Determination of Forsythin in Xiaoer Ganmao Granules by Capillary Electrophoresis

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Abstract: This paper set up the determination of forsythin content in Xiaoer Ganmao Granules 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 Xiaoer Ganmao Granules was 0.699 mg/g (RSD = 6.04%) (n = 5). The recovery was in the range of 101.1% - 119.6% (n=4). This method is suitable for the detection of the content of forsythin in Xiaoer Ganmao Granules.

1. Introduction

Xiaoer Ganmao Granules consists of cablin potchouli herb, chrysanthemum, weeping forsythiae capsule, rehmannia root, cynanchum atratum Bunge, lycium chinense mill. root bark, gypsum, indigowoad leaf and indigowoad root compound preparation. It has the effect of eliminating headache and refreshing, clearing heat and detoxifying. It use treatment of xiaoer catarrh, fever, head pain, cough phlegm and sore throat [1,2]. Yang et al [1] established the quality standards for Xiaoer Ganmao Granules. Pogostemonis herba, chrysanthemi flos, and forsythiae fructus were identified with TLC. Phillyrin was determined by HPLC. Diamonsil C18(4.6 mm×250 mm, 5 μm) column was used. The column temperature was at 30°C. Acetonitrile-water (24:76) was used as a mobile phase. The detection wavelength was at 202 nm. The flow rate was 1.0 mL/min. Zhang et al [2] establisted the method for quality control of Xiaoer Ganmao Granules. Menthol in the Herba Menthae and pathocohol in the cablin patchouli were identified by gas chromatographic, using a column packed with PEG-20 M as stationary phase. Concentration was 10%. The column length was 2 M. Nitrogen gas was utilized as carrier gas and FID. Zhang et al [3] established a GC method for determination of patchouli alcohol, menthol, α-pinene, β-pinene in Xiaoer Ganmao Granules. HP-5 capillary column (30m×0.32mm×0.25μm) was used. The increase of column temperature was controlled by programming. The initial temperature was 40°C and maintained for 5 min, and then rose at 8°C /min up to 180°C for 2.5min. The injector temperature was 250°C. The detector temperature was 300°C. The carrier gas was nitrogen. The flow rate was 2.0 ml/min and the injection volume was 1μL. The split ratio was 20:1. Zheng et al [4] established a method for determination of the content of forsythin in Xiaoer Ganmao Granules. The Diamonsil C18 column (4.6 mm×250 mm, 5 μm) was used and acetonitrile-water (24:76) as mobile phase. The flow rate was 1 mL /min and detective wavelength was set at 202 nm. Zhang et al [5] established an RP-HPLC method for determination of forsythin in Xiaoer Ganmao Granules. The Shim-pack C18 column (4.6 mm×250 mm, 5 μm) was adopted and acetonitrile-water (20:80) as mobile phase. The flow rate was 1 mL /min and detective wavelength was set at 277nm. In recent years, capillary electrophoresis has been widely applied [6-8]. In this paper, the

forsythin content in Xiaoer Ganmao Granules 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.).

Forsythin (Chinese Drugs and Biological Products); Xiaoer Ganmao Granules (Huaren shanjiu 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

Xiaoer Ganmao Granules sample solution: Xiaoer Ganmao Granules powder was accurately weighed 2.956 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 Xiaoer Ganmao Granules sample solution.

Forsythin standard solution: Forsythin was accurately weighed 0.0018 g and 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.

