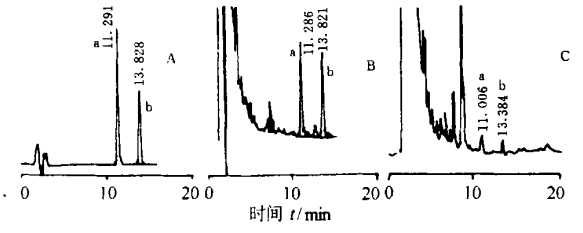

a吴茱萸碱 b吴茱萸次碱

a-evodiamine b-rutaecarpine

图 2 对照品 (A)、吴茱萸药材 (B)和萸黄连 (C)的 HPLC图谱

Fig. 2 HPLC chromatograms of reference substances (A), *E. rutaecarpa* (B) and *C. chinensis* processed with *E. rutaecarpa* (C)

萸黄连中小檗碱的含量较黄连低,其中 20%, 30%萸黄连中小檗碱的含量较高,本结论与文献[3]一致,辅料量增大则小檗碱的含量减少。

3.2 由表 1图 1可知,萸黄连中确实吸收了吴茱萸中的成分,萸黄连中吴茱萸用量越大,吴茱萸碱及吴茱萸次碱的含量越大。另外,因为 10%、20%萸黄连的 HPLC图谱中,吴茱萸碱的峰面积很小,与其他成分没有完全分离,故数据不做统计。

4 讨论

4.1 本实验建立了小檗碱、吴茱萸碱、吴茱萸次碱的含量测定方法,简便、可靠,可以作为黄连和吴茱萸药材、萸黄连饮片以及由二者配伍而成的中成药(如左金丸、变通丸、甘露散等)的质量控制方法。

4.2 萸黄连中吸收了吴茱萸的主要成分吴茱萸碱、吴茱萸次碱。薄层色谱也观察到萸黄连中吴茱萸的成分,以硅胶 G为薄层板,正丁醇-冰醋酸-水(7∶1∶2)为展开剂,于紫外光(365 nm)下检视,与黄连、吴茱萸药材对应位置,萸黄连显相同颜色的斑点。

4.3 从实验结果可以看出,在炮制过程中不同用量的吴茱萸对黄连中的成分是有影响的,故确定最佳辅料用量非常重要。以主要成分小檗碱为考察指标,吴茱萸用量以 20%为宜。

4.4 本实验仅以黄连中的主要成分为指标,若能结合药理或临床进行研究,则结果更有说服力。中药炮制中有许多药物是采用药汁进行炮制,可以达到增效、减毒的目的,这与方剂配伍理论相似。方剂中可以根据具体病情对方中药物进行加减,那么中药炮制中药汁(如生姜汁、甘草汁、吴茱萸汁、黑豆汁等)炮制药物的用量根据什么进行判定?如果可能结合药理与临床进行研究,那么指标如何选择?这些问题都有待于进一步探讨。

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Determination of scutellarin in Breviscarpin Tablet by HPLC

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Abstract **Object** To establish an HPLC method for the determination of scutellarin in Breviscarpin Tablet. **Methods** Column: Diamonsil C_{18} (4.6 mm× 150 mm, 5μm); mobile phase: acetonitrile-0.5% acetate solution (22∶78); detection wavelength was at 335 nm; temperature was at 30℃; flow velocity was 1.2 mL/min. **Results** The calibration curve showed a good linearity within the range of 0-6.0 μg. The average recovery was 100.43% and *RSD* was 0.58%. **Conclusion** The method is convenient, fast and reliable to operate and suitable for the quality control of Breviscarpin Tablet.

Key words Breviscarpin Tablet; scutellarin; HPLC

高效液相色谱法测定灯盏花素片中灯盏花乙素的含量

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* 收稿日期: 2002-11-08

摘 要: 目的 建立高效液相色谱法测定灯盏花素片中灯盏花乙素的含量。方法 色谱柱 Diamonsil C_{18} (4.6 mm× 150 mm, 5μm),流动相: 乙腈-0.5%乙酸溶液(22∶78),检测波长: 335 nm,柱温: 30℃,流速: 1.2 mL/min。结果 灯盏花乙素线性范围为 0~6.0μg,加样回收率为 100.43%,*RSD*为 0.58%。结论 该方法简便、快速、准确,可用于灯盏花素片的质量控制。

关键词: 灯盏花素片;灯盏花乙素;高效液相色谱
中图分类号: R286.02 文献标识码: B 文章编号: 0253-2670(2003)04-0322-03

The dried whole plant of *Erigeron breviscapus* (Vant.) Hand.-Mazz. is cold in nature and bitter in taste, possessing the efficacy of dispelling cold and inducing diaphoriesis, expelling wind and removing dampness, and activating the collateral circulation to relieve pain[1]. Breviscarpin is an extract from *E. breviscapus*. Pharmacological study showed that breviscarpin could significantly reduce the blood viscosity, improve the blood flow, decrease the vascular resistance, and inhibit the platelet aggregation and thrombosis formation. It has been used in clinic for the treatment of the paralysis induced by cerebrovascular accidents[2], e.g., hypertension, cerebral haemorrhage, cerebral thrombosis and polyneuritis and chronic arachnitis. The total effective rate is 95.8%. As reported the UV spectrophotometric method was used to determine the content of scutellarin in Breviscarpin Tablet. However, since breviscarpin is a mixture consisted of several flavone glycosides, the selectivity of UV spectrophotometric method is not well enough to meet the requirement of its quality control[3]. In the present study, an HPLC method used to separate and determine the content of scutellarin in Breviscarpin Tablet is established.

