



密穗马先蒿中黄酮和降倍半萜成分

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[摘要] 目的:研究密穗马先蒿 *Pedicularis densispica* 全草的化学成分。方法:运用多种分离手段,根据理化及波谱(1D, 2D-NMR, MS)数据确定化合物的结构。结果:分离并鉴定了10个化合物,分别为 acacetin (1), apigenin-7-*O*- β -glucopyranoside (2), kaempferol-3,7-*O*- α -dirhamnopyranoside (3), scutellarein-7-*O*- β -glucopyranoside (4), chrysoeriol-7-*O*- β -glucopyranoside (5), pedicutriconic acid (6), dearabinosyl pneumonanthoside (7), salidroside (8), darendoside B (9), maltol- β -*D*-glucopyranoside (10), 化合物类型涉及黄酮、降倍半萜等。结论:以上化合物均为首次从该植物中分离得到,除6,8外,其余化合物均为首次从该属植物中分离得到。

[关键词] 密穗马先蒿;黄酮;降倍半萜

马先蒿属植物600种以上,为多年生或一(二)年生半寄生草本^[1]。马先蒿属植物药用种类繁多,在民间应用历史悠久,疗效优良,享有盛誉^[2]。近年来,该属植物的药用价值逐渐引起研究者的重视,又由于该属植物在世界许多地区都有分布,国内外对该属植物的一些种进行了化学成分的研究,从中分离鉴定环烯醚萜、苯丙素、核苷、黄酮等类型化合物^[3-5],其中环烯醚萜、苯丙素、黄酮等类型化学成分是马先蒿属植物特征性成分,具有抗氧化、抗疲劳及抗肿瘤等生物活性^[6]。药理研究表明密穗马先蒿 *Pedicularis densispica* Franch 对预防运动性贫血的发生有积极的作用^[7]。为揭示密穗马先蒿的生物活性物质基础,本课题组对密穗马先蒿全草进行了化学成分研究。之前已报道了其环烯醚萜成分^[8],本文报道从中分离得到的10个其他化合物。经波谱数据和物理常数分析,这些化合物分别鉴定为 acacetin (1), apigenin-7-*O*- β -glucopyranoside (2), kaempferol-3,7-*O*- α -dirhamnopyranoside (3), scutellarein-7-*O*- β -glucopyranoside (4), chrysoeriol-7-*O*- β -glucopyranoside (5), pedicutriconic acid (6), dearabinosyl pneumonanthoside (7), salidroside (8), darendo-

side B (9), maltol- β -*D*-glucopyranoside (10)。以上化合物均为首次从该植物中分离得到,除6,8外,其余化合物均为首次从该属植物中分离得到。

1 材料

质谱(FAB-MS)用 VG Autospec-3000 型质谱仪测定;核磁共振谱(NMR)用 Bruker AM-400 和 DRX-500 超导核磁共振仪测定,TMS 为内标;薄层色谱板和柱色谱硅胶由青岛海洋化工厂生产;Sephadex LH-20 为 Pharmacia 公司生产;HPLC (Zorbax ODS-C18)。

样品于2004年8月采集于云南省中甸,由中国科学院昆明植物研究所王红研究员鉴定为密穗马先蒿 *P. densispica*。

2 提取与分离

密穗马先蒿全草样品 8.5 kg 粉碎,95%乙醇回流提取3次,每次3h,将提取液减压浓缩得到的浸膏溶于水中,先用石油醚脱脂,再分别以乙酸乙酯、正丁醇萃取,减压浓缩回收溶剂。乙酸乙酯萃取物 52 g 经硅胶(200~300目)柱色谱,氯仿-甲醇梯度洗脱(100:1~20:1),得到 Fr. 1~3 3个组分。Fr. 1 经再次硅胶柱色谱,以石油醚-氯仿(10:1~1:1)洗脱,先后得化合物 1(60 mg),6(20 mg)。Fr. 3 经硅胶柱色谱,以氯仿-甲醇(50:1~30:1)洗脱,得 Fr. 3.1 和 Fr. 3.2 2个流分。Fr. 3.1 经 Sephadex LH-20 凝胶柱色谱,以甲醇洗脱,得化合物 4(7 mg),8(5 mg)。Fr. 3.2 经 Sephadex LH-20 凝胶柱色谱,以甲醇洗脱,得化合物 5(23 mg),10(11 mg)。