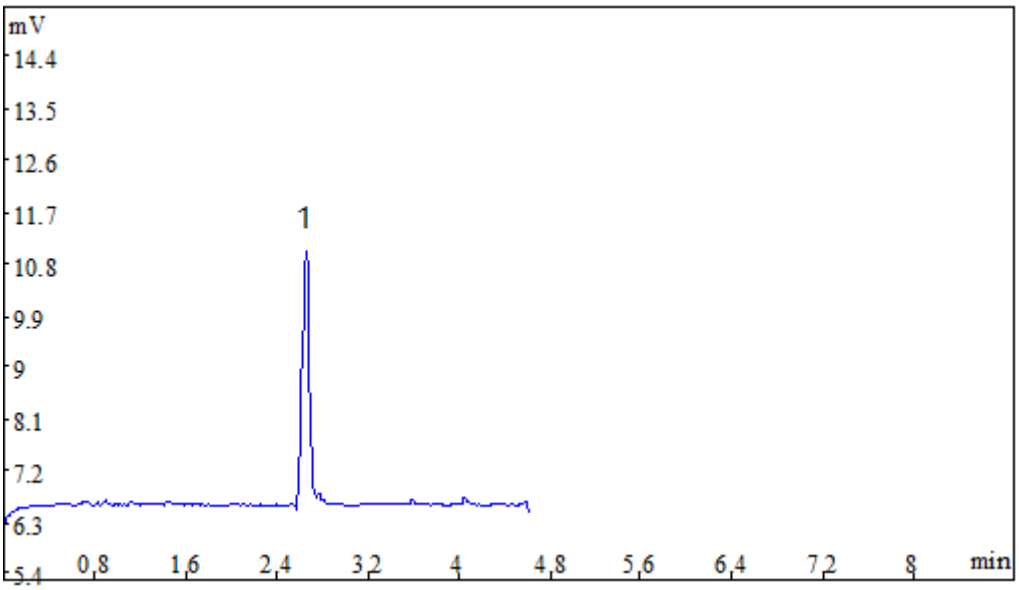

Fig.1 Electrophorogram of forsythin standard solution
1-forsythin

3.2.2. Precision test

A forsythin standard solution precisely drew and continuously injected for sixt 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, Xiaoer Ganmao Granules sample solution was run. Separation chromatogram of the Xiaoer Ganmao Granules sample solution was showed in Figure 2. Measured forsythin content in Xiaoer Ganmao Granules was 0.699 mg/g (RSD = 6.04%) (n = 5).

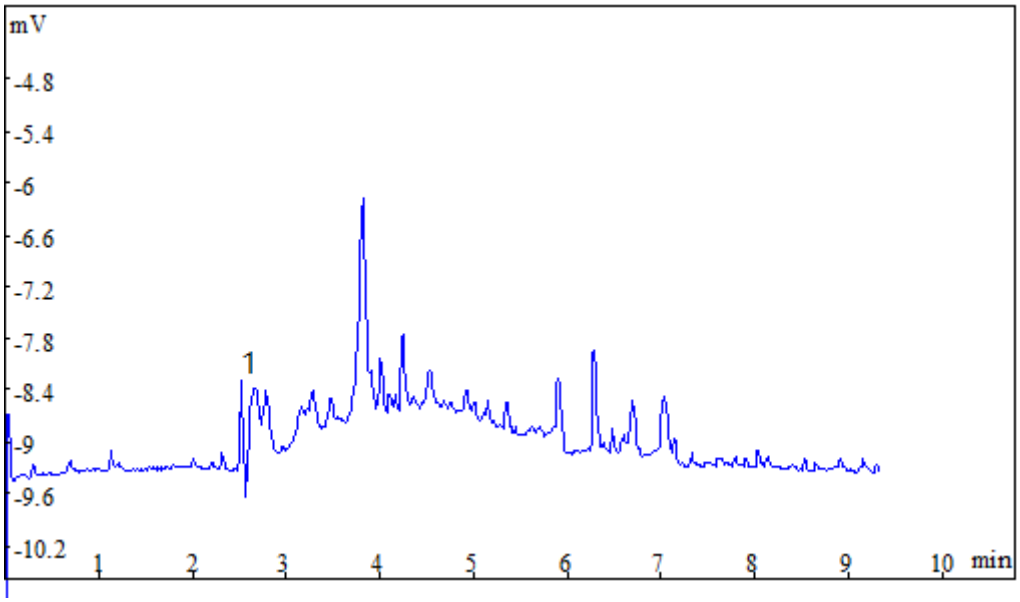

Fig.2 Electrophorogram of Xiaoer Ganmao Granules sample solution
1-forsythin

3.2.4. Recovery

After determination for six times, the recovery of forsythin in Xiaoer Ganmao Granules sample was in the range of 101.1% - 119.6% (n=4).

4. Conclusion

This paper set up the determination of forsythin content in Xiaoer Ganmao Granules by high performance capillary electrophoresis method. The content of forsythin in Xiaoer Ganmao Granules was 0.699 mg/g (RSD = 6.04%) (n = 5).

Acknowledgments

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References

[1] Yang Kan, World Chinese Medicine, 10(3), 427-430(2015)
[2] Zhang Jin, Mao Qing, Zheng Xiaoping, Shi Ke, Zhou Xiangmin, Chin J of Pharm Anal, 28(3), 480-482(2008)
[3] Zhang Xiaolong, Sun Xiao, Ma Xinfei, Sun Si, Drug Testing, 11, 110-111(2014)
[4] Zheng Xiaoping, Yang Kan, China pharmaceuticals, 20(7), 26(2011)
[5] Zhang Jin, Mao Qing, Zheng Xiaoping, Shi Ke, Zhou Xiangmin, Chin J of Pharm Anal, 27(7), 1065-1067(2007)
[6] Hai-Xing Liu, Xiao-Ping Zhang, Yun-Chen Zhang, Lin-Tong Wang and Hui-Qin Wang, Determination of paeonol in yanyan tablets by Capillary Electrophoresis, International Conference on Advances in Energy, Environment and Chemical Engineering, 2015, 739-741(2015)
[7] Li-Kun Han, Hai-Xing Liu*, Lintong Wang, The Analysis of Ceftezole sodium and Cefpiramide sodium by Capillary Zone Electrophoresis, 2016 5th International Conference on Advanced Materials and Computer Science, 80, 255-258(2016)
[8] Hai-Xing Liu*, Chun-Yan Wang, Xue-Wei Peng, Determination of Betaine in Lycium barbarum L. by high Performance Capillary Electrophoresis, Materials Science and Engineering, 274, 012074(2017)