

金丝李茎皮化学成分研究

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摘要: 目的 对金丝李 *Garcinia paucinervis* 的茎皮化学成分进行分离鉴定, 并进行抗肿瘤活性筛选。方法 采用硅胶、反相 RP-18, 制备薄层色谱, Sephadex LH-20 等柱色谱方法对乙醇提取物石油醚萃取部位化学成分进行分离和纯化, 并根据其理化性质及波谱数据鉴定结构; 采用 MTT 法进行抗肿瘤活性筛选。结果 从金丝李乙醇提取物中分离得到 10 个化合物, 分别鉴定为 cambogin (1)、焦袂康酸 (2)、 β -谷甾醇 (3)、胡萝卜昔 (4)、7-prenyljacareubin (5)、parvifolixanthone A (6)、formoxanthone A (7)、termicalcicolanone A (8)、1, 3, 5, 6-tetrahydroxy-4-prenylxanthone (9)、isogarcinol (10)。抗肿瘤活性筛选结果表明, 化合物 5 和 7 对 HL-60、SMMC-7721、A549、MCF-7 和 SW480 细胞株有一定的抑制作用。结论 化合物 2~5 和 7~10 均为首次从该植物中分离得到, 同时首次报道了化合物 5 的 ¹³C-NMR 数据。

关键词: 金丝李; 焦袂康酸; 7-prenyljacareubin; formoxanthone A; 抗肿瘤活性

中图分类号: R284.1 文献标志码: A 文章编号: 0253 - 2670(2012)03 - 0436 - 04

Chemical constituents from stem barks of *Garcinia paucinervis*

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Abstract: Objective To study the chemical constituents from the stem barks of *Garcinia paucinervis* and their antitumor activities.

Methods Chemical constituents were isolated and purified from petroleum ether extract of *G. paucinervis* by chromatography on silica gel, RP-18, Sephadex LH-20 column, and preparative TLC. Their structures were identified on the basis of spectroscopic analysis and chemical evidence. The antitumor activity was screened by MTT assay. **Results** Ten compounds were isolated and identified as cambogin (1), pyromeconic acid (2), β -sitosterol (3), daucosterol (4), 7-prenyljacareubin (5), parvifolixanthone A (6), formoxanthone A (7), termicalcicolanone A (8), 1, 3, 5, 6-tetrahydroxy-4-prenylxanthone (9), and isogarcinol (10). Compounds 5 and 7 showed moderate cytotoxic activity against HL-60, SMMC-7721, A549, MCF-7, and SW480 cell lines. **Conclusion** Compounds 2—5 and 7—10 are isolated from this plant for the first time and the ¹³C-NMR spectroscopic data of compound 5 are firstly reported.

Key words: *Garcinia paucinervis* Chun et How; pyromeconic acid; 7-prenyljacareubin; formoxanthone A; antitumor activity

金丝李 *Garcinia paucinervis* Chun et How 系藤黄科藤黄属植物, 主要分布在广西和云南部分地区, 是产于石灰岩山地的珍贵用材树种, 木材坚重、耐腐、耐水性特强且不受虫蛀, 为机械、军事、造船、建筑工业和高级家具等用材。《中华本草》记载金丝李具有清热解毒、消肿; 主痈肿疮毒、烫伤等功效。金丝李叶的化学成分和药理研究已有相关报道^[1-2],

而其茎皮的化学成分还未见报道。为了更好地保护和开发藤黄属植物资源并从中寻找更多的生物活性物质, 本实验继续对金丝李的茎皮进行研究。通过正相硅胶柱色谱、制备薄层色谱和重结晶等手段从金丝李的茎皮乙醇提取物中分离得到 10 个化合物, 经光谱分析, 分别鉴定为 cambogin (1)、焦袂康酸 (pyromeconic acid, 2)、 β -谷甾醇 (β -sitosterol, 3)、

收稿日期: 2011-08-24

基金项目: 国家自然科学基金资助项目 (20702061); 中国科学院创新基金 (KSCX2-YW-R-172)

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胡萝卜苷 (daucosterol, **4**)、7-prenyljacareubin (**5**)、parvifolixanthone A (**6**)、formoxanthone A (**7**)、termicalcicolanone A (**8**)、1, 3, 5, 6-tetrahydroxy-4-prenylxanthone (**9**)、isogarcinol (**10**)。其中, 化合物**2~5**和**7~10**为该植物中首次分离得到, 同时发现化合物**5**和**7**具有一定的体外抗肿瘤活性。