[稿件编号] 20110128012

[基金项目] 国家杰出青年科学基金项目(30725048)

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正丁醇萃取物 200 g 硅胶(200~300目)柱色谱,氯仿-甲醇梯度洗脱(30:1~6:1),得到 Fr. A~E 5 个组分。Fr. A 经硅胶柱色谱,以氯仿-甲醇(30:1)洗脱,制备型 HPLC(Zorbax ODS-C18,水-甲醇 80:20,流速 10 mL·min⁻¹)得化合物 **7** (24 mg);Fr. E 经硅胶柱色谱,以氯仿-甲醇(9:1)洗脱,再经 Sephadex LH-20 凝胶柱色谱,甲醇洗脱,得化合物 **2** (20 mg), **3** (50 mg), **9** (11 mg)。

3 结构鉴定

化合物 **1** 黄色粉末状固体, C₁₆H₁₂O₅。FAB-MS *m/z* 283 [M - H]⁻。 ¹H-NMR (DMSO-*d*₆, 500 MHz) δ: 3.81 (3H, s, 4'-OMe), 6.17 (1H, s, H-6), 6.45 (1H, s, H-8), 6.77 (1H, s, H-3), 7.04 (2H, d, *J* = 8.2 Hz, H-3', 5'), 7.95 (2H, d, *J* = 8.2 Hz, H-2', 6'), 12.89 (1H, s, OH-5)。 ¹³C-NMR (DMSO-*d*₆, 125 MHz) δ: 164.3 (s, C-2), 103.5 (d, C-3), 181.8 (s, C-4), 161.5 (s, C-5), 99.0 (d, C-6), 163.3 (s, C-7), 94.1 (d, C-8), 157.4 (s, C-9), 103.8 (s, C-10), 122.9 (s, C-1'), 128.3 (d, C-2', 6'), 114.6 (d, C-3', 5'), 162.3 (s, C-4'), 55.6 (q, 4'-OMe)。以上数据与文献[9]中数据对照基本一致,故鉴定该化合物为 acacetin。

化合物 **2** 黄色粉末状固体, Molish 反应呈阳性, C₂₁H₂₀O₁₀。FAB-MS *m/z* 431 [M - H]⁻。 ¹H-NMR (DMSO-*d*₆, 500MHz) δ: 3.06~3.70 (6H, m, H of Glc), 5.04 (1H, d, *J* = 7.3 Hz, H-1" of Glc), 6.41 (1H, s, H-6), 6.81 (1H, s, H-8), 6.81 (1H, s, H-3), 6.87 (2H, d, *J* = 8.5 Hz, H-3', 5'), 7.91 (2H, d, *J* = 8.5 Hz, H-2', 6')。 ¹³C-NMR (DMSO-*d*₆, 125 MHz) δ: 164.5 (s, C-2), 102.7 (d, C-3), 181.9 (s, C-4), 162.9 (s, C-5), 99.5 (d, C-6), 162.9 (s, C-7), 94.9 (d, C-8), 156.9 (s, C-9), 105.3 (s, C-10), 120.2 (s, C-1'), 128.6 (d, C-2', 6'), 116.3 (d, C-3', 5'), 161.1 (s, C-4'), 100.0 (d, C-1"), 73.1 (d, C-2"), 77.2 (d, C-3"), 69.6 (d, C-4"), 76.5 (d, C-5"), 60.7 (t, C-6")。其数据与文献[10]值一致,鉴定该化合物为 apigenin-7-*O*-β-glucopyranoside。

化合物 **3** 黄色粉末状固体, Molish 反应呈阳性, C₂₇H₃₀O₁₄。FAB-MS *m/z* 577 [M - H]⁻。 ¹H-NMR (CD₃OD + C₅D₅N, 400 MHz) δ: 0.98 (3H, d, *J* = 5.3 Hz, H-6"), 1.28 (3H, d, *J* = 5.9 Hz, H-6"), 3.30~4.36 (8H, m, H of Rha), 5.48 (1H, br s,

