

阔叶黄檀三萜类化学成分分析

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[摘要] 目的:对黄檀属植物中阔叶黄檀(*Dalbergia latifolia*)中的三萜类化学成分进行分离并对分离得到的单体化合物进行结构鉴定。方法:将阔叶黄檀心材50.0 kg,粉碎,用70%乙醇加热回流提取3次,每次2 h,合并滤液,减压回收溶剂得到浸膏。浸膏经水分散,通过石油醚、二氯甲烷、乙酸乙酯及水饱和正丁醇萃取,制备得到上述4个萃取部位。然后分别采用硅胶, LH-20 羟丙基葡聚糖凝胶, C₁₈ 色谱柱(ODS)及大孔吸附树脂等色谱分离技术对各个萃取部位进行化学成分分离。通过理化分析、波谱分析及质谱分析等手段,对分离得到的各单体化合物进行结构鉴定。结果:从阔叶黄檀中分离鉴定得到7个三萜类化合物,分别为3-O- α -L-arabinopyranosyl-28-O- β -D-glucopyranosyl pomolic acid (1), β -sitostenone (2), β -sitosterol (3), 甾甾醇(4), β -香树脂酸乙酸酯(5), β -香树脂醇-3-棕榈酸酯(6), 3-乙酰齐墩果酸(7)。结论:化合物1, 5, 6为首次从该属植物中分离得到, 化合物2, 4为首次从该植物中分离得到。上述研究成果为进一步阐明阔叶黄檀药效物质基础提供了科学依据, 同时为该植物资源综合开发利用打下了良好的基础。

[关键词] 阔叶黄檀; 三萜类; 结构鉴定

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Triterpenoids from Heartwood of *Dalbergia latifolia*

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[Abstract] **Objective:** To investigate the chemical constituents of triterpenoids in the heartwood of *Dalbergia latifolia*. **Method:** The heartwood samples of *D. latifolia* were crushed into powder and extracted with 70% ethanol for 3 times, 2 h for each time. The filtrate liquid was collected and the solvent was decompressed and recovered to obtain extract. The extract was dispersed in water and then extracted successively with petroleum ester, dichloromethane, ethylacetate and butanol to obtain petroleum ester fraction, dichloromethane fraction, ethylacetate fraction and butanol fraction. The chemical compounds in the extract fractions were isolated by column chromatography technologies such as silica gel, Sephadex LH-20, reverse phase silica gel and macroporous resin, and the structures of the obtained monomeric compounds were identified through physical and chemical analysis, spectroscopic methods and mass spectrum analysis method. **Result:** Seven triterpenoids were isolated and identified as 3-O- α -L-arabinopyranosyl-28-O- β -D-glucopyranosyl pomolic acid (1), β -sitostenone (2), β -sitosterol (3), stigmasterol (4), β -amyirin acetate (5), β -amyirin-3-palmitate (6), and 3-acetoxy-oleanoic acid (7). **Conclusion:** Compounds 1, 5, and 6 were isolated from *Dalbergia* genus for the first time. Compounds 2 and 4

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were isolated from this plant for the first time.

[Key words] *Dalbergia latifolia*; triterpenoids; structure identification

阔叶黄檀广泛分布于东南亚国家等地^[1],常用于治疗腹泻、消化不良、麻风及肥胖等疾病^[2]。现代药理研究表明,阔叶黄檀具有良好的抗癌与抗氧化作用,例如, Niraimathi 和 Sundaraganapathy^[3-4] 经体外实验研究,发现阔叶黄檀甲醇部位对埃利希氏腹水癌(EAC),乳腺癌细胞(MCF 7),肝癌细胞株 HepG2 及宫颈癌细胞株 HeLa 等具有增殖抑制作用。Khalid 等^[5] 研究发现阔叶黄檀树皮乙醇提取物对 DPPH, NO 及硫氰酸铁自由基具有较强的清除能力。目前,该植物化学成分研究报道以黄酮类和甾醇类化学成分为主^[6-9],而三萜类化学成分报道较少,目前仅得到 β -sitosterol^[5] 与乙酰齐墩果酸^[7]。为此,本课题组对该植物中三萜类化学成分进行了研究,分离得到 7 个三萜类化合物,即 3-O- α -L-arabinopyranosyl-28-O- β -D-glucopyranosyl pomolic acid (1), β -sitostenone (2), β -sitosterol (3), 甾醇(4), β -香树脂酸乙酸酯(5), β -香树脂醇-3-棕榈酸酯(6), 3-乙酰齐墩果酸(7)。其中,化合物 1, 5, 6 为首次从该属植物中分离得到,化合物 2, 4 为首次从该植物中分离得到。本研究为进一步阐明阔叶黄檀药效物质基础提供了科学依据,同时为该植物资源综合利用打下了良好的基础。

