云实皮抗炎活性部位的化学成分

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[摘要] **目的**:研究云实皮抗炎活性部位的化学成分。**方法**:采用硅胶柱色谱及 Sephadex LH-20 等色谱技术分离纯化,根据理化性质及波普数据鉴定结构。**结果**:从云实皮的醇提物活性部位分离得到 9 个化合物,结构鉴定为:(±)原苏木素 B(**1**),东莨菪亭-7-*O*-*β*-*D*-吡喃葡萄糖苷(**2**),4-羟基-3-甲氧基苯-1-*O*-*β*-*D*-吡喃葡萄糖苷(**3**),(1′*R*, 3′*S*, 5′*R*, 8′*S*, 2*Z*, 4*E*)-二氢红花菜豆酸-3′-*O*-*β*-*D*-吡喃葡萄糖苷(**4**),(1′*R*, 3′*S*, 5′*R*, 8′*S*, 2*E*, 4*E*)-二氢红花菜豆酸-3′-*O*-*β*-*D* 吡喃葡萄糖苷(**5**),甘草素(**6**),异莨菪亭(**7**),7-羟基-3-(4′-羟基亚苄基)-色原-4-酮(**8**),3-去氧苏木查尔酮(**9**)。**结论**:化合物 **2**~**5**,**7** 为首次从该属植物中分离得到,**6**,**8** 为首次从云实中分离的到。

[关键词] 豆科; 云实; 化学成分; 结构鉴定
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Chemical Constituents of Anti-in Flammatory Fraction of *Caesalpinia decapetala*

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[**Abstract**] **Objective**: To study the chemical constituents of the anti-inflammatory fraction of *Caesalpinia decapetala*. **Method**: The compounds were isolated and purified by silica gel and Sephadex LH-20 column chromatography and their structures were determined by chemical evidence and pectral analysis. **Result**: Nine compounds were obtained from *C. decapetala* and they were identified as (±) protosappanin B (**1**), scopoletin-7-*O*-*β*-*D*-glucopyranoside (**2**), 4-hydroxy-3-methoxypheny-1-*O*-*β*-*D*-glucopyranoside (**3**), (1′*R*, 3′*S*, 5′*R*, 8′*S*, 2Z, 4*E*) -dihydrophaseic acid-3′-*O*-*β*-*D*-glucopyranoside (**4**), (1′*R*, 3′*S*, 5′*R*, 8′*S*, 2E, 4*E*) -dihydrophaseic acid-3′-*O*-*β*-*D*-glucopyranoside (**5**), liquiritigenin (**6**), isoscopoletin (**7**), 7-hydroxy-3-(4′-hydroxybenzylidene) -chroman-4-one (**8**), 3-deoxysappanchalcone (**9**). **Conclusion**: Compounds **2-5**, **7** were isolatedfrom thegenus *C.* for the firs time, and compounds **6**, **8** were obtained from the *C. decapetala* for the first time.

[**Key words**] Fabaceae; *Caesalpinia decapetala*; chemical constituents; structure determination

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云实皮,又名阎王刺,是豆科植物云实的干燥根或根皮。云实主要分布在亚热带和热带地区,我国主产于贵州、云南、广西等地,共包括 17 种,民间用药达 13 种[1]。该植物具有清热除湿、杀虫之功效,主要为民间用药,其根、皮、种子入药可用于治疗发热[2],痢疾、疟疾、消渴、小儿疳积和风湿病等疾病[3-4]。目前关于云实的相关报道大部分局限于化学成分的研究:如李茂星等从云实的乙酸乙酯萃取部位先后共分离得到 14 个化合物[5];张琼等分别从云实的三氯甲烷和乙酸乙酯部位分离得到 2 和 5 个化合物,其中 2 个为新化合物[6];欧阳晓卫针对云实茎中的酚性成分进行了研究[7],上述研究中均未见到活性相关的报道。但在魏小华关于云实的综述中提到云实具有多种药理活性[8],因此本课题以活性为导向,将乙醇提取物经大孔树脂分离得到的 4 个组分进行了抗炎活性筛选,对具较好抗炎活性的 45% 乙醇洗脱组分进行了分离,从中获得 9 个化合物,其中化合物 **2**~**5**,**7** 为首次从该属植物中分离得到,**6**,**8** 为首次从云实中分离得到。该研究为阐明云实皮的药用物质基础提供了一定的科学依据。