1 仪器与材料

API QSTAR Pulsa 质谱仪; X—4 显微熔点测定仪 (巩义市予华仪器有限责任公司); Bruker AM—400/500 核磁共振仪 (TMS 为内标); 柱色谱硅胶及薄板色谱硅胶 (GF₂₅₄ 10~40 μm) 为青岛美高化工有限公司产品; RP-18 (40~63 μm) 为德国 Merck 公司产品; Sephadex LH-20 (40~70 μm) 为瑞典 GE Healthcare Bio-Sciences AB 公司产品。所用有机试剂均为分析纯。

金丝李 *Garcinia paucinervis* Chun et How 茎皮于2010年7月采于云南省中国科学院西双版纳热带植物园, 由中国科学院西双版纳热带植物园陶国达高级实验师鉴定, 标本(20100722)存放于中国科学院西双版纳热带植物园民族植物学实验室。

2 提取与分离

金丝李茎皮 15.5 kg, 室温阴干, 粉碎, 用 60 L 95%乙醇冷浸提 3 次, 每次 7 d, 合并提取液, 减压回收乙醇得总浸膏 755 g。将浸膏悬浮于 1.5 L 温水 (60 °C) 中, 依次用石油醚、醋酸乙酯萃取, 每次萃取溶剂量为 2 L, 萃取 3 次, 合并萃取液, 减压回收溶剂, 得石油醚部位 201 g、醋酸乙酯部位 292 g。取石油醚部位, 硅胶柱色谱 (200~300 目) 分离, 石油醚-醋酸乙酯 (90:10→20:80) 梯度洗脱, 用薄层色谱检测合并相似点将其分为 G1~G6。G2 (74 g) 经硅胶柱色谱 (300~400 目) 分离, 以石油醚-丙酮 (90:10→70:30) 为流动相, 分得 3 个组分, 其中石油醚-丙酮 (70:30) 洗脱部分再用柱色谱法分得 G2a~G2f 6 个亚组分。G2f 用反相柱 RP-18 色谱分离, 以甲醇-水 (60:40→100:0) 依次洗脱得化合物**2** (7 mg)。G2b 用反相柱 RP-18 色谱分离, 以甲醇-水 (60:40→100:0) 依次洗脱得化合物**5** (62 mg)、**7** (35 mg)。G2c 用反相柱 RP-18 色谱分离, 以甲醇-水 (60:40→100:0) 依次洗脱得化合物**3** (10 mg)、**10** (22 mg)、**4** (11 mg)、**8** (30 mg)。石油醚-丙酮 (80:20) 洗脱部分分得 3 个亚组分 A2a~A2c, A2b 用反相柱 RP-18 色谱分离, 以甲醇-水 (60:40→100:0) 依次洗脱得化合

物**6** (7 mg), **9** (10 mg)。石油醚-丙酮 (90:10) 洗脱部分分得 3 个亚组分 B2a~B2c, B2a 经反相柱 RP-18 色谱和葡聚糖凝胶 Sephadex LH-20 分离, 洗脱得化合物**1** (17 mg)。

3 结构鉴定

化合物 1: 分子式 C₃₈H₅₂O₆, ESI-MS *m/z*: 605 [M+H]⁺, 无色无定形粉末。¹H-NMR (500 MHz, DMSO-*d*₆) δ: 7.12 (1H, d, *J* = 2.0 Hz, H-12), 6.92 (1H, dd, *J* = 2.0, 8.0 Hz, H-16), 6.71 (1H, d, *J* = 8.0 Hz, H-15), 5.16 (1H, t, *J* = 7.5 Hz, H-35), 4.89 (1H, t, *J* = 7.0 Hz, H-25), 4.76 (1H, t, *J* = 5.0 Hz, H-18), 2.84 (1H, dd, *J* = 3.5, 14.0 Hz, H-29a), 2.54 (1H, m, H-17a), 2.52 (1H, m, H-24a), 2.36 (1H, dd, *J* = 5, 13.6 Hz, H-17b), 2.22 (1H, d, *J* = 14.4 Hz, H-7a), 2.20 (1H, m, H-24b), 2.02 (1H, m, H-7b), 1.88 (1H, m, H-34a), 1.80 (1H, m, H-34b), 1.61 (3H, s, H-37), 1.57 (3H, s, H-28), 1.56 (9H, s, H-38, 20, 21), 1.52 (3H, s, H-27), 1.36 (1H, m, H-30), 1.20 (3H, s, H-32), 1.16 (3H, s, H-22), 0.98 (1H, m, H-29b), 0.93 (1H, s, H-33), 0.92 (3H, s, H-23); ¹³C-NMR (125 MHz, DMSO-*d*₆) δ: 206.5 (C-9), 193.3 (C-3), 191.4 (C-10), 170.5 (C-1), 150.9 (C-14), 145.2 (C-13), 132.7 (C-19), 132.6 (C-36), 132.0 (C-26), 128.7 (C-11), 125.2 (C-25), 124.8 (C-16), 122.0 (C-2, 35), 120.5 (C-18), 115.2 (C-12), 115.0 (C-15), 86.4 (C-31), 67.6 (C-4), 50.7 (C-8), 45.6 (C-6), 45.2 (C-5), 42.2 (C-30), 38.3 (C-7), 29.1 (C-34), 28.8 (C-24), 28.3 (C-33), 27.6 (C-29), 26.2 (C-23), 25.7 (C-21), 25.6 (C-28), 25.4 (C-37), 25.0 (C-17), 22.1 (C-22), 21.0 (C-32), 17.9 (C-20, 27), 17.8 (C-38)。以上光谱数据与文献报道对照^[3], 鉴定化合物**1**为 cambogin。