1 材料

AVANCE III HD 600 MHz 型核磁共振波谱仪(德国 Bruker 公司), 1260 系列高效液相色谱仪(美国 Agilent 公司), Triple TOF 5600 型质谱仪(美国 AB Sciex 公司), LC3000 型半制备高效液相色谱仪(北京创新通恒科技有限公司); Lunna 10 μ m C₁₈ 制备型液相色谱柱[菲罗门(广州)科学仪器有限公司], LH-20 羟丙基葡聚糖凝胶(Sephadex LH-20, 美国 GE Healthcare 公司), 薄层色谱硅胶和柱色谱硅胶(青岛海洋化工厂); 所用试剂均为分析纯。

阔叶黄檀购自广西防城港, 经广西大学林产品质量检测中心鉴定为蝶形花科植物阔叶黄檀 *Dalbergia latifolia* 的心材, 凭证标本(20140702)存于江西中医药大学现代中药制剂教育部重点实验室标本室。

2 提取与分离

取阔叶黄檀心材 50.0 kg, 粉碎, 用 70% 乙醇加热回流提取 3 次, 每次 2 h, 合并提取液, 回收溶剂,

浓缩至无醇味, 得浸膏 4.5 kg。加适量水于浸膏中分散, 得到混悬液, 依次用石油醚、二氯甲烷、乙酸乙酯、正丁醇进行萃取, 回收溶剂, 分别得到石油醚萃取部位 75.0 g, 二氯甲烷萃取部位 2.0 kg, 乙酸乙酯萃取部位 1.8 kg, 正丁醇萃取 30.0 g。

石油醚萃取部位(75.0 g)经硅胶柱色谱, 用石油醚-乙酸乙酯(20:1~1:1)梯度洗脱, 得到 12 个流分, 即 Fr. 1~Fr. 12。Fr. 9(340.0 mg)经 Sephadex LH-20 柱色谱(二氯甲烷-甲醇 1:1)洗脱, 得到 5 个流分, 即 Fr. 9.A~Fr. 9.E。将 Fr. 9.B(72.3 mg)流分经 Sephadex LH-20 纯化得到化合物 1(2.3 mg)。

二氯甲烷萃取部位(1.5 kg)经硅胶柱色谱, 以石油醚-乙酸乙酯(10:1~1:2)梯度洗脱, 得到 21 个流分, 即 Fr. 1~Fr. 21。Fr. 4(7.3 g)经 Sephadex LH-20 纯化, 得到化合物 2(36.8 mg), 化合物 5(4.7 mg)。Fr. 10(5.6 g)经硅胶柱色谱, 以石油醚-丙酮(12:1)等度洗脱, 得到化合物 3(2.5 mg), 化合物 4(2.3 mg)。Fr. 13(44.6 g)经硅胶柱色谱, 以二氯甲烷, 二氯甲烷-甲醇(100:1~20:1)梯度洗脱, 得到 20 个流分, 即 Fr. 13.A~Fr. 13.T。Fr. 13.K(832.1 mg)经硅胶柱色谱分离, 以二氯甲烷-丙酮(50:1)等度洗脱, 得到 6 个流分 Fr. 13.K.1~Fr. 13.K.6。Fr. 13.K.1(169.3 mg)经 Sephadex LH-20 柱色谱(二氯甲烷-甲醇 1:1)洗脱, 得到 6 个流分, 即 Fr. 13.K.1.A~Fr. 13.K.1.F。Fr. 13.K.1.B(125.6 mg)利用半制备型液相色谱仪, 以甲醇-水(72:28)作为流动相, 等度洗脱得到化合物 6(2.2 mg, 保留时间为 42.28 min)。

乙酸乙酯萃取部位(1.2 kg)经硅胶柱色谱, 以二氯甲烷, 二氯甲烷-甲醇(100:1~5:1)梯度洗脱得到 19 个流分, 即 Fr. 1~Fr. 19。Fr. 2(1.5 g)经 Sephadex LH-20 柱色谱(二氯甲烷-甲醇 1:1), 得到化合物 7(23.2 mg)。各化合物结构图见图 1。

3 结构鉴定

化合物 1 黄色粉末, ESI-MS m/z 734.20 [M+H]⁺。¹H-NMR(600 MHz, CDCl₃) δ : 3.13(1H, dd, J =11.5, 4.1 Hz, H-3), 1.98(2H, m, H-11), 5.30(1H, t, J =5.9 Hz, H-12), 2.58(1H, dd, J =13.3, 9.3 Hz, H-16A), 0.97(3H, s, H-23), 0.77(3H, s, H-24), 1.03(3H, s, H-25), 0.83(3H, s, H-26),

