

Determination of Forsythin in Lianqiao Baidu Pills by Capillary Electrophoresis

Li-Kun Han, Na-Na Zhang, Hai-Xing Liu *

Chemistry & Chemical and Environmental Engineering College, Weifang University,
Weifang 261061, P.R. China

Corresponding author e-mail: haixingliu@tom.com

Abstract: This paper investigated the determination of forsythin content in Lianqiao Baidu 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 forsythin in Lianqiao Baidu Pills was 2.838 mg/g (RSD = 8.05%) (n = 6). The recovery was in the range of 82.3%-105.2% (n=6). This method is suitable for the detection of the content of forsythin in Lianqiao Baidu Pills.

1. Introduction

Lianqiao Baidu Pills consists nineteen herbs of weeping forsythiae capsule, honeysuckle flower, bunge corydalis, mongolian snakegourd root, baical skullcap root, golden thread, amur corktree bark, rhubarb, lightyellow sophora root, fineleaf schizonepeta herb, divaricate saposhnikovia root, white Chih, forbes notopterygium rhizome, ephedra herb, peppermint, thorowax root, chinese angelica, red paeony root, liquoric root, etc. It has the effect of clearing heat, detoxifying, eliminating headache and diminishing swelling. The TLC method was developed to improve the quality standard of Lianqiao Baidu Pills for ephedrae herba, sophorae flavescentis radix, paeoniae radix rubra, and RP-HPLC method was established by Wu et al [1] for the determination of chlorogenic acid in Lianqiao Baidu Pills. Zorbax SB-C18 (4.6 mm×150 mm, 5 μm) column was used with methanol-acetonitrile (2:1)-0.04% phosphoric acid (10:90) as the mobile phase at flow rate of 0.8 mL/min. The detection wavelength was 326 nm. Pan et al [2] established the method for the determination of baicalin content in Lianqiao Baidu Pills by HPLC. The Kromasil C18 (5μm, 100A, 250×4.6mm) column was used. The mobile phase was methanol:water:phosphoric acid (47:53:0.2), The detection wavelength was 280 nm, flow rate was 1 mL/min, and the column temperature was 40°C. An HPLC method was established by Li et al [3] for determining Baicalin in Lianqiao Baidu pills. The Agilent TC-C18 column (4.6 mm×250 mm, 5 μm) was used and the mobile phase was methol-water-phosphoric acid(47:53:0.2), and the detection wavelength was 278 nm. The column temperature was 30°C. An online micellar electrokinetic chromatography-electrospray ionization mass spectrometry method for simultaneous determining forsythin, chrysophanol and emodin in Lianqiao Baidu Pills was established by Zeng et al [4]. The samples were carried by an uncoated capillary (50 μm × 78 cm) on the operating voltage of 25 kV using 40 lauric acid-100mmol/L ammonia mixture containing 20% acetonitrile (pH=9.0) as the running buffer. The effect of experimental conditions, such as selection of bases, concentration of lauric acid, addition of organic solvent, applied voltage and the composition of sheath liquid on the separation and ionization of herbicides was optimized. The baseline separation of three components



was achieved within 14 minutes with satisfactory repeatability and sensitivity. Jia et al [5] established the HPLC determination of Lianqiao Baidu tables emodin and chrysophanol. The C18 (4.6 mm×250 mm, 5 μm) column was selected. Methanol-0.1% phosphoric acid (82:18) was the mobile phase. The detection wavelength was 254nm. The velocity was 1.0 mL/min. In recent years, capillary electrophoresis has been widely applied [6-8]. In this paper, the forsythin content in Lianqiao Baidu 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 μm inner diameter, 52 cm overall length, 44 cm effective length) from Hebei Yongnian Ruifeng Chromatographic Devices Co., Ltd.).

Forsythine (Chinese Drugs and Biological Products); Lianqiao Baidu Pills (Neimenggu tianqi zhongmeng 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

Lianqiao Baidu Pills sample solution: Lianqiao Baidu Pills powder was accurately weighed 2.2178 g, added 40 mL water with 30% ethanol, extracted time of 5h at 60°C, filtered, washed and set the volume to 50 mL that was the Lianqiao Baidu 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, forsythin 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, forsythin standard solution was prepared and its concentrations were 0.45, 0.225, 0.112, 0.056, 0.028, 0.014 mg/mL. Each standard solution was run for three times under the above electrophoresis conditions and the results averaged. The chromatogram of forsythin 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 forsythin (peak area: y μV·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.