1 材料

核磁共振谱 DRX-500 或 INOVA-400 型核磁共振仪(美国 Varian 公司,TMS 为内标),真空干燥箱(上海医用恒温设备厂),WATERS 超高效液相色谱 ACQU I TYUPLC 系统(美国沃特世公司),B-490 型旋转蒸发仪(瑞士 BUCHI),柱层析硅胶(青岛海洋化工厂),薄层色谱板(青岛海洋化工厂),反相硅胶(德国 Merck 公司),Sephadex LH-20 (Amersham Pharmacia Biotech 公司),MCI 树脂(日本三菱公司);乙腈为色谱纯,化其余学试剂均为分析纯。

云实皮药材由贵州民族药厂提供,由贵阳医学院龙庆德副教授鉴定为云实 *Caesalpinia decapetala* (Roth) Alston 的干燥根和根皮。

2 提取与分离

云实皮药材粗粉 15 kg,70% 乙醇回流提取 3 次,每次 2 h,滤液合并回收乙醇并浓缩至无醇味,以 D_{101} 大孔树脂吸附,依次用水,45% 乙醇,75% 乙醇,95% 乙醇洗脱,洗脱液回收乙醇并浓缩至浸膏,得到不同极性的 4 个组分;以醋酸致小鼠腹膜炎为模型对所获得的 4 个组分进行抗炎活性筛选,结果表明 45% 乙醇洗脱部位抗炎活性较强。45% 乙醇洗脱部位经硅胶柱层析(三氯甲烷-甲醇梯度洗脱 10∶1 ~ 0∶1)得 4 个组分。组分 1 经反复硅胶、Sephadex LH-20 和反相柱色谱得化合物 **6**(12 mg)、化合物 **7**(20 mg)、化合物 **8**(8 mg)、化合物 **9**(15 mg);组分 2 经 MCI(甲醇/水)初步分离后再经 Sephadex LH-20 和反复硅胶柱色谱得化合物 **1**(500 mg) 和化合物 **2**(130 mg);组分 3 通过 Sephadex LH-20 和硅胶柱色谱分离后再运用反相 RP-18 柱色谱和高效液相制备技术得化合物 **3**(17 mg)、化合物 **4**(80 mg)、化合物 **5**(90 mg)。

3 结构鉴定

化合物 **1** 淡褐色粉末(甲醇);ESI-MS *m/z* 305 $[M+H]^+$, 303 $[M-H]^-$。^{1}H-NMR (400 MHz, CD_3COCD_3) δ:7.02,7.00 (1H,1H, d, J = 8.4 Hz, H-1), 6.85 和 6.75 (1H,1H, brs, H-9), 6.78,6.72 (1H,1H, m, H-12), 6.63(1H, dd, J = 8.4, 2.0 Hz, H-2), 6.58 (1H, overlapped, H-2), 6.57,6.49 (1H,1H, d, J = 2.0 Hz, H-4), 4.36, 4.13 (1H,1H, d, J = 12.0 Hz, H-6b), 3.87 (1H, d, J = 12.0 Hz, H-6a), 3.56 (1H, overlapped, H-6a), 3.60,3.58 (1H,1H, overlapped, C_7-CH_b), 3.48,3.39 (1H,1H, d, J = 11.6 Hz, C_7-CH_a), 2.75 (1H, d, J = 13.2 Hz, H-8b),2.67 (1H, d, J = 13.2Hz, H-8a), 2.56 (1H, d, J = 13.2Hz, H-8b), 2.55 (1H, d, J = 13.2Hz, H-8a);^{13}C-NMR (100 MHz, CD_3COCD_3) δ: 160.5/159.1 (C-3), 158.8/158.7 (C-4a), 144.5/144.5/144.4/144.4 (C-9,12), 133.2/132.2 (C-1), 132.2/131.6 (C-12a), 128.2/127.2(C-12b), 125.1/123.2(C-8a), 119.6/118.8(C-12), 117.3/117.1(C-9), 111.7/110.0(C-2), 108.8/107.9(C-4), 77.1/75.7(C-6), 72.6/72.1(C-7), 68.0/65.6(C_7-CH_2), 42.5/39.8(C-8)。以上数据与文献[9]报道的(±)原苏木素 B 基本一致,并根据 HMQC 和 HMBC 确认全部碳氢信号归属,故鉴定该化合物为(±)原苏木素 B。