H-1"), 5.62 (1H, br s, H-1"), 6.39 (1H, br s, H-6), 6.63 (1H, br s, H-8), 6.95 (2H, d, *J* = 7.9 Hz, H-3', 5'), 7.75 (2H, d, *J* = 7.9 Hz, H-2', 6')。 ¹³C-NMR (CD₃OD + C₅D₅N, 100 MHz) δ: 157.7 (s, C-2), 136.4 (s, C-3), 179.5 (s, C-4), 162.8 (s, C-5), 100.5 (d, C-6), 163.3 (s, C-7), 95.5 (d, C-8), 159.4 (s, C-9), 107.4 (s, C-10), 122.2 (s, C-1'), 132.0 (d, C-2', 6'), 116.6 (d, C-3', 5'), 161.8 (s, C-4'), 103.6 (d, C-1"), 72.1 (d, C-2"), 72.2 (d, C-3"), 73.6 (d, C-4"), 71.7 (d, C-5"), 18.3 (q, C-6"), 99.9 (d, C-1"), 71.9 (d, C-2"), 72.1 (d, C-3"), 73.2 (d, C-4"), 71.3 (d, C-5"), 17.9 (q, C-6")。以上数据与文献[11]中数据对照基本一致,故鉴定该化合物为 kaempferol-3,7-*O*-α-dirhamnopyranoside。

化合物 **4** 黄色粉末状固体, Molish 反应呈阳性, C₂₁H₂₀O₁₁。FAB-MS *m/z* 448 [M]⁻。 ¹H-NMR (DMSO + CD₃OD, 400 MHz) δ: 3.21~3.70 (6H, m, H of Glc), 5.25 (1H, d, *J* = 7.0 Hz, H-1" of Glc), 6.18 (1H, s, H-8), 6.37 (1H, s, H-3), 6.88 (2H, d, *J* = 8.8 Hz, H-3', 5'), 8.05 (2H, d, *J* = 8.8 Hz, H-2', 6')。 ¹³C-NMR (DMSO + CD₃OD, 100 MHz) δ: 164.3 (s, C-2), 103.9 (d, C-3), 179.3 (s, C-4), 158.6 (s, C-5), 135.3 (s, C-6), 161.6 (s, C-7), 95.0 (d, C-8), 158.8 (s, C-9), 105.4 (s, C-10), 122.8 (s, C-1'), 132.3 (d, C-2', 6'), 116.1 (d, C-3', 5'), 163.0 (s, C-4'), 100.2 (d, C-1"), 75.7 (d, C-2"), 78.5 (d, C-3"), 71.3 (d, C-4"), 78.0 (d, C-5"), 62.6 (t, C-6")。以上数据与文献[12]中数据对照基本一致,故鉴定该化合物为 scutellarein-7-*O*-β-glucopyranoside。

化合物 **5** 黄色粉末状固体, Molish 反应呈阳性, C₂₂H₂₂O₁₁。 ¹H-NMR (DMSO-*d*₆, 400 MHz) δ: 3.19~3.72 (6H, m, H of Glc), 3.85 (3H, s, 3'-OMe), 5.09 (1H, d, *J* = 6.8 Hz, H-1"), 6.44 (1H, d, *J* = 1.6 Hz, H-6), 6.78 (1H, s, H-8), 6.79 (1H, s, H-3), 7.06 (1H, d, *J* = 8.5 Hz, H-5'), 7.42 (1H, d, *J* = 1.7 Hz, H-2'), 7.52 (1H, dd, *J* = 8.5, 1.7 Hz, H-6'), 9.43 (1H, s, OH-4'), 12.91 (1H, s, OH-5)。 ¹³C-NMR (DMSO-*d*₆, 100 MHz) δ: 164.1 (s, C-2), 103.8 (d, C-3), 182.0 (s, C-4), 163.0 (s, C-5), 100.0 (d, C-6), 161.2 (s, C-7), 94.9 (d, C-8), 157.0 (s, C-9), 105.4 (s, C-10), 122.9 (s, C-1'), 112.1 (d, C-2'), 146.8 (s, C-3'), 151.4 (s, C-4'), 113.2 (d, C-5'), 118.9



(d, C-6'), 55.8 (q, 3'-OMe), 99.6 (d, C-1''), 73.2 (d, C-2''), 76.5 (d, C-3''), 69.6 (d, C-4''), 77.2 (d, C-5''), 60.7 (t, C-6''). 其数据与文献[13]相符, 化合物鉴定为 *chrysoeriol-7-O-β-glucopyranoside*。

