

显脉香茶菜中二萜类成分研究

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摘要: 目的 对显脉香茶菜 *Rabdosia nervosa* 的化学成分进行研究。方法 采用现代色谱方法进行分离、纯化, 通过现代波谱技术对化合物进行结构鉴定。**结果** 从显脉香茶菜中又分离得到 10 个二萜类化合物, 分别鉴定为长管贝壳杉素 E (1)、牛尾草乙素 (2)、parvifoline G (3)、四川香茶菜丁素 (4)、黄花香茶菜甲素 (5)、黄花香茶菜乙素 (6)、毛果香茶菜贝壳松醇 (7)、延命草醇 (8)、 11β -hydroxy-6, 7-seco-6, 19: 6, 20-diepoxy-1 α , 7-olide-*ent*-kaur-15-one (9)、isodocarpin (10)。**结论** 化合物 1~9 为首次从该植物中分得。

关键词: 显脉香茶菜; 二萜; 长管贝壳杉素 E; 牛尾草乙素; 四川香茶菜丁素

中图分类号: R284.14 文献标志码: A 文章编号: 0253-2670(2012)02-0247-04

Studies on diterpenoids from *Rabdosia nervosa*

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Key words: *Rabdosia nervosa* (Hemsl.) C. Y. Wu et H. W. Li; diterpenoids; longikaurin E; isodoternifolin B; rabdosichuanin D

显脉香茶菜 *Rabdosia nervosa* (Hemsl.) C. Y. Wu et H. W. Li 为唇形科香茶菜属多年生草本植物, 又称兰花柴胡、蛇总管和大叶蛇总管。在民间常以茎、叶入药, 具有清热解毒, 除湿之功效, 治疗急性传染性肝炎、毒蛇咬伤、皮肤瘙痒等疾病^[1]。

对不同产地的显脉香茶菜进行化学成分研究表明, 由于生长环境不同, 所得到的次生代谢产物有较大差异^[2-4]。本课题组曾从江西产显脉香茶菜中分离得到多种二萜类成分^[5-7], 为进一步寻找其活性成分, 对产于江西宜丰地区的显脉香茶菜化学成分进行研究, 从其茎叶的乙醇提取物中又分离得到 10 个二萜类成分, 分别鉴定为长管贝壳杉素 E (longikaurin E, 1)、牛尾草乙素 (isodoternifolin B, 2)、小叶香茶菜素 G (parvifoline G, 3)、四川香茶菜丁素 (rabdosichuanin D, 4)、黄花香茶菜甲素 (sculponeatin A, 5)、黄花香茶菜乙素 (sculponeatin B, 6)、毛果香茶菜贝壳松醇 (lasiokaurinol, 7)、延命草醇 (enmenol, 8)、 11β -hydroxy-6, 7-seco-6, 19: 6, 20-diepoxy-1 α , 7-olide-*ent*-kaur-15-one (9)、毛果青茶菜素 (isodocarpin, 10)。其中, 化合物 1~9 为首次从该植物中分离得到。

1 材料与仪器

XT—4A 显微熔点测定仪, Eqvinoxtm55—A590/3F 型红外分光光度计, Bruker Avance—400 超导核磁共振仪; 柱色谱硅胶和薄层色谱用硅胶(青岛海浪化工有限公司); Pharmadex LH-20 (安玛西亚生物技术上海有限公司); 化学试剂均为分析纯。显脉香茶菜药材于 2007 年 7 月采自江西宜丰地区, 经江西中医学院赖学文教授鉴定为显脉香茶菜 *Rabdosia nervosa* (Hemsl.) C. Y. Wu et H. W. Li。

