

Determination of Forsythin in Yinqiao Jiedu Pills by Capillary Electrophoresis

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Abstract. This paper developed the determination of forsythia content in Yinqiao Jiedu Pills by high performance capillary electrophoresis (HPCE) method. The borax solution was chosen as buffer solution, and its concentration was 10 mmol at a constant voltage of 20kV and injecting time of 10s. The content of forsythia in Yinqiao Jiedu Pills was 4.615 mg/g (RSD = 10.1%) (n = 6). This method is suitable for the detection of the content of forsythia in Yinqiao Jiedu Pills.

1. Introduction

Yinqiao Jiedu Pills are consists of honeysuckle flower, weeping forsythia capsule, peppermint, fine leaf schizonepeta herb, fermented soybean, great burdock achene, platy codon root, common lophatherum herb and liquoric root nine Chinese drug. It has the effect of eliminating headache and refreshing, clearing heat and detoxifying [1]. Zhu [2] established a method for the determination content of forsythia glycosides in Yinqiao Jiedu Tablets by HPLC. The HPLC was performed on C18 column (4.6 mm×250 mm, 5 μm) with acetonitrile-water (25:75) as mobile phase. The wavelength of UV detector was 277 nm. Zhong et al [3] established the HPLC determination method of chromogenic acid for developing the quality standard of Yinqiao Jiedu Oral Liquid. The HPLC separation was performed on an E. Merck Lichrospher 100RP-10 column (4 mm×250 mm, 5 μm). The mobile phase was composed of acetonitrile-0.4% phosphoric acid (10:100). The flow rate was 1.0 ml/min. The wavelength of UV detector was 327 nm. Song et al [4] established a method for determination of arctiin and forsythia in Yinqiao Jiedu Granules by HPLC. The analytical column C18 (4.6 mm×250 mm, 5 μm) was used. The mobile phase was composed of acetonitrile and water (27: 73) at a flow rate of 1.0 ml/min. The detection wavelength was 280 nm and column temperature was 30°C. Quan et al [5] established an HPLC method for the determination of chromogenic acid and arctiin in Yinqiao Jiedu Pills. The separation was performed on a Thermo ODS-2 Hyperil column (4.6 mm×250 mm, 5 μm), the mobile phase was composed of acetonitrile-0.1% phosphoric acid with gradient elution. The flow rate was 1.0 ml/min. The wavelength of UV detector was 280 nm and column temperature was 30°C. Zhang et al [6] established a high performance liquid chromatographic method for the determination of forsythoside An in Yinqiao Jiedu Tablets from different drug manufacturers. The HPLC method was performed on a Kromasil 100A C18 column (4.6 mm×150 mm, 5 μm), with a mobile phase as acetonitrile-0.4% acetic acid (13:87) at a flow rate of 1.0 mL/min. The detection wavelength was 330 nm and the column temperature was 30°C. The injection volume was 10 μL. Chen et al [7] established the method of the determination of arctiin content in yinqiao jiedu pills by RP-HPLC so as to provide a rapid and effective analytic approach for the quality



control. Chromatographic column was Shimadzu C18 (4.6 mm×250 mm). The mobile phase was methanol-water (47:53). The flow rate was 1 mL/min. The detection wavelength was 280 nm. The external standard method was used to investigate the chromatographic peak of the drug under the chromatographic condition. Simultaneously, the specificity, linear relationship, accuracy and the others were observed. Eight-wavelength HPLC fingerprints of Yinqiao Jiedu Pills were established by Sun et al [8] to comprehensively evaluate the quality of Yinqiao Jiedu Pills. Information entropy-weight method was applied to integrate the qualitative and quantitative information of Yinqiao Jiedu Pills at eight-wavelength fingerprints, in which the quality of 15 batches of Yinqiao Jiedu Pills was assessed by the 6-level systematically quantified fingerprint method. In this paper, the forsythia content in Yinqiao Jiedu Pills was determined by High Performance Capillary Electrophoresis.

2. Experimental section

2.1. Instruments and Reagents

Experimental instruments: CL-1030-type high performance capillary electrophoresis (Beijing Cailu Scientific Instrument Co., Ltd.); HW2000-type chromatography workstation (Nanjing Qianpu Software Ltd.); Capillary (75 μ m inner diameter, 52 cm overall length, 44 cm effective length) from Hebei Yongnian Ruifeng Chromatographic Devices Co., Ltd.).

Forsythine (Chinese Drugs and Biological Products); Yinqiao Jiedu Pills (Shanxi huakang pharmaceutical Co., Ltd.); other reagents used in the experiments were all analytical grade; Double-distilled water was used.

2.2. Experimental Methods

Before the start of the experiment, capillary was successively washed with 1 mol·L⁻¹ hydrochloric acid solution, double-distilled water, 1 mol·L⁻¹ sodium hydroxide solution, double-distilled water, buffer solution, each for 8 min. After three times running, capillary was cleaned again using the above method.

Measurements were carded out at 20 kV voltage and experimental temperature at 20°C. UV detection wavelength was 277 nm. Injection time was 10s (7.5 cm height difference).

2.3. Sample Preparation

Yinqiao Jiedu Pills sample solution: Yinqiao Jiedu Pills powder was accurately weighed 1.8914 g, added 40 mL water with 30% ethanol, extracted time of 3h at 60°C, filtered, washed and set the volume to 50 mL that was the Yinqiao Jiedu Pills sample solution.