化合物 6 黄色粉末状固体, C₁₆H₁₄O₆。FAB-MS *m/z* 301 [M - H]⁻。¹H-NMR (CDCl₃, 500 MHz) δ: 3.76 (3H, s, 3-OMe), 3.80 (3H, s, 2-OMe), 3.87 (3H, s, 5-OMe), 6.46 (1H, s, H-1), 7.07 (1H, dd, *J* = 1.0, 7.8 Hz, H-6), 7.14 (1H, t, *J* = 7.8 Hz, H-7), 7.65 (1H, dd, *J* = 1.0, 7.8 Hz, H-8), 12.60 (1H, s, OH-4)。¹³C-NMR (CDCl₃, 125 MHz) δ: 91.0 (d, C-1), 160.0 (s, C-2), 132.0 (s, C-3), 154.1 (s, C-4), 153.2 (s, C-4a), 146.3 (s, C-4b), 148.3 (s, C-5), 115.6 (d, C-6), 123.6 (d, C-7), 116.7 (d, C-8), 121.2 (s, C-8a), 104.3 (s, C-8b), 181.1 (s, C-9), 56.5 (q, 2-OMe), 60.9 (q, 3-OMe), 56.4 (q, 5-OMe)。其数据与文献[14]相符, 化合物鉴定为 *pedicutricone A*。

化合物 7 无色固体, Molish 反应显阳性, C₁₉H₃₀O₇。FAB-MS *m/z* 369 [M - H]⁻。¹H-NMR (C₅D₅N, 400 MHz) δ: 0.88 (3H, s, H-12), 0.91 (3H, s, H-11), 1.37 (3H, d, *J* = 6.3 Hz, H-10), 1.72 (3H, s, H-13), 2.10 (1H, d, *J* = 16.4 Hz, H-2a), 2.41 (1H, d, *J* = 16.4 Hz, H-2b), 2.44 (1H, d, *J* = 10.9 Hz, H-6), 3.59 (1H, s, H-2'), 3.90 (1H, m, H-5'), 4.04 (1H, m, H-4'), 4.25 (1H, m, H-6'a), 4.38 (1H, m, H-3'), 4.50 (1H, d, *J* = 11 Hz, H-6'b), 4.65 (1H, m, H-9), 4.93 (1H, d, *J* = 7.7 Hz, H-1'), 5.61 (1H, dd, *J* = 15.4, 9.3 Hz, H-7), 5.85 (1H, dd, *J* = 15.4, 6.5 Hz, H-8), 5.97 (1H, s, H-4)。¹³C-NMR (C₅D₅N, 100 MHz) δ: 36.1 (s, C-1), 47.9 (t, C-2), 198.2 (s, C-3), 126.1 (d, C-4), 161.5 (s, C-5), 55.5 (d, C-6), 127.8 (d, C-7), 137.6 (d, C-8), 76.0 (d, C-9), 21.2 (q, C-10), 27.2 (q, C-11), 27.8 (q, C-12), 23.2 (q, C-13), 102.8 (d, C-1'), 75.4 (d, C-2'), 78.6 (d, C-3'), 71.7 (d, C-4'), 78.5 (d, C-5'), 62.8 (t, C-6')。其数据与文献[15]值相符, 化合物鉴定为 *dearabinosyl pneumonanthoside*。

化合物 8 无色固体, Molish 反应呈阳性, C₁₄H₂₀O₇。FAB-MS *m/z* 299 [M - H]⁻。¹H-NMR (CD₃OD, 500 MHz) δ: 2.83 (1H, m, H-7a), 3.17 (1H, t, *J* = 8.4 Hz, H-7b), 3.25 ~ 3.34 (2H, m, H-8), 3.25 ~ 4.01 (6H, m, H of Glc), 4.28 (1H, d, *J* =