2 提取与分离

干燥的显脉香茶菜茎、叶 10 kg 以 95% 乙醇提取 3 次, 每次 2 h, 合并提取液, 并减压浓缩至稠膏, 用甲醇溶解, 滤除不溶物, 滤液加 0.4% 的活性炭加热煮沸脱色 3 次, 抽滤, 浓缩为浸膏 (800 g)。与硅藻土 1:1 拌样, 置改良索氏提取器分为石油醚 (50 g)、醋酸乙酯 (270 g)、丙酮 (200 g)、甲醇 (230 g) 4 个部位。石油醚部位 40 g 经硅胶柱色谱, 以石油醚-醋酸乙酯梯度洗脱, 8:1 部分抽滤得到化合物 1 (21 mg); 7:1 部分经反复重结晶得化合物 2 (8 mg)。醋酸乙酯部分 150 g 经硅胶柱色谱, 以氯仿-丙酮梯度洗脱, 20:1 部分经过 Sephadex LH-20 柱, 氯仿-

收稿日期: 2011-05-18

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甲醇 (1:1) 洗脱得化合物 **3** (33 mg); 15:1 部分再经过 ODS 柱色谱得化合物 **4** (10 mg)、**10** (7 mg); 12:1 部分经反复硅胶柱色谱得化合物 **5** (20 mg)、**6** (49 mg)、**9** (10 mg); 10:1 部分经过 Sephadex LH-20 柱, 氯仿-甲醇 (1:1) 洗脱, 经重结晶得化合物 **7** (26 mg) 和 **8** (17 mg)。

3 结构鉴定

化合物 1: 无色绒毛状结晶 (丙酮-甲醇), mp 255~256 °C。C₂₂H₃₀O₆。IR $\nu_{\text{max}}^{\text{KBr}}$ (cm⁻¹): 3 288, 1 731, 1 702, 1 650, 1 250, 895。¹H-NMR (400 MHz, C₅D₅N) δ : 8.88 (1H, s, 7-OH), 6.63 (1H, d, J = 11.2 Hz, 6 β -OH), 6.01 (1H, s, H-17a), 5.33 (1H, s, H-17b), 5.47 (1H, m, H-11 β), 4.33 (1H, dd, J = 7.5, 11.2 Hz, H-6 α), 4.27 (1H, d, J = 9.6 Hz, H-20a), 4.23 (1H, d, J = 9.6 Hz, H-20b), 3.60 (1H, d, J = 7.0 Hz, H-5 β), 2.10 (3H, s, -OAc), 1.18 (3H, s, 18-CH₃), 1.14 (3H, s, 19-CH₃); ¹³C-NMR (100 MHz, C₅D₅N) δ : 34.2 (C-1), 18.5 (C-2), 41.6 (C-3), 33.7 (C-4), 54.0 (C-5), 74.9 (C-6), 95.1 (C-7), 58.6 (C-8), 58.8 (C-9), 37.3 (C-10), 69.1 (C-11), 38.1 (C-12), 31.4 (C-13), 26.5 (C-14), 208.6 (C-15), 152.1 (C-16), 118.3 (C-17), 33.8 (C-18), 22.8 (C-19), 68.2 (C-20), 170.0, 21.8 (-OAc)。其波谱数据与文报道献基本一致^[8], 故鉴定化合物 **1** 为长管贝壳杉素 E。

化合物 2: 无色针晶 (甲醇), mp 238~239 °C。C₂₂H₃₂O₆。IR $\nu_{\text{max}}^{\text{KBr}}$ (cm⁻¹): 3 324, 1 645, 1 240, 902。¹H-NMR (400 MHz, C₅D₅N) δ : 8.80 (1H, s, OH-7 β), 6.70 (1H, d, J = 11.2 Hz, OH-6 β), 5.22 (1H, t, J = 5.2 Hz, H-15 α), 5.13 (1H, brs, H-17a), 5.10 (1H, brs, H-17b), 4.26 (1H, dd, J = 1.6, 8.8 Hz, H-20a), 4.10 (1H, dd, J = 1.6, 8.8 Hz, H-20b), 3.99 (1H, d, J = 7.0 Hz, H-6 α), 2.69 (1H, dd, J = 4.8, 9.0 Hz, H-13 α), 1.50 (1H, d, J = 7.0 Hz, H-5 β), 2.07 (3H, s, -OAc), 1.11 (3H, s, 18-CH₃), 1.02 (3H, s, 19-CH₃); ¹³C-NMR (100 MHz, C₅D₅N) δ : 30.8 (C-1), 18.5 (C-2), 41.0 (C-3), 33.5 (C-4), 56.0 (C-5), 74.0 (C-6), 97.2 (C-7), 50.6 (C-8), 45.7 (C-9), 36.3 (C-10), 68.3 (C-11), 41.3 (C-12), 35.5 (C-13), 26.0 (C-14), 74.1 (C-15), 159.1 (C-16), 108.3 (C-17), 33.8 (C-18), 22.3 (C-19), 68.9 (C-20), 169.8, 21.9 (-OAc)。以上数据与文献报道基本一致^[9], 故鉴定化合物 **2** 为牛尾草乙素。

