

2.5. HPLC analysis for ferulic acid

HPLC was performed for analyzing ferulic acid with a P4000 pump, a Merck L-4200 UV–vis detector and LC-10 AT (YWG C_{18}, 5 μm, 250 mm × 4.6 mm i.d.) column. The flow rate was 1.0 mL/min and the oven temperature was 45 °C. The mobile phase consisted of methanol and water (1% HAc) (35:65, v/v). The injection volume was 10 μL. The detector was set at 320 nm. All quantitative analyses were made by the external standard method, using ferulic acid as an analytical standard.

3. Results and discussion

3.1. Optimization of ultrasonic extraction

3.1.1. Solvent selection

Solvent properties that affect the behavior of ultrasonic cavitations include vapor pressure, viscosity, and surface tension. Of these properties, medium vapor pressure is known to be the most conducive to ultrasound activity. Another important factor for the choice of solvent is its polarity as ferulic acid is a polar compound. Besides meeting above requirements, ethanol was also in compliance with good manufacturing practice (GMP). So ethanol was selected as the extraction solvent.

3.1.2. Effect of ethanol–water compositions

The effect of ethanol content in the solvent on the extraction obtained after 40 min at 25 °C is shown in Fig. 1, where *E* (extraction yield) and *C* (content of ferulic acid in extracts) are