化合物 **2** 白色粉末(三氯甲烷-甲醇);ESI-MS *m/z* 355 $[M+H]^+$, 399 $[M+HCOO]^-$。^{1}H-NMR (400 MHz, DMSO-d_6) δ:7.97 (1H, d, J = 9.6 Hz, H-4), 7.30 (1H, s, H-5), 7.16 (1H, s, H-8), 6.33 (1H, d, J = 9.6 Hz, H-3), 5.39 (1H, d, J = 4.8 Hz, 6′-OH), 5.16 (1H, d, J = 4.4 Hz, 2′-OH), 5.10,5.08 (2H, s, 3′, 4′-OH), 4.60 (1H, m, H-1′), 3.81 (3H, s, C_6-OCH_3), 3.69(1H, m, H-6′b), 3.45 (1H, overlapped, H-6′a), 3.43 (1H, overlapped, H-3′), 3.29 (1H, overlapped, H-5′), 3.27 (1H, overlapped, H-4′), 3.16 (1H, m, H-

2′); ^{13}C-NMR (100 MHz, DMSO-d_6) δ: 160.6 (C-2), 149.9 (C-7), 148.9 (C-9), 144.3 (C-4), 113.3 (C-3), 112.3 (C-10), 109.6 (C-5), 103.0 (C-8), 99.5 (C-1′), 77.1 (C-3′), 76.8 (C-5′), 73.1 (C-2′), 69.6 (C-4′), 60.6 (C-6′), 56.0 (C_6-OCH_3)。以上数据与参考文献[10]报道的东莨菪亭-7-*O*-β-*D*-吡喃葡萄糖苷基本一致,故鉴定该化合物为东莨菪亭-7-*O*-β-*D*-吡喃葡萄糖苷。

化合物 **3** 白色粉末(甲醇); ESI-MS *m/z* 303 [M + H]$^+$, 301 [M − H]$^-$。^{1}H-NMR (400 MHz, D_2O) δ: 7.00 (1H, d, *J* = 8.4 Hz, H-6), 6.55 (1H, br. s, H-2), 6.42 (1H, br. d, *J* = 8.4 Hz, H-5), 4.90 (1H, d, *J* = 6.4 Hz, H-1′), 3.77 (3H, s, C_3-OCH_3); ^{13}C-NMR (100 MHz, D_2O) δ: 155.2 (C-1), 153.1 (C-3), 142.0 (C-4), 120.3 (C-5), 109.8 (C-6), 104.6 (C-2), 104.1 (C-1′), 79.0 (C-3′), 78.5 (C-5′), 75.9 (C-2′), 72.3 (C-4′), 63.4 (C-6′), 58.8 (C_3-OCH_3)。以上数据与参考文献[11]报道的 4-羟基-3-甲氧基苯-1-*O*-β-*D*-吡喃葡萄糖苷基本一致,故鉴定该化合物为 4-羟基-3-甲氧基苯-1-*O*-β-*D*-吡喃葡萄糖苷。