化合物 3: 白色针状结晶 (甲醇-丙酮), mp 151~152 °C。C₂₆H₃₄O₉。IR $\nu_{\text{max}}^{\text{KBr}}$ (cm⁻¹): 3 425, 3 220, 1 726,

1 664, 1 640。¹H-NMR (400 MHz, C₅D₅N) δ : 9.2 (1H, s, OH-7 β), 5.98 (1H, t, J = 2.5 Hz, H-15 α), 5.17 (1H, dd, J = 8.5, 12 Hz, H-1 β), 5.15 (1H, d, J = 3.8 Hz, H-11 β), 5.01 (2H, brs, H-17), 5.02 (1H, dd, J = 2.0, 9.4 Hz, H-20a), 4.35 (1H, dd, J = 2.0, 9.4 Hz, H-20b), 3.58 (1H, s, H-5 β), 2.76 (1H, d, J = 12 Hz, H-13 α), 2.65 (1H, dd, J = 4.7, 8.6 Hz, H-9 β), 2.14, 2.12, 2.07 (各 3H, s, 3 \times -OAc) 1.47 (3H, s, 18-CH₃), 1.05 (3H, s, 19-CH₃); ¹³C-NMR (100 MHz, C₅D₅N) δ : 76.0 (C-1), 24.5 (C-2), 38.5 (C-3), 34.0 (C-4), 62.0 (C-5), 206.9 (C-6), 93.7 (C-7), 48.7 (C-8), 46.5 (C-9), 42.3 (C-10), 69.3 (C-11), 40.8 (C-12), 35.2 (C-13), 25.4 (C-14), 75.4 (C-15), 155.0 (C-16), 109.3 (C-17), 34.2 (C-18), 21.6 (C-19), 64.8 (C-20), 170.0, 169.5, 169.3, 21.2, 21.3, 21.4 (3 \times -OAc)。以上数据与文献报道基本一致^[10], 鉴定化合物 **3** 为小叶香茶菜素 G。

化合物 4: 无色块状结晶 (甲醇-丙酮), mp 240~242 °C。C₂₄H₃₄O₈。IR $\nu_{\text{max}}^{\text{KBr}}$ (cm⁻¹): 3 425, 3 220, 1 726, 1 614, 1 250, 1 025。¹H-NMR (400 MHz, C₅D₅N) δ : 8.20, 6.63 (各 1H, brs, 2 \times -OH), 5.48 (1H, brs, H-17a), 5.11 (1H, brs, H-17b), 5.17 (1H, m, H-11 β), 5.06 (1H, dd, J = 5.0, 11.2 Hz, H-1 β), 4.88 (1H, brs, H-15 α), 4.84 (1H, d, J = 9.0 Hz, H-20a), 4.33 (1H, d, J = 9.0 Hz, H-20b), 4.40 (1H, d, J = 7.0 Hz, H-6 α), 2.72 (1H, m, H-13 α), 1.97 (1H, d, J = 7.0 Hz, H-5 β), 2.13, 2.09 (各 3H, s, 2 \times -OAc), 1.25 (3H, s, 18-CH₃), 1.15 (3H, s, 19-CH₃); ¹³C-NMR (100 MHz, C₅D₅N) δ : 77.0 (C-1), 25.5 (C-2), 38.6 (C-3), 33.6 (C-4), 55.7 (C-5), 75.7 (C-6), 97.1 (C-7), 51.9 (C-8), 46.9 (C-9), 40.7 (C-10), 70.5 (C-11), 41.6 (C-12), 35.6 (C-13), 26.6 (C-14), 74.8 (C-15), 160.8 (C-16), 108.1 (C-17), 33.9 (C-18), 22.5 (C-19), 65.1 (C-20), 170.4, 170.1, 22.2, 21.9 (2 \times -OAc)。其波谱数据与文献报道基本一致^[11], 故鉴定化合物 **4** 为四川香茶菜丁素。