化合物 2: 分子式 C₅H₄O₃, 无定形粉末。¹H-NMR (500 MHz, CDCl₃) δ: 7.88 (1H, s, H-2), 7.80 (1H, d, *J* = 5.6 Hz, H-6); ¹³C-NMR (125 MHz, CDCl₃) δ: 173.7 (C-4), 155.5 (C-6), 146.7 (C-3), 138.8 (C-2), 113.7 (C-5)。以上光谱数据与文献报道对照^[4], 鉴定化合物**2**为焦袂康酸。

化合物 3: 分子式 C₂₉H₅₀O, 白色针状晶体 (CHCl₃), 与 β-谷甾醇对照品共薄层, 其 R_f 值一致, 混合后熔点不下降, 鉴定化合物**3**为 β-谷甾醇。

化合物 4: 分子式 C₃₅H₆₀O₆, 白色粉末, Libermann-Burchard 反应和 Molish 反应均呈阳性, 与胡萝卜苷对照品共薄层, 其 R_f 值一致, 鉴定化合

物**4**为胡萝卜昔。

化合物5: 分子式C₂₃H₂₂O₆, ESI-MS m/z: 395 [M+H]⁺, 黄色无定形粉末。¹H-NMR (400 MHz, acetone-d₆) δ: 13.64 (1H, s, 1-OH), 7.52 (1H, s, H-8), 6.67 (1H, d, J = 10.0 Hz, H-4'), 6.30 (1H, s, H-4), 5.73 (1H, d, J = 10.0 Hz, H-5'), 5.40 (1H, t, J = 7.2 Hz, H-2''), 3.44 (2H, t, J = 7.2 Hz, H-1''), 1.76 (3H, s, H-5''), 1.75 (3H, s, H-4''); ¹³C-NMR (100 MHz, acetone-d₆) δ: 181.4 (C-9), 160.8 (C-1), 158.3 (C-3), 157.7 (C-4a), 150.6 (C-6), 145.4 (C-10a), 133.5 (C-3''), 132.1 (C-5), 128.6 (C-5''), 126.9 (C-7), 122.6 (C-2''), 116.5 (C-4'), 115.7 (C-8), 113.7 (C-8a), 105.0 (C-2), 103.6 (C-9a), 95.4 (C-4), 78.9 (C-6''), 28.8 (C-1''), 28.4 (C-7', 8'), 25.9 (C-4''), 17.8 (C-5'').以上光谱数据与文献报道对照^[5], 鉴定化合物**5**为7-prenyljacareubin。本研究首次报道该化合物的碳谱数据。

