Determination of Arctiin in Xiaoer Qingyan Granules by Capillary Electrophoresis

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Abstract. This paper investigated the determination of arctiin content in Xiaoer Qingyan granules by high performance capillary electrophoresis (HPCE) method. The borax solution of 37.5 mmol concentration containing 12.5% methanol was chosen as buffer solution. The experiment was performed at a constant voltage of 16 kV and UV detection wavelength of 277 nm. The content of arctiin in Xiaoer Qingyan granules was 3.678 mg/g (RSD=1.72%) (n=6). The recovery was in the range of 80.9%-113.4% (n=6). This method is suitable for the detection of the content of arctiin in Xiaoer Qingyan granules.

1. Introduction
Xiaoer Qingyin granules consists of figwort root, mongolian dandelion herb, great burdock achene (fried), peppermint, cicada slough, indigowoad root, weeping Forsythia capsule, tree peony bark and natural indigo 9 traditional Chinese medicine etc. It has the functions of clearing away heat and the surface, detoxifying and alleviating pharynx. It is used for the treatment of fever, headache, cough, hoarse voice, and swelling and pain in the throat caused by wind-heat in children. Li et al [1] established a HPLC-DAD method for the simultaneous determining 9 compounds (neochlorogenic acid, chlorogenic acid, cryptochlorogenic acid, sweroside, secoxyloganin, forsythiside A, 3,4-dicaffeoylquinic acid, 3,5-dicaffeoylquinic acid and 4,5-dicaffeoylquinic acid) in Xiaoer Qingjie granules. The analysis was carried out on an Agilent Zorbax SB C18 column (250 mm×4.6 mm, 5 μm) by the mixture of 0.1% formic acid and acetonitrile with flow rate of 1.0 mL/min at gradient elution. The DAD detector wavelength was set at 325 nm for neochlorogenic acid, chlorogenic acid, cryptochlorogenic acid, forsythiside A, 3,4-dicaffeoylquinic acid, 3,5-dicaffeoylquinic acid and 4,5-dicaffeoylquinic acid, and 240 nm for sweroside and secoxyloganin. The column temperature was 30°C. Hu et al [2] established the RP-HPLC method for the determining indigo and indirubin in Xiaoer Qingyan granules. The analysis was performed on the Agilent HC-C18 (250 mm×4.6 mm, 5 mm) column with the mobile phase of acetonitrile-0.1% Hac (78: 22) at a flow rate of 1.0 mL/min. The column temperature was 30°C and the detection wavelength was set at 292 nm. Cui et al [3] established a HPLC method for the determination content of Forsythin in Xiaoer Qingie granules. The analysis was tested on Agilent C18 (250×4.6mm, 5μm) column. The mobile phase was composed of acetonitrile-water (24: 76) with flow rate of 1.0 ml/min. The detection wavelength was 277 nm and column temperatures was 30°C. Li et al [4] established the method for the determination of Forsythin in Xiaoer Qingyan Granules using HPLC method. The VP-ODS C18 column (150 mm×4.6 mm, 5 mm)
was used with the mobile phase of 0.1% phosphoric acid-acetonitrile at gradient elution and flow rate of 1.0mL/min. The detection wavelength was set at 277nm. Zhang et al [5] developed a method for simultaneous determining content of harpagide, harpagoside, chlorogenic acid, caffeic acid and phillyrin in Xiaoer Qingyan granules by HPLC. The analysis was carried out on Zorbax SB-C<sub>18</sub> column with solvent system composed of acetonitrile-0.1% phosphoric acid and flow rate of 1.0 mL/min. The detecting wavelength was 210nm (0-13min, harpagide), 327nm (13-25min, chlorogenic acid and caffeic acid), 277nm (25-29min, phillyrin), 210nm (29-40min, harpagosid); the column temperature was 30°C, and sample size was 10μL. Qiu yan [6] established a HPLC method for the determining caffeicacid content in Xiaoer Qingyan granules. The SHISEIDO CAPCELL PAK C<sub>18</sub> column was used with mobile phase of phosphate buffered solution-Acetonitrile (78: 22) at a flow rate of 1.0mL/min. The detection wavelength was 327 nm and the temperature was 40°C. Sui Yi [7] established a rapid identifying method of illegally added amidopyrine in Xiaoer Qingjie granules. Illegally added acetanilide detumescence compound amidopyrine from four different companies were screened by liquid chromatogram and mass-spectrography, and reference substance of secondary mass spectra were compared. In this paper, the arctiin content in Xiaoer Qingyan 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 (75μm inner diameter, 52cm overall length, 44cm effective length) from Hebei Yongnian Ruifeng Chromatographic Devices Co., Ltd.).
Arctiin (Chinese Drugs and Biological Products); Xiaoer Qingyan granules (Fujian Quanzhou Shanshangshan Pharmaceutical limited company, Batch number: 170906); 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<sup>-1</sup> hydrochloric acid solution, double-distilled water, 1 mol·L<sup>-1</sup> sodium hydroxide solution, double-distilled water, buffer solution, each for 5 min. After three times running, capillary was cleaned again using the above method.
Measurements were carried out at 16 kV voltage and experimental temperature at 21°C. UV detection wavelength was 277 nm. Injection time was 10s (7.5 cm height difference).

2.3. Sample Preparation
Xiaoer Qingyan granules sample solution: Xiaoer Qingyan granules was accurately weighed 3.0706 g, added 40 mL water containing 80% methanol, extracted time of 48h at 21°C, filtered, washed and set the volume to 50 mL that was the Xiaoer Qingyan granules sample solution.
Arctiin standard solution: Arctiin was accurately weighed 0.0046 g, added 2 mL water.

3. Results and Discussion

3.1. Selection electrophoresis conditions
The experiment was carried out at 16 kV voltage. UV detection wavelength was 277 nm.
Based on past experiment experience, 37.5 mmol/L borax solution containing 12.5% methanol was chosen as electrolyte solution.
3.2. Quantitative analysis

3.2.1. Standard curve. First, arctiin standard solution was prepared and its concentrations were 2.3, 1.15, 0.575, 0.2875, 0.1437, 0.0718, 0.0359 mg/mL. Each standard solution was run for three times under the above electrophoresis conditions and the results averaged. The chromatogram of arctiin 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 arctiin (peak area: \( y \mu \text{V}\cdot\text{s}, \) density: \( x \) mg/mL) and the linear range was as follows: \( y = -2653.6 + 84674.5x(r=0.998), \) 0.0359-2.3 mg/mL.

![Figure 1. Electrophorogram of arctiin standard solution 1-arctiin](image)

3.2.2. Precision test. A arctiin standard solution precisely drew and continuously injected for six times under electrophoretic separation conditions, the RSD of arctiin migration time and peak area were 2.25% and 4.91%, indicating good precision.

3.2.3. Determination of sample content. Under selected electrophoresis conditions, Xiaoer Qingyan granules sample solution was run. Separation chromatogram of the Xiaoer Qingyan granules sample solution was showed in Figure 2. Measured arctiin content in Xiaoer Qingyan granules was 3.678mg/g (RSD=1.72%) (n=6).

![Figure 2. Electrophorogram of Xiaoer Qingyan granules sample solution 1-arctiin](image)
3.2.4. Recovery. After determination for six times, the recovery of arctiin in Xiaoer Qingyan granules sample was in the range of 80.9%-113.4% (n=6). The average recovery was 95.6%.

4. Conclusion
This paper investigated the determination of arctiin content in Xiaoer Qingyan granules by high performance capillary electrophoresis method. Measured arctiin content in Xiaoer Qingyan granules was 3.678mg/g (RSD=1.72%) (n=6).

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