化合物 5: 白色块状结晶 (甲醇), mp 302~304 °C。C₂₀H₂₄O₆。IR $\nu_{\text{max}}^{\text{KBr}}$ (cm⁻¹): 1 734, 1 710, 1 640。¹H-NMR (400 MHz, C₅D₅N) δ : 7.15 (1H, brs, 11 β -OH), 6.20 (1H, d, J = 5.0 Hz, H-6 β), 6.08 (1H, brs, H-17a), 5.39 (1H, brs, H-17b), 5.68 (1H, dd, J = 6.0, 10.0 Hz, H-1 β), 4.68 (1H, dd, J = 4.2, 5.0 Hz, H-11 α), 4.38 (1H, d, J = 10.0 Hz, H-20a), 4.23 (1H, d, J = 10.0 Hz, H-20b), 4.11 (1H, d, J = 8.0 Hz, H-19a), 3.56 (1H, d, J = 8.0 Hz, H-19b), 3.69 (1H, d, J = 11.0 Hz,

H-14 β), 3.22 (1H, dd, J = 5.2, 9.0 Hz, H-13 β), 2.92 (1H, d, J = 5.0 Hz, H-5 β), 2.46 (1H, dd, J = 8.9, 14.0 Hz, H-12 β), 2.27 (1H, d, J = 4 Hz, H-9 α), 1.1 (3H, s, 18-CH₃); ¹³C-NMR (100 MHz, C₅D₅N) δ : 78.1 (C-1), 22.2 (C-2), 28.7 (C-3), 40.8 (C-4), 52.8 (C-5), 110.3 (C-6), 170.4 (C-7), 54.8 (C-8), 44.7 (C-9), 49.2 (C-10), 64.1 (C-11), 39.9 (C-12), 34.0 (C-13), 33.1 (C-14), 200.0 (C-15), 149.9 (C-16), 118.1 (C-17), 30.0 (C-18), 71.3 (C-19), 76.1 (C-20)。其波谱数据与文献报道基本一致^[12], 鉴定化合物**5**为黄花香茶菜甲素。

化合物6:白色针晶(甲醇-丙酮), mp 243~245 °C。C₂₀H₂₆O₆。IR $\nu_{\text{max}}^{\text{KBr}}$ (cm⁻¹): 3 435, 1 751, 1 640。¹H-NMR (400 MHz, C₅D₅N) δ : 6.42 (1H, brs, 11 β -OH), 6.20 (1H, d, J = 5.0 Hz, H-6 β), 5.90 (1H, dd, J = 6.0, 10.0 Hz, H-1 β), 5.71 (1H, brs, H-15 β), 5.55 (1H, brs, H-17a), 5.24 (1H, brs, H-17b), 4.60 (1H, dd, J = 4.0, 4.0 Hz, H-11 α), 4.58 (1H, ABd, J = 8.9 Hz, H-20a), 4.23 (1H, ABd, J = 8.9 Hz, H-20b), 4.11 (1H, d, J = 8.0 Hz, H-19a), 3.56 (1H, d, J = 8.0 Hz, H-19b), 3.19 (1H, d, J = 11.0 Hz, H-14 β), 3.02 (1H, d, J = 5.2 Hz, H-13 β), 2.99 (1H, d, J = 5.0 Hz, H-5 β), 2.46 (1H, dd, J = 8.9, 14.0 Hz, H-12 β), 2.80 (1H, d, J = 4.0 Hz, H-9 α), 1.10 (3H, s, 18-CH₃); ¹³C-NMR (100 MHz, C₅D₅N) δ : 80.4 (C-1), 24 (C-2), 30.1 (C-3), 42.2 (C-4), 56.7 (C-5), 112.1 (C-6), 176.2 (C-7), 52.7 (C-8), 40.5 (C-9), 50.8 (C-10) 65.6 (C-11), 45.7 (C-12), 38.0 (C-13), 34.5 (C-14), 78.9 (C-15), 160.0 (C-16), 108.9 (C-17), 31.3 (C-18), 73.1 (C-19), 77.5 (C-20)。以上数据与文献报道一致^[12], 鉴定化合物**6**为黄花香茶菜乙素。

