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Original Article

Simultaneous determination of four amides in *Saururus chinensis* by matrix solid phase dispersion and high-performance liquid chromatography method

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ABSTRACT

A rapid and simple analytical method was established for the determination of four amides (*N-p-trans*-coumaroyltyramine, aristolactam AII, sauristolactam and aristolactam BII) in *Saururus chinensis* by matrix solid phase dispersion (MSPD) and high-performance liquid chromatography-diode array detector (HPLC-DAD). In the optimized MSPD, 0.2 g *S. chinensis* powder was blended with 0.4 g silica gel, and 5 mL methanol was selected as elution solvent. The MSPD extraction achieved higher extraction recovery of four amides, and required less sample, solvent and preparation time, comparing with the conventional methods (Soxhlet and ultrasonic extraction). The assay was performed on a TSK gel ODS-100Z column (4.6 mm × 250 mm, 5 μm) at 30 °C. Acetonitrile and 0.4% acetic acid aqueous solution was used as mobile phase by gradient elution at the flow rate of 1.0 mL/min. The detection wavelength was 280 nm. All the analytes showed good linear regression ($R^2 \geq 0.9998$) within the concentration ranges. The validated method showed good precision and stability with relative standard deviations (RSDs) ≤ 3.18%. The recoveries were in the range of 96.57–99.65%, with RSDs less than 2.74%.

1. Introduction

Saururus chinensis, a well known folk medicine in China and Southern Korea, has been widely used for the treatment of edema, jaundice, gonorrhea and several inflammatory diseases. Meanwhile, *S. chinensis* was also used as functional food and food supplementation [1,2]. A great many lignans, flavonoids, aristolactams and phenolic acids have been isolated from its aerial parts and rhizome [3–5]. Aristolactams (ALs) showed significant neuroprotective activity against glutamate-induced toxicity in primary cultured rat cortical cells [6], antioxidant [7] and anti-tumor activities [8,9]. It is interesting to note that aristolochic acids (AAs), known to be

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nephrotoxic, carcinogenic and mutagenic [10], can metabolize to ALs in the rat, in addition, AL-I showed higher nephrotoxicity than AA-I [11]. Hence, many countries have banned the use of herbs containing AAs and ALs. The U.S. Food and Drug Administration declared to discontinuation of products that contain AAs and ALs [12]. *N-p-trans*-coumaroyltyramine, the other amide, exhibited significant alpha-glucosidase inhibitory [13], antioxidative and anti-inflammatory activities [14]. Therefore, there was an urgent need to establish determination and quantitative analysis of ALs in *S. chinensis* both for its safety and efficacy. However, to our knowledge, there was little report about determination of ALs in *S. chinensis*, excepting our previous research [15].

In addition, the conventional extraction methods including Soxhlet and sonication were time-consuming, high solvent consumption, requiring additional filtration, concentration or even purification steps. Matrix solid phase dispersion (MSPD) technology, invented by Barker in 1989, was a simple, convenient and low-cost extraction method [16]. Recently, MSPD has been used widely for the extraction of investigated compounds from foods and medicinal plants [17–19]. Some active compounds have been extracted from plants by MSPD, such as flavonoids [19–21], saponins [22], lignans [23], phenols [24,25] and essential oils [26]. However, as far as we know, this technique has never been applied to the extraction of amides from *S. chinensis*.

Thus, in the present study, a method based on MSPD extraction combined with high-performance liquid chromatography-diode array detector (HPLC-DAD) was established and systematically validated for determination of four amides, namely, *N-p-trans*-coumaroyltyramine, aristolactam AII, sauristolactam and aristolactam BII in *S. chinensis*. The MSPD method was compared with the traditional Soxhlet and sonication extraction methods. The validated MSPD-HPLC method was used to determine the four amides in *S. chinensis* from different regions.