化合物 **4** 无色粉末(三氯甲烷-甲醇); ESI-MS *m/z* 447 [M + H]$^+$, 445 [M − H]$^-$。^{1}H-NMR (500 MHz, CD_3OD) δ: 7.93 (1H, d, *J* = 15.5 Hz, H-4), 6.44 (1H, d, *J* = 15.5 Hz, H-5), 5.77 (1H, s, H-2), 4.34 (1H, d, *J* = 7.5 Hz, H-1″), 4.24 (1H, m, H-3′), 3.78 (1H, br d, *J* = 7.5 Hz, H-7′), 3.73 (1H, d, *J* = 7.5 Hz, H-7′), 3.85 ~ 3.64 (2H, m, H-6″), 3.33 (1H, overlapped, H-3″), 3.29 (1H, overlapped, H-5″), 3.26 (1H, overlapped, H-4″), 3.12 (1H, t, *J* = 7.5 Hz, H-2″), 2.17 (1H, br dd, *J* = 13.2, 6.9 Hz, H-4′), 2.04 (3H, s, H-6), 1.96 (1H, br dd, *J* = 13.2, 6.9 Hz, H-2′), 1.80 (1H, overlapped, H-4′), 1.77 (1H, overlapped, H-2′), 1.14 (3H, s, H-9′), 0.91 (3H, s, H-10′); ^{13}C-NMR (125 MHz, CD_3OD) δ: 147.9 (C-3), 132.6 (C-4), 130.6 (C-5), 120.8 (C-2), 101.7 (C-1″), 86.3 (C-5′), 81.9 (C-8′), 76.7 (C-3″), 76.6 (C-5″), 75.8 (C-7′), 73.8 (C-2″), 72.6 (C-3′), 70.3 (C-4″), 61.4 (C-6″), 48.0 (C-1′), 41.5 (C-2′), 41.4 (C-4′), 19.8 (C-6), 18.4 (C-9′), 15.0 (C-10′)。以上数据与参考文献[12]报道的(1′*R*, 3′*S*, 5′*R*, 8′*S*, 2*Z*,4*E*)-二氢红花菜豆酸-3′-*O*-β-*D*-吡喃葡萄糖苷基本一致,故鉴定该化合物为(1′*R*, 3′*S*, 5′*R*, 8′*S*, 2*Z*,4*E*)-二氢红花菜豆酸-3′-*O*-β-*D*-吡喃葡萄糖苷。

化合物 **5** 无色粉末(三氯甲烷-甲醇); ESI-MS *m/z* 445 [M − H]$^-$, 893 [2M + H]$^+$。^{1}H-NMR (400 MHz, CD_3OD) δ: 7.90 (1H, d, *J* = 16.0 Hz, H-4), 6.34 (1H, d, *J* = 16.0 Hz, H-5), 5.82 (1H, br s, H-2), 4.36 (1H, d, *J* = 8.0 Hz, H-1″), 4.25 (1H, m, H-3′), 3.79 (1H, br d, *J* = 7.6 Hz, H-7′), 3.75 (1H, d, *J* = 7.6 Hz, H-7′), 3.87 ~ 3.66 (2H, m, H-6″), 3.35 (1H, overlapped, H-3″), 3.28 (1H, overlapped, H-5″), 3.27 (1H, overlapped, H-4″), 3.14 (1H, t, *J* = 8.0 Hz, H-2″), 2.18 (1H, br dd, *J* = 13.6, 6.4 Hz, H-4′), 2.01 (3H, s, H-6), 1.98 (1H, overlapped, H-2′), 1.82 (1H, overlapped, H-4′), 1.79 (1H, overlapped, H-2′), 1.16 (3H, s, H-9′), 0.93 (3H, s, H-10′); ^{13}C-NMR (125 MHz, CD_3OD) δ: 144.8 (C-3), 132.7 (C-4), 131.9 (C-5), 125.4 (C-2), 103.1 (C-1″), 87.6 (C-5′), 83.3 (C-8′), 78.0 (C-3″), 78.0 (C-5″), 77.1 (C-7′), 75.1 (C-2″), 73.9 (C-3′), 71.6 (C-4″), 62.7 (C-6″), 49.3 (C-1′), 42.8 (C-2′), 42.7 (C-4′), 20.8 (C-6), 19.7 (C-9′), 16.4 (C-10′)。以上数据与参考文献[12]报道的(1′*R*, 3′*S*, 5′*R*, 8′*S*,2*E*,4*E*)-二氢红花菜豆酸-3′-*O*-β-*D* 吡喃葡萄糖苷基本一致,故鉴定该化合物为(1′*R*, 3′*S*, 5′*R*, 8′*S*,2*E*,4*E*)-二氢红花菜豆酸-3′-*O*-β-*D* 吡喃葡萄糖苷。