化合物7:白色块状结晶(甲醇), mp 221~224 °C。C₂₂H₃₂O₇。IR $\nu_{\text{max}}^{\text{KBr}}$ (cm⁻¹): 3 545, 3 320, 1 726, 1 240。¹H-NMR (400 MHz, C₅D₅N) δ : 8.50, 8.01, 6.55 (各 1H, brs, 3×-OH), 5.70 (2H, brs, H-17), 5.35 (1H, brs, H-15 α), 5.09 (1H, s, H-14 α), 4.87 (1H, dd, J = 8.0, 1.0 Hz, H-1 β), 4.57 (1H, d, J = 10.0 Hz, H-20a), 4.39 (1H, d, J = 10.0 Hz, H-20b), 4.25 (1H, d, J = 6.0 Hz, H-6 α), 3.10~2.80 (2H, m, H-9 β , 13 α), 2.02 (3H, s, -OAc), 1.58 (1H, d, J = 6.0 Hz, H-5 β), 1.15 (3H, s, 18-CH₃), 1.14 (3H, s, 19-CH₃); ¹³C-NMR (100 MHz, C₅D₅N) δ : 76.9 (C-1), 26.0 (C-2), 38.8 (C-3), 34.2 (C-4), 58.2 (C-5), 73.8 (C-6), 100.2 (C-7), 53.9 (C-8), 46.7 (C-9), 40.2 (C-10), 17.2 (C-11), 33.3 (C-12), 44.8

(C-13), 76.4 (C-14), 73.4 (C-15), 161.3 (C-16), 109.7 (C-17), 33.2 (C-18), 22.3 (C-19), 63.5 (C-20), 170.4, 21.9 (-OAc)。其波谱数据与文献报道一致^[13], 鉴定化合物**7**为香茶菜贝壳松醇。

化合物8:无色块状结晶(氯仿-甲醇), mp 248~249 °C。C₂₀H₃₀O₆。IR $\nu_{\text{max}}^{\text{KBr}}$ (cm⁻¹): 3 324, 3 245, 1 651, 1 040。¹H-NMR (400 MHz, C₅D₅N) δ : 8.54, 7.88, 7.84, 6.55 (各 1H, brs, 4×-OH), 5.78 (1H, s, H-17a), 5.70 (1H, s, H-17b), 5.40 (1H, s, H-15 α), 5.19 (1H, s, H-14 α), 4.87 (1H, d, J = 10.0 Hz, H-20a), 4.49 (1H, d, J = 10.0 Hz, H-20b), 4.32 (1H, d, J = 5.0 Hz, H-6 α), 3.77 (1H, s, H-1 β), 1.25 (3H, s, 18-CH₃), 1.20 (3H, s, 19-CH₃); ¹³C-NMR (100 MHz, C₅D₅N) δ : 73.3 (C-1), 31.0 (C-2), 39.6 (C-3), 34.1 (C-4), 57.7 (C-5), 73.7 (C-6), 99.1 (C-7), 53.2 (C-8), 46.3 (C-9), 41.6 (C-10), 18.4 (C-11), 33.4 (C-12), 45.1 (C-13), 76.4 (C-14), 73.1 (C-15), 161.2 (C-16), 109.7 (C-17), 32.6 (C-18), 21.7 (C-19), 65.4 (C-20)。其波谱数据与文献报道基本一致^[14], 鉴定化合物**8**为延命草醇。