2. Materials and methods

2.1. Chemicals, reagents and materials

Standard compounds (*N-p-trans*-coumaroyltyramine, aristolactam AII, sauristolactam and aristolactam BII) were isolated from the aerial parts of *S. chinensis*. Their structures were elucidated based on spectroscopic analysis (^{1}HNMR, ^{13}CNMR) and literatures [27–29]. The purity of each compound was more than 98% detected by HPLC. Their chemical structures were shown in Fig. 1. Six samples of *S. chinensis* (S1–S6) were collected from Jiangsu, Zhejiang, Anhui and Hubei province. The botanical origin of materials was identified by Prof. Jianwei Chen. Voucher specimens were deposited at Herbarium of Nanjing University of Chinese Medicine.

Acetonitrile (chromatographic grade) was purchased from Merck (Darmstadt, Germany). Deionized water was purified by a Milli-Q water system (Millipore, USA). Silica gel (200–300 mesh) was purchased from Qingdao Haiyang Chemical Subsidiary Factory (Qingdao, China). Diatomaceous earth was obtained from Hengxing Technology Inc. (Tianjin, China). C_{18} (particle size 40–63 μm) was purchased from SiliCycle (Quebec, Canada). Neutral alumina (100–200 mesh) was purchased from Tianjin Chemical agent Co. Ltd. (Tianjin, China). Other reagent solution was of analytical grade from Beijing Reagent Company (Beijing, China).

2.2. Chromatographic conditions and instrument

The analysis were performed on an Agilent 1200 liquid chromatography system (Agilent Technologies, USA), equipped with a G1315D DAD detector, a G1312A double pump, a G1329A autosampler and a G1316A column temperature controller. The separations were carried out on a TSK gel ODS-100Z column (4.6mm×250mm,5μm). The column temperature was 30°C and the detection wavelength was 280nm. The mobile phase, consisted of 0.4% (v/v) acetic acid-water solution (A) and acetonitrile (B), was programmed by the following linear gradient elution: 0–8min, 30% B (v/v); 8–14 min, 30–40% B (v/v); 14–30 min, 40–50% B (v/v); 30–40 min, 50–100% B (v/v); 40–42 min, 100% B (v/v); 42–44 min, 100–30%B(v/v). The flow rate was 1.0mL/min, and injection volume was 10μL.

2.3. Preparation of standard solutions

Stock standard solutions of *N-p-trans*-coumaroyltyramine (830.0 μg/mL), aristolactam AII (778.0 μg/mL), sauristolactam (632.0 μg/mL) and aristolactam BII (566.0 μg/mL), were prepared in methanol. Working standard solutions were obtained by diluting the mixed stock solutions with methanol to give six different concentrations for calibration curves. The solutions were filtered through a 0.45 μm membrane prior to injection.

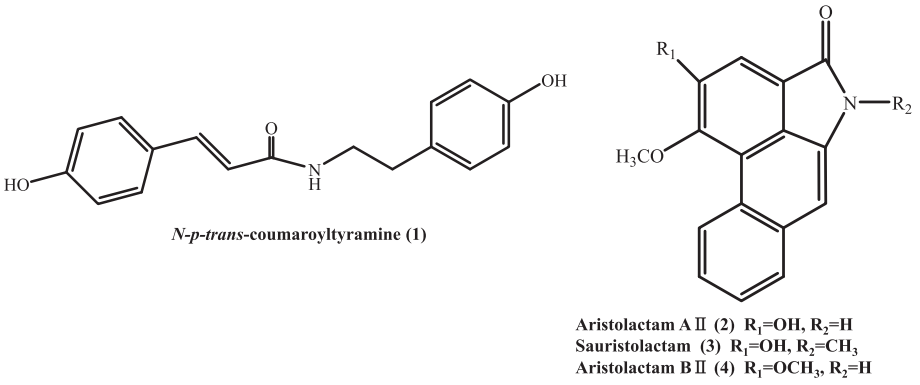

Fig. 1 – Chemical structures of the investigated amides in *Saururus chinensis*.

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