化合物6: 分子式C₂₈H₃₂O₆, ESI-MS m/z: 465 [M+H]⁺, 黄色无定形粉末。¹H-NMR (400 MHz, acetone-d₆) δ: 13.61 (1H, s, 1-OH), 7.57 (1H, s, H-8), 5.40 (1H, t, J = 7.2 Hz, H-2), 5.24 (2H, overlap, H-2', H-2''), 3.67 (2H, d, J = 7.2 Hz, H-1), 3.43 (4H, overlap, H-1', 1''), 1.83 (3H, s, H-5''), 1.78 (3H, s, H-5''), 1.76 (3H, s, H-5), 1.74 (3H, s, H-4), 1.65 (6H, s, H-4'', H-4'); ¹³C-NMR (100 MHz, acetone-d₆) δ: 181.4 (C-9), 160.8 (C-3), 159.5 (C-1), 153.4 (C-4a), 151.2 (C-6), 146.3 (C-10a), 133.4 (C-3''), 132.5 (C-3), 132.4 (C-3''), 131.9 (C-5), 126.5 (C-7), 123.5 (C-2'), 123.0 (C-2''), 122.7 (C-2), 117.1 (C-8), 113.6 (C-8a), 110.9 (C-2), 107.1 (C-4), 103.2 (C-9a), 28.7 (C-1), 25.9 (C-5''), 4), 25.8 (C-5''), 22.1 (C-1''), 22.0 (C-1''), 18.0 (C-4''), 17.9 (C-4''), 17.8 (C-5)。以上光谱数据与文献报道对照^[6], 鉴定化合物**6**为parvifolixanthone A。

化合物7: 分子式C₂₃H₂₂O₆, ESI-MS m/z: 395 [M+H]⁺, 黄色针晶(甲醇), mp 115~118 °C。¹H-NMR (500 MHz, acetone-d₆) δ: 13.10 (1H, s, 1-OH), 7.42 (1H, s, H-8), 6.57 (1H, d, J = 10.0 Hz, H-4'), 6.32 (1H, s, H-2), 5.88 (1H, d, J = 10.0 Hz, H-5'), 5.37 (1H, t, J = 7.3 Hz, H-2''), 3.55 (2H, d, J = 7.3 Hz, H-1''), 1.85 (3H, s, H-4''), 1.64 (3H, s, H-5''), 1.48 (6H, s, H-7', 8'); ¹³C-NMR (125 MHz, acetone-d₆) δ: 181.2 (C-9), 163.3 (C-3), 162.2 (C-1), 155.5 (C-4a), 146.9 (C-6), 146.4 (C-10a), 134.4 (C-3''), 132.3 (C-4''),

131.8 (C-5), 123.3 (C-2''), 122.1 (C-5'), 119.0 (C-7), 115.2 (C-8a), 113.1 (C-8), 107.5 (C-4), 103.2 (C-9a), 98.4 (C-2), 78.8 (C-6'), 28.3 (C-7', 8'), 25.9 (C-4''), 22.2 (C-1''), 18.0 (C-5'')。以上光谱数据与文献报道对照^[7], 鉴定化合物**7**为formoxanthone A。

化合物8: 分子式C₂₃H₂₂O₆, ESI-MS m/z: 395 [M+H]⁺, 黄色无定形粉末。¹H-NMR (400 MHz, acetone-d₆) δ: 13.19 (1H, s, 1-OH), 7.40 (1H, d, J = 8.8 Hz, H-5), 7.31 (1H, d, J = 8.8 Hz, H-6), 6.71 (1H, d, J = 10.0 Hz, H-4'), 5.73 (1H, d, J = 10.0 Hz, H-5''), 5.30 (1H, t, J = 6.8 Hz, H-2''), 4.17 (2H, d, J = 6.8 Hz, H-1''), 1.83 (3H, s, H-4''), 1.63 (3H, s, H-5''), 1.48 (6H, s, H-7', 8'); ¹³C-NMR (100 MHz, acetone-d₆) δ: 184.6 (C-9), 152.3 (C-7), 152.2 (C-10a), 150.8 (C-3), 148.3 (C-1), 145.0 (C-4), 131.3 (C-3''), 128.9 (C-8), 128.6 (C-5''), 124.3 (C-6), 124.0 (C-2''), 119.3 (C-8a), 116.9 (C-5), 116.3 (C-4'), 104.4 (C-2), 104.3 (C-9a), 78.8 (C-6''), 28.2 (C-7', 8'), 26.2 (C-1''), 25.9 (C-4''), 18.2 (C-5'')。以上光谱数据与文献报道对照^[8], 鉴定化合物**8**为termicalcicolanone A。