1 Apparatus and reagents

Apparatus. HP1100 HPLC, G1311A-quaternary pump, G1322A-deaerator, G1316A-thermostatped colum compartment, 1314A-UV variable-wavelength detector, HPRev. A. 0501 chemical workstation (Agilent Technologies).

Reagents. The acetonitrile (C. P. grade), methanol and acetic acid (A. R. grade), the ultrapure water, scutellarin (bought from Yunnan Pharmaceutical Institute) and Breviscarpin Tablet (from Guangdong Huanqiu Pharmaceutical Co. Ltd.) were used in the experiment.

2 Chromatographic condition

Column: Diamonsil C_{18} (150 mm× 4.6 mm, 5μm), mobile phase: acetonitrile-0.5% acetate solution (22∶78), detection wavelength: 335 nm, temperature: 30℃, flow velocity: 1.2 mL/min. The chromatograms of scutellarin and Breviscarpin Tablet were shown in Fig. 1.

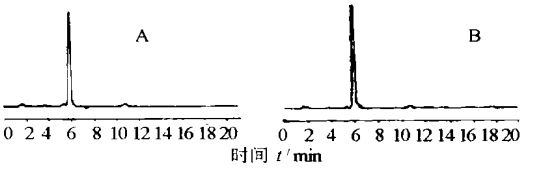

Fig. 1 HPLC chromatograms of scutellarin (A) and Breviscarpin Tablet (B)

3 Experiment

3.1 Linear relation. A stock solution (0.3 mg/mL) of scutellarin was prepared by dissolving a quantitative sample of scutellarin in methanol and ultrasonic oscillation for 30 minutes. After filtration by millipore filter of 0.45μm, the sampling volumes were 2.5, 5.0, 7.5, 10.0, 15.0 and 20.0 μL, respectively. According to the recorded chromatogram, the peak areas with statistical treatment as a function of the concentration (C) gave the good linearity for sample weight in the range of 0~6.0μg. A was $360.06C-4.585$, $r=0.9999$.

3.2 Precision. Injecting continuously the sample of stock solution 10μL for 6 times and measuring the peak areas of scutellarin, the *RSD* was 0.82%.

3.3 Repeatability. Weighing up 6 portions of scutellarin, preparing the solution as the same process in 3.1, the volume of sample introduction in 10μL, according to the chromatogram recorded, the *RSD* was 0.99%.

3.4 Solution stability. Samples of the stock solution that were placed at room temperature for 1, 2, 4, 8, 12 hours, respectively, were injected. The peak areas were invariable and *RSD* was

0.85%, indicating that the solution was stable at least for 12 hours.

3.5 Recovery. The quantitative Breviscarpin Tablet 6 portions were weighed up and put into 10 mL measuring flask, then dissolved in methanol and ultrasonic oscillation for 30 minutes after adding the stock solution of scutellarin. According to the process in 3.6, the results were obtained with average recovery of 100.43% and *RSD* was 0.58% (n= 6).

3.6 Sample measurement. The quantitative Breviscapin Tablet 9 portions were weighed up and put into 50 mL measuring flask, then dissolved in methanol and ultrasonic oscillation for 30 minutes. Filtrate (1 mL) of the second filtration was put into a 10 mL measuring flask and fixed the volume, then 10 μL sample was injected. The measured contents were listed in table 1.

Table 1 Scutellarin in Breviscarpin Tablet (n= 3)

No.	average scutellarin contents/%	*RSD*/%
1	21.97	1.41
2	22.16	0.93
3	21.69	1.83

4 Conclusion

4.1 The selection of mobile phase. The mobile phases of different system and proportions were compared, such as methanol-acetic acid solution, methanol-H_2O-triethylamine, methanol-phosphoric acid-isopropanol, acetonitrile-0.5% acetate solution, and acetonitrile-H_2O-trethylamine. The optimized effect for isolating scutellarin was obtained by acetonitrile-0.5% acetate solution (22∶78) as the mobile phase.

4.2 The selection of extractive method. Several extractive methods, such as Soxhlet extract, heating in water bath, ultrasonic extracting, and different extractive solvents, such as methanol, ethanol, *n*-butanol, ethyl acetate, chloroform and ligarine were used, while using methanol as a solvent and the ultrasonic extracting for 30 minutes was found to be a simple method with little disturbance.

4.3 The range of pH values. Scutellarin is a flavonoid compound, which has several phenolic hydroxyl to display the weak acidity. Comparing the different pH values, the peaks are acuity and symmetry in the pH range at 2.5- 3. That is in coincidance with *Chinese Pharmacopoeia*.

The results indicated that this method is stable, accurate and convenient for controlling quality of the medicine.

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刺糖中氨基酸成分的研究

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刺糖为豆科植物骆驼刺 *Alhagi pseudalhagi* Desv.叶中分泌液凝结而成的糖粒,为一味传统维吾尔医药,在《维吾尔医常用药材》中早有记载[1~3],至今临床仍在使用。据文献报道刺糖中主要成分为糖类(淀粉,鼠李糖等)和维生素 C,维生素 B_1。但是关于刺糖的氨基酸成分至今未见报道。本实验测定了刺糖中氨基酸成分。

1 材料与仪器

* 收稿日期: 2002-08-08
基金项目: 新疆维吾尔自治区教育厅基金资助项目(96022)
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