Determination of Arctiin in Lingqiao Jiedu Tablet by Capillary Electrophoresis

Likun Han, Nana Zhang, Haixing Liu*, Lintong Wang

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 arctiin content in Lingqiao Jiedu Tablet 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 Lingqiao Jiedu Tablet was 5.002 mg/g (RSD=2.85%) (n=7). The recovery was in the range of 85%-120.7% (n=5). This method is suitable for the detection of the content of arctiin in Lingqiao Jiedu Tablet.

1. Introduction

Lingqiao Jiedu Tablet consists of antelope horn, honeysuckle flower, weeping forsythiae capsule, peppermint, fineleaf schizonepeta herb, fermented soybean, great burdock achene, platycodon root, common lophatherum herb, liquorice root and borneol 11 traditional Chinese medicine. It has the effect of pungent cooling and relieving exterior, clearing away heat and detoxification. It is used for the treatment of anemophobia fever, limp acheing tired, headache, rhinobyon, cough and sore throat. A headspace gas chromatography method was developed by Bai et al [1] for the determining contents of menthone, menthol and pulegone in Yinqiao Jiedu Mixture. The drug was investigated and adopted HP-5 capillary column (30m×0.25mm, 0.25 μm) with column temperature of 50-240 ℃ at programmed temperature. FID detector temperature was 250 ℃ with shunt ratio of 20:1. Headspace injection thermostat balanced at 90 ℃ for 40 min with slight oscillation. The sample flow path temperature was 100 ℃. The transmission line temperature was 110 ℃. Lingqiao Jiedu Pellets was obtained by Ren Qian [2] using extrusion-spheronization method. Formulation composition and process factors influencing physical properties, such as spherical shape degree and productivity, were investigated. It had a film coating by fluidized bed. The extraction of arctiin and arctigenin from Fructus Arctii was tested by Sun et al [3]. The microwave-assisted extraction progress of arctiin and arctigenin was investigated to obtain the optimum conditions using single factor and orthogonal experiments. The contents of arctiin and arctigenin were obtained by high performance liquid chromatography method. The optimum conditions were as follows: 40% ethanol concentration, microwave power of 500W, material to liquid ratio of 1:15, extraction time of 200s, extraction 3 times. Under these conditions, the yields of arctiin and arctigenin were 7.14% and 0.53%. Luo et al [4] established a method for determining content of ten elements of Cr, Cu, Fe, Mg, Mn, Ni, Pb, Sr, Ti and Zn in Arctium tomentosum Mill seeds. The samples were digested by nitric acid-perchloric acid-hydrochloric acid,
the content of ten elements in the Arctium tomentosum Mill seeds was measured using ICP-AES. The plasma kinetics and tissue distribution of arctigenin after subcutaneous injection in Wistar rats was tested by Han et al [5]. Wistar rats were injected subcutaneously with arctigenin at 0.1, 0.3 and 1.0 mg/kg, blood and tissue samples were collected at different time points after injection, and concentrations of arctigenin in the samples were detected by liquid chromatography-mass spectrometry/mass spectrometry (LC-MS/MS). Tang et al [6] determined the content of paracetamol, chlorogenic acid, Chlorphenamine maleate, forsythiaside A, glycyrrhizin and arctiin in Vitamin C Yinqiao tablets. The HPLC system was performed on Phenomenex Gemini C18 (250 mm × 4.60 mm, 5μm) at 35°C. The mobile phase was consisted of acetonitrile-1% acetic acid solution in gradient elution mode. The flow rate was set at 1.0 mL/min and the detection wavelength was set at 280 nm. Cui et al [7] determined the content of arctii, arctigenin and chlorogenic acid in Qingfei Xiaoyan Wan by HPLC. The symmetry C18 color table was applied while the mobile phase was methanol-water (50: 50) and the detection wavelength was 280 nm for arctii and arctigenin, the mobile phase was acetonitrile-0.4% phosphoric acid (13: 87) and the detection wavelength was 327 nm for the chlorogenic acid. In this paper, the arctiin content in Lingqiao Jiedu Tablet 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.). Arctiin (Chinese Drugs and Biological Products); Lingqiao Jiedu Tablet (Beijing Tongrentang Pharmaceutical limited company, Batch number: 15124758; 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 5 min. After three times running, capillary was cleaned again using the above method.
Measurement 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
Lingqiao Jiedu Tablet sample solution: Lingqiao Jiedu Tablet was accurately weighed 2.1759 g, added 40 mL water containing 80% methanol, extracted time of 24h at 21°C, filtered, washed and set the volume to 50 mL that was the Lingqiao Jiedu Tablet 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: \( y \mu V\cdot s \), 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.

![Fig.1 Electrophorogram of arctiin standard solution](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, Lingqiao Jiedu Tablet sample solution was run. Separation chromatogram of the Lingqiao Jiedu Tablet sample solution was showed in Figure 2. Measured arctiin content in Lingqiao Jiedu Tablet was 5.002mg/g (RSD=2.85%) (n=7).
3.2.4. Recovery. After determination for five times, the recovery of arctiin in Lingqiao Jiedu Tablet sample was in the range of 85%-120.7% (n=5). The average recovery was 99.5%.

4. Conclusion
This paper investigated the determination of arctiin content in Lingqiao Jiedu Tablet by high performance capillary electrophoresis method. Measured arctiin content in Lingqiao Jiedu Tablet was 5.002mg/g (RSD=2.85%) (n=7).

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References
[1] Bai Ganggang, Zhang Li, Chen Xiaopeng, Chou Yajing, Determination of three compounds in by Yinqiao Jiedu Mixture by headspace gas chromatography method, Chinese Traditional Patent Medicine, 41(3) (2019), 657-659.
[2] Ren Qian, Study on the Preparation of Lingqiao Jiedu Pellets, Journal of Traditional Chinese Medicine Information, 13(1) (2006), 49-50.
[3] Sun Zhaoyun, Song Jiying, Shan Hu, Lv Haitao, Microwave-assisted extraction of arctiin and arctigenin from Fructus Arctii, Science and Technology of Food Industry, 34 (19) (2013), 191-194.
[4] Luo Xiuzhen, Wang Jun, Li Pengfei, Zhou Xiaoying, Determination of Trace Elements in Arctium tomentosum Mill Seeds by ICP-AES, Journal of Anhui Agri. Sci., 43 (14) (2015), 89.
[5] Han Shu, Gu Yuan, Yang Yuanhui, Shang Qingjie, Yao Jingchun, Liu Wanhui, Si Duanyun, Plasma Kinetics and Tissue Distribution of Arctigen in Rats after Subcutaneous Injection, Journal of Yantai University, 30 (2) (2017), 131-137.
[6] Tang Danfeng, Zheng Qin, Luo Jun, Zhu Meifang, Wu Haixia, Zhang Delin, Yang Ming,
Determination of 6 kinds of paracetamol, chlorogenic acid, Chlorphenamine maleate, forsythiaside A, glycyrrhizin and arctiin in Vitamin C Yinqiao tablets by HPLC, *LISHIZHEN MEDICINE AND MATERIA MEDICA RESEARCH, 28 (2) (2017), 348-350.*

[7] Cui Qingxin, Liao Chengmei, Chen Zhikai, Ma Fang, Tao Jin, Determined content of three anti-inflammatory ingredients in Qingfei Xiaoyan Wan by using HPLC method, *Experimental Technology and Management, 33 (10) (2016), 55-58.*