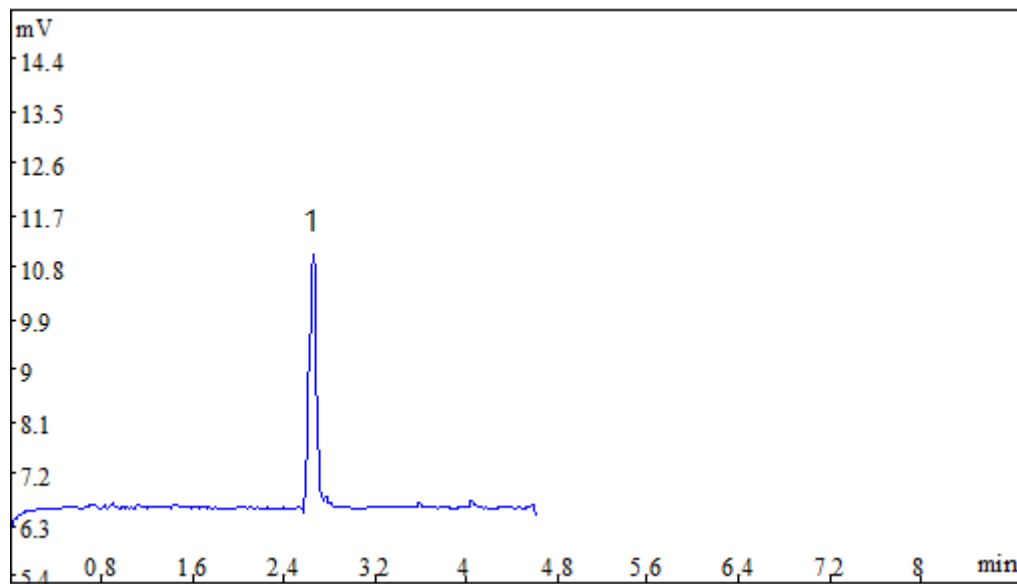


Fig.1 Electrophorogram of forsythin standard solution
1-forsythine

3.2.2. Precision test

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

3.2.3. Determination of sample content

Under selected electrophoresis conditions, Lianqiao Baidu Pills sample solution was run. Separation chromatogram of the Lianqiao Baidu Pills sample solution was showed in Figure 2. Measured forsythin content in Lianqiao Baidu Pills was 2.838 mg/g (RSD = 8.05%) (n = 6).

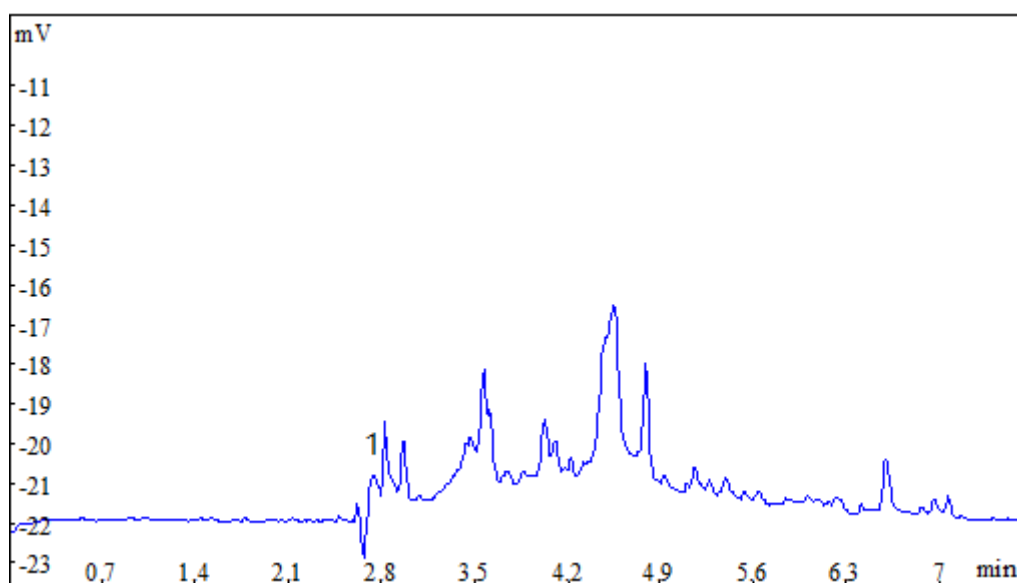


Fig.2 Electrophorogram of Lianqiao Baidu Pills sample solution
1-forsythine

3.2.4. Recovery

After determination for six times, the recovery of forsythin in Lianqiao Baidu Pills sample was in the range of 82.3% - 105.2% (n=6).

4. Conclusion

This paper investigated the determination of forsythin content in Lianqiao Baidu Pills by high performance capillary electrophoresis method. The content of forsythin in Lianqiao Baidu Pills was 2.838 mg/g (RSD = 8.05%) (n = 6).

Acknowledgments

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