7.8 Hz, H-1' of Glc), 6.68 (2H, d, *J* = 8.3 Hz, H-3, 5), 7.06 (2H, d, *J* = 8.3 Hz, H-2, 6)。¹³C-NMR (CD₃OD, 125 MHz) δ: 156.8 (s, C-1), 116.1 (d, C-2, 6), 130.9 (d, C-3, 5), 130.8 (s, C-4), 36.4 (t, C-7), 72.1 (t, C-8), 104.4 (d, C-1'), 75.1 (d, C-2'), 78.1 (d, C-3'), 71.7 (d, C-4'), 78.0 (d, C-5'), 62.8 (t, C-6')。以上数据与文献[16]中数据对照基本一致, 故鉴定该化合物为 *salidroside*。

化合物 9 无色固体, Molish 反应呈阳性, C₂₁H₃₂O₁₂。FAB-MS *m/z* 475 [M - H]⁻。¹H-NMR (CD₃OD, 400 MHz) δ: 1.24 (3H, d, *J* = 6.2 Hz, H-6''), 2.80 (2H, t, *J* = 7.0 Hz, H-7), 3.80 (3H, s, OMe), 3.25 ~ 4.02 (m, H of sugar and H-8), 4.28 (1H, d, *J* = 7.9 Hz, H-1' of Glc), 5.14 (1H, d, *J* = 1.3 Hz, H-1'' of Rha), 6.67 (1H, dd, *J* = 8.2, 2.0 Hz, H-6), 6.72 (1H, d, *J* = 2.0 Hz, H-2), 6.81 (1H, d, *J* = 8.2 Hz, H-5)。¹³C-NMR (CD₃OD, 100 MHz) δ: 133.0 (s, C-1), 117.1 (d, C-2), 147.5 (s, C-3), 147.4 (s, C-4), 112.9 (d, C-5), 121.1 (d, C-6), 36.5 (t, C-7), 71.9 (t, C-8), 104.2 (d, C-1'), 75.6 (d, C-2'), 84.6 (d, C-3'), 70.2 (d, C-4'), 77.8 (d, C-5'), 62.7 (t, C-6'), 102.8 (d, C-1''), 72.3 (d, C-2''), 72.2 (d, C-3''), 74.0 (d, C-4''), 70.1 (d, C-5''), 17.9 (q, C-6'')。以上数据与文献[17]中数据对照基本一致, 故鉴定该化合物为 *darendoside B*。

化合物 10 无色固体, Molish 反应呈阳性, C₁₂H₁₆O₈。FAB-MS *m/z* 287 [M - H]⁻。¹H-NMR (CD₃OD, 500 MHz) δ: 2.46 (3H, s, H-7), 3.25 ~ 3.83 (6H, m, H of Glc), 4.80 (1H, d, *J* = 7.2 Hz, H-1' of Glc), 6.45 (1H, d, *J* = 5.6 Hz, H-5), 8.00 (1H, d, *J* = 5.6 Hz, H-6)。¹³C-NMR (CD₃OD, 125 MHz) δ: 164.6 (s, C-2), 143.7 (s, C-3), 177.2 (s, C-4), 117.3 (d, C-5), 157.1 (d, C-6), 15.8 (q, C-7), 105.5 (d, C-1'), 75.5 (d, C-2'), 78.6 (d, C-3'), 71.2 (d, C-4'), 78.1 (d, C-5'), 62.6 (t, C-6')。其数据与文献[18]相符, 化合物鉴定为 *maltol-β-D-glucopyranoside*。

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Flavonoids and nor-sesquiterpenes of *Pedicularis densispica*

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[Abstract] **Objective:** To study the chemical constituents of the whole plants of *Pedicularis densispica*. **Method:** The chemical constituents were isolated by various chromatographic methods and their structures were determined by chemical evidences and spectral data. **Result:** Ten compounds were isolated and identified as acacetin (1), apigenin-7-O- β -glucopyranoside (2), kaempferol-3,7-O- α -dirhamnopyranoside (3), scutellarein-7-O- β -glucopyranoside (4), chrysoeriol-7-O- β -glucopyranoside (5), pedicutriconic A (6), dearabinosyl pneumonanthoside (7), salidroside (8), darendoside B (9), and maltol- β -D-glucopyranoside (10). **Conclusion:** These compounds were isolated from the titled plant for the first time. Except compounds 6 and 8, the others were obtained for the first time from the genus *Pedicularis*.

[Key words] *Pedicularis densispica*; flavonoids; nor-sesquiterpenes

doi:10.4268/cjcm20111914

[责任编辑 丁广治]