化合物9:白色颗粒(甲醇-丙酮), mp 231~233 °C。C₂₀H₂₆O₆。IR $\nu_{\text{max}}^{\text{KBr}}$ (cm⁻¹): 1 734, 1 710, 1 010。¹H-NMR (400 MHz, C₅D₅N) δ : 6.91 (1H, brs, 11 β -OH), 6.10 (1H, d, J = 5.0 Hz, H-6 β), 5.50 (1H, dd, J = 6.0, 10.0 Hz, H-1 β), 4.41 (1H, dd, J = 4.2, 5.0 Hz, H-11 α), 4.27 (1H, d, J = 10 Hz, H-20a), 4.16 (1H, d, J = 10 Hz, H-20b), 3.99 (1H, d, J = 8.0 Hz, H-19a), 3.44 (1H, d, J = 8.0 Hz, H-19b), 3.74 (1H, d, J = 11.0 Hz, H-14 β), 3.59 (1H, dd, J = 5.2, 9.0 Hz, H-13 β), 2.94 (1H, d, J = 5.0 Hz, H-5 β), 2.14 (1H, dd, J = 8.9, 14.0 Hz, H-12 β), 1.01 (3H, s, 18-CH₃), 0.98 (3H, s, 17-CH₃); ¹³C-NMR (100 MHz, C₅D₅N) δ : 78.5 (C-1), 22.8 (C-2), 28.9 (C-3), 41.2 (C-4), 53.7 (C-5), 111.2 (C-6), 171.6 (C-7), 56.1 (C-8), 46.5 (C-9), 49.8 (C-10), 64.3 (C-11), 30.1 (C-12), 32.4 (C-13), 35.0 (C-14), 215.1 (C-15), 49.2 (C-16), 10.4 (C-17), 30.1 (C-18), 72.5 (C-19), 76.7 (C-20)。波谱数据与文献报道基本一致^[12], 鉴定化合物**9**为11 β -hydroxy-6, 7-seco-6, 19: 6, 20-diepoxy-1 α , 7-olate-*ent*-kaur-15-one。

化合物10:黄白色颗粒(甲醇), mp 270~272 °C。C₂₀H₂₆O₅。IR $\nu_{\text{max}}^{\text{KBr}}$ (cm⁻¹): 3 435, 1 751, 1 640, 1 055, 899。¹H-NMR (400 MHz, CDCl₃) δ : 6.07 (1H, s, H-17a), 5.50 (1H, s, H-17b), 5.35 (1H, s, H-6 α), 4.41 (1H, dd, J = 6.0, 11.5 Hz, H-1 β), 4.08 (1H, d, J =

8.9 Hz, H-20a), 3.99 (1H, d, $J = 8.9$ Hz, H-20b), 3.12 (1H, dd, $J = 4.0, 9.0$ Hz, H-13 β), 2.54 (1H, dd, $J = 5.0, 13.0$ Hz, H-9 α), 2.40 (1H, d, $J = 12$ Hz, H-14 β), 2.09 (1H, dd, $J = 4.0, 11.9$ Hz, H-14 α), 1.96 (1H, s, H-5 β), 1.03 (3H, s, 18-CH₃), 0.98 (3H, s, 19-CH₃); ¹³C-NMR (400 MHz, CDCl₃) δ : 76.4 (C-1), 23.4 (C-2), 37.1 (C-3), 32.7 (C-4), 53.7 (C-5), 101.4 (C-6), 171.5 (C-7), 55.8 (C-8), 45.5 (C-9), 49.5 (C-10), 19.6 (C-11), 35.0 (C-12), 34.7 (C-13), 29.0 (C-14), 199.9 (C-15), 150.0 (C-16), 118.0 (C-17), 32.8 (C-18), 23.1 (C-19), 73.5 (C-20)。其波谱数据与文献报道基本一致^[15], 鉴定化合物 **10** 为毛果青茶菜素。

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