化合物9: 分子式C₁₈H₁₆O₆, ESI-MS m/z: 329 [M+H]⁺, 暗黄色无定形粉末。¹H-NMR (400 MHz, acetone-d₆) δ: 13.45 (1H, s, 1-OH), 7.62 (1H, d, J = 8.8 Hz, H-5), 6.96 (1H, d, J = 8.8 Hz, H-7), 6.51 (1H, s, H-2), 5.27 (1H, d, J = 7.2 Hz, H-2''), 3.34 (2H, d, J = 7.2 Hz, H-1''), 1.77 (3H, s, H-4''), 1.63 (3H, s, H-5''); ¹³C-NMR (100 MHz, acetone-d₆) δ: 181.1 (C-9), 163.3 (C-3), 161.5 (C-1), 156.4 (C-4a), 151.9 (C-6), 146.8 (C-10a), 133.1 (C-3''), 131.5 (C-5), 123.3 (C-2''), 117.4 (C-8), 114.8 (C-8a), 113.5 (C-7), 111.2 (C-4), 102.9 (C-9a), 94.0 (C-2), 25.8 (C-4''), 21.9 (C-1''), 17.8 (C-5'')。以上光谱数据与文献报道对照^[9], 鉴定化合物**9**为1, 3, 5, 6-tetrahydroxy-4-prenylxanthone。

化合物10: 分子式C₃₈H₅₂O₆, ESI-MS m/z: 605 [M+H]⁺, 白色无定形粉末。¹H-NMR (400 MHz, acetone-d₆) δ: 7.36 (1H, d, J = 2.0 Hz, H-12), 7.13 (1H, dd, J = 2.0, 8.2 Hz, H-16), 6.83 (1H, d, J = 8.2 Hz, H-15), 5.19 (1H, t, J = 6.4 Hz, H-35), 4.94 (1H, t, J = 6.4 Hz, H-25), 4.93 (1H, t, J = 6.4 Hz, H-18), 3.05 (1H, dd, J = 3.6, 14.4 Hz, H-29a), 2.78 (1H, m, H-24a), 2.54 (1H, dd, J = 6.8, 13.2 Hz, H-17a), 2.36 (1H, dd, J = 4.8, 13.2 Hz, H-17b), 2.20 (1H, m, H-24b), 2.02 (1H, m, H-7b), 1.88 (1H, m, H-34a),

1.78 (3H, s, H-28), 1.71 (3H, s, H-27a), 1.69 (3H, s, H-37), 1.64 (3H, s, H-38a), 1.56 (6H, s, H-38b, 20), 1.29 (3H, s, H-27b), 1.36 (1H, m, H-30), 1.16 (3H, s, H-32a), 1.21 (2H, s, H-29b), 1.16 (3H, s, H-32b), 0.99 (3H, s, H-22), 0.94 (3H, s, H-33); ^{13}C -NMR (100 MHz, acetone- d_6) δ : 207.3 (C-9), 194.3 (C-4), 192.2 (C-10), 171.2(C-2), 151.1(C-14), 145.7 (C-13), 134.0 (C-19), 133.8 (C-36), 133.2 (C-26), 131.1 (C-11), 126.5 (C-3), 126.5 (C-25), 123.6 (C-16), 122.9 (C-35), 121.4 (C-18), 115.8 (C-12), 115.5 (C-15), 87.0 (C-31), 68.8 (C-5), 51.9 (C-1), 46.9 (C-6), 46.5 (C-7), 43.7

(C-30), 39.4 (C-8), 30.3 (C-34), 29.7 (C-24), 29.0 (C-33), 28.8 (C-29), 27.6 (C-37), 26.3 (C-27), 26.2 (C-20), 26.1 (C-22), 25.9 (C-17), 22.7(C-23), 21.5 (C-32), 18.4 (C-28), 18.2 (C-21), 17.9 (C-38)。以上光谱数据与文献报道对照^[10], 鉴定化合物 **10** 为 isogarcinol。

4 抗肿瘤活性筛选

采用 MTT 法^[11]进行抗肿瘤活性筛选, 发现化合物 **5** 和 **7** 对 HL-60、SMMC-7721、A549、MCF-7 和 SW480 细胞株均有一定的抑制作用。化合物 **5** 对结肠癌 SW480 细胞抑制活性明显强于阳性对照药顺铂(表 1)。

表 1 化合物 **5** 和 **7** 的细胞毒活性

Table 1 Cytotoxic activity of compounds **5** and **7**

化合物	IC ₅₀ / ($\mu\text{g}\cdot\text{mL}^{-1}$)				
	HL-60	SMMC-7721	A549	MCF-7	SW480
formoxanthone A (7)	12.16	20.35	20.35	16.63	20.44
7-prenyljacareubin (5)	15.23	21.70	16.51	12.25	8.14
顺铂	1.50	15.48	14.05	13.44	13.61
紫杉醇	<0.008	<0.008	<0.008	<0.008	<0.008

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