化合物 **6** 黄色针晶(甲醇); ESI-MS *m/z* 255 [M − H]$^-$, 511 [2M − H]$^-$。^{1}H-NMR (400 MHz, CD_3OD) δ: 7.72 (1H, d, *J* = 7.2 Hz, H-6), 7.33 ~ 7.30 (2H, m, H-2′, 6′), 6.83 ~ 6.80 (2H, m, H-3′, 5′), 6.49 (1H, dd, *J* = 7.2, 2.0 Hz, H-5), 6.35 (1H, d, *J* = 2.0 Hz, H-8), 5.36 (1H, dd, *J* = 10.4, 2.4 Hz, H-2), 3.07-3.01 (1H, dd, *J* = 13.6, 10.4 Hz, H-3), 2.68 (1H, dd, *J* = 13.6, 2.4 Hz, H-3); ^{13}C-NMR (100 MHz, CD_3OD) δ: 193.5 (C-4), 166.8 (C-7), 165.6 (C-9), 159.0 (C-4′), 131.3 (C-1′), 129.9 (C-5, 2′), 129.0 (C-6′), 116.3 (C-3′, 5′), 115.0 (C-10), 111.7 (C-6), 103.8 (C-8), 81.0 (C-2), 44.9 (C-3)。以上数据与参考文献[13]报道的甘草素基本一致,故鉴定该化合物为甘草素。

化合物 **7** 黄色柱状结晶(甲醇); ESI-MS *m/z* 193 [M + H]$^+$, 191 [M − H]$^-$。^{1}H-NMR (400 MHz, CD_3OD) δ: 7.81 (1H, d, *J* = 7.6 Hz, H-4), 7.06 (1H, s, H-8), 6.73 (1H, s, H-5), 6.17

(1H, d, J = 7.6 Hz, H-3), 3.87 (3H, s, C_6-OCH_3);^{13}C-NMR (100 MHz, CD_3OD) δ:164.0 (C-2), 152.9 (C-7), 151.4 (C-9), 147.1 (C-6), 146.1 (C-4), 112.6 (C-10), 112.5 (C-5), 109.9 (C-3), 103.9 (C-8), 56.8 (C_6-OCH_3)。以上数据与参考文献[14]报道的异莨菪亭基本一致,故鉴定该化合物为异莨菪亭。

化合物**8** 淡黄色柱状结晶(甲醇);ESI-MS m/z 269.0 [M + H]$^+$, 267 [M - H]$^-$。^{1}H-NMR (400 MHz, CD_3OD) δ:7.76 (1H, br d, J = 6.8 Hz, H-5), 7.66 (1H, br s, H-9), 7.21 (2H, d, J = 6.0 Hz, H-2′,6′), 6.85 (2H, d, J = 6.0 Hz, H-3′,5′), 6.49 (1H, br d, J = 6.8 Hz, H-6), 6.27 (1H, br s, H-8), 5.31 (2H, s, H-2);^{13}C-NMR (100 MHz, CD_3OD) δ:183.0 (C-4), 166.6 (C-7), 164.8 (C-8a), 160.5 (C-4′), 138.1 (C-9), 133.5 (C-2′,6′), 130.7 (C-5), 129.6 (C-3), 127.1 (C-1′), 116.7 (C-3′,5′), 115.9 (C-4a), 112.1 (C-6), 103.6 (C-8), 69.0 (C-2)。以上数据与参考文献[15]报道的7-羟基-3-(4′-羟基苄)-色原-4-酮基本一致,故鉴定该化合物为7-羟基-3-(4′-羟基亚苄基)-色原-4-酮。

化合物**9** 黄色粉末(甲醇);ESI-MS m/z 271 [M + H]$^+$, 269 [M - H]$^-$。^{1}H-NMR (400 MHz, CD_3OD) δ: 7.59 (1H, overlapped, H-6′), 7.55 (1H, overlapped, H-β), 7.50 (2H, dd, J = 6.0, 2.0 Hz, H-2, 6), 7.41 (1H, d, J = 12.4 Hz, H-α), 6.82 (2H, d, J = 6.0, 2.0 Hz, H-3, 5), 6.51 (1H, d, J = 2.0 Hz, H-3′), 6.46 (1H, dd, J = 6.8, 2.0 Hz, H-5′), 3.88 (3H, s, $C_{2'}$-OCH_3);^{13}C-NMR (100 MHz, CD_3OD) δ:193.1 (CO), 164.5 (C-4′), 162.5 (C-2′), 161.2 (C-4), 144.1 (C-β), 133.7 (C-6′), 131.4 (C-2, 6), 128.1 (C-1), 125.1 (C-α), 121.8 (C-1′), 116.9 (C-3, 5), 108.9 (C-5′), 100.1 (C-3′), 56.1 ($C_{2'}$-OCH_3)。以上数据与参考文献[7]报道的3-去氧苏木查尔酮基本一致,故鉴定该化合物为3-去氧苏木查尔酮。

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