Forsythine standard solution: Forsythine was accurately weighed 0.0018 g, added 4 mL water with 30% ethanol.

3. Results and Discussion

3.1. Selection electrophoresis conditions

Based on past experiment experience, we chose 10 mmol/L borax solution as a running buffer solution.

According to the literature, forsythia maximum absorption wavelength was at 277 nm, so we chose the 277 nm detection wavelength.

3.2. Quantitative analysis

3.2.1. Standard curve. First, forsythia standard solution that the concentration were 0.45, 0.225, 0.112, 0.056, 0.028, 0.014 mg/mL was prepared. Each standard solution was run for three times under the above electrophoresis conditions and the results averaged. The chromatogram of forsythia standard solution was showed in Figure 1. Taking concentration as the abscissa and peak area as the ordinate, the standard curve was drew. Linear regression equation of forsythia (peak area: y μ V·s, density: x mg/mL) and the linear range was as follows: $y = -927.3 + 36773.4x$ ($r = 0.987$), 0.014 -0.45 mg/mL.

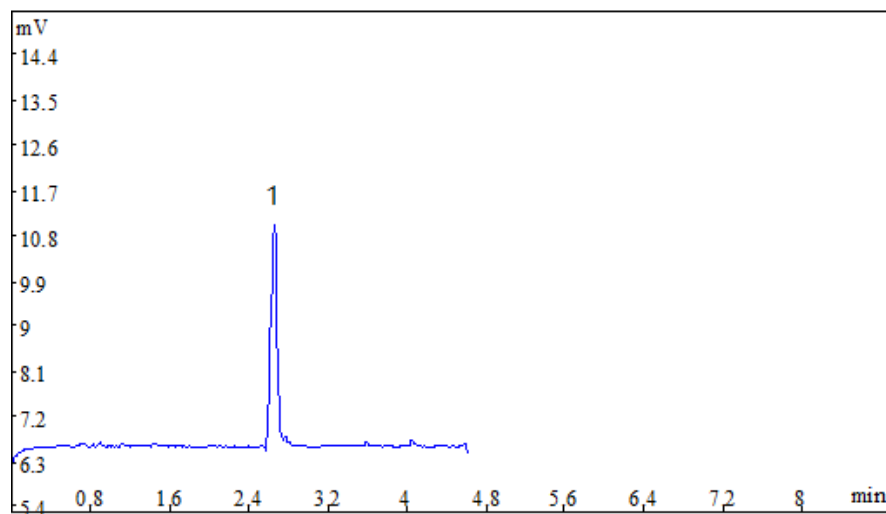


Figure 1. Electrophorogram of forsythia standard solution
1-forsythin

3.2.2. Precision test. A forsythia standard solution precisely drew and continuously injected for six times under electrophoretic separation conditions, the RSD of forsythia peak area were 1.08%, indicating good precision.

3.2.3. Determination of sample content. Under selected electrophoresis conditions, Yinqiao Jiedu Pills sample solution was run. Separation chromatogram of the Yinqiao Jiedu Pills sample solution was showed in Figure 2. Measured forsythia content in Yinqiao Jiedu Pills was 4.615 mg/g (RSD = 10.1%) (n = 6).

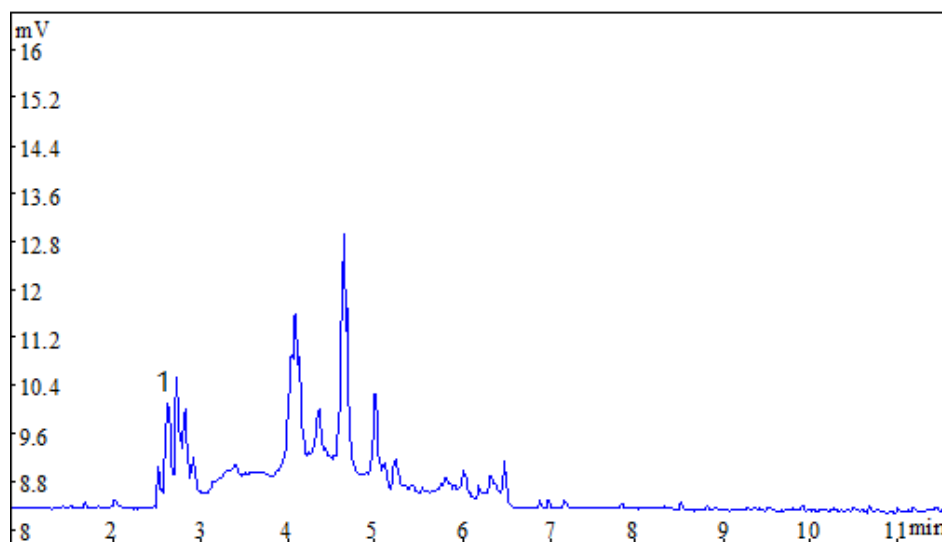


Figure 2. Electrophorogram of Yinqiao Jiedu Pills sample solution
1-forsythin

3.2.4. Recovery. After determination for six times, the recovery of forsythia in Yinqiao Jiedu Pills sample was in the range of 80.4% - 127.0% (n=4).

4. Conclusion

This paper developed the determination of forsythia content in Yinqiao Jiedu Pills by high performance capillary electrophoresis (HPCE) method. The content of forsythia in Yinqiao Jiedu Pills was 4.615 mg/g (RSD = 10.1%) (n = 6).

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