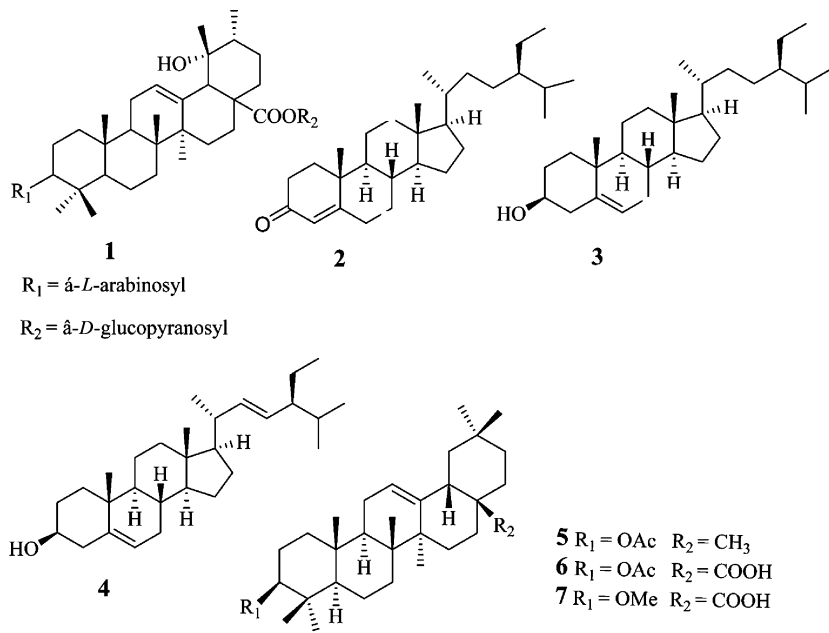


图 1 化合物 1~7 化学结构式
Fig.1 Structure of compound 1-7

1. 31 (3H, s, H-27), 1. 19 (3H, s, H-29), 0. 93 (3H, d, $J = 5. 3$ Hz, H-31), 4. 27 (1H, d, $J = 6. 7$ Hz, H-Arabinose-1'), 3. 52 (1H, dd, $J = 9. 4, 4. 4$ Hz, H-Arabinose-3'), 3. 81 (1H, dd, $J = 19. 2, 6. 9$ Hz, H-Arabinose-5'A), 3. 52 (1H, dd, $J = 9. 4, 4. 4$ Hz, H-Arabinose-5'B), 5. 33 (1H, d, $J = 8. 1$ Hz, H-Glucose-1'), 3. 33 (1H, ddd, $J = 11. 8, 5. 2, 4. 4$ Hz, H-Glucose-5'), 3. 81 (1H, dd, $J = 19. 2, 6. 9$ Hz, H-Glucose-6'A), 3. 66 (1H, dd, $J = 12. 0, 3. 8$ Hz, H-Glucose-6'B); $^{13}\text{C-NMR}$ (151 MHz, CDCl_3) δ : 38. 76 (C-1), 5. 64 (C-2), 89. 40 (C-3), 39. 82 (C-4), 55. 57 (C-5), 18. 03 (C-6), 32. 70 (C-7), 41. 17 (C-8), 36. 42 (C-10), 23. 25 (C-11), 128. 31 (C-12), 138. 10 (C-13), 41. 49 (C-14), 28. 24 (C-15), 25. 62 (C-16), 53. 50 (C-18), 71. 37 (C-19), 41. 49 (C-20), 25. 64 (C-21), 38. 42 (C-22), 27. 15 (C-23), 15. 57 (C-24), 14. 57 (C-25), 16. 17 (C-26), 23. 29 (C-27), 177. 21 (C-28), 25. 77 (C-29), 15. 20 (C-30), 105. 72 (3-Arabinose, C-1'), 72. 25 (C-2'), 72. 87 (C-3'), 68. 11 (C-4'), 65. 00 (C-5'), 94. 33 (28-Glucose, C-1'), 72. 39 (C-2'), 76. 79 (C-3'), 69. 63 (C-4'), 77. 13 (C-5'), 60. 94 (C-6'). 以上数据与文献[10]报道基本一致,故鉴定化合物 1 为 3-O- α -L-arabinopyranosyl-28-O- β -D-glucopyranosyl pomolic acid。

化合物 2 黄色粉末,ESI-MS m/z 412 [M +

H] $^+$ 。 $^1\text{H-NMR}$ (600 MHz, CDCl_3) δ : 5. 73 (4H, s, H-4), 1. 19 (3H, s, H-19), 0. 89 (3H, m, H-21, 26, 27, 29), 0. 73 (3H, d, $J = 11. 08$ Hz, H-18); $^{13}\text{C-NMR}$ (151 MHz, CDCl_3) δ : 36. 22 (C-1), 33. 97 (C-2), 123. 83 (C-4), 171. 83 (C-5), 33. 06 (C-6), 32. 15 (C-7), 34. 08 (C-8), 55. 98 (C-9), 39. 72 (C-10), 21. 13 (C-11), 38. 70 (C-12), 42. 48 (C-13), 56. 10 (C-14), 24. 29 (C-15), 28. 30 (C-16), 53. 91 (C-17), 12. 09 (C-18), 17. 48 (C-19), 35. 79 (C-20), 18. 81 (C-21), 35. 72 (C-22), 26. 15 (C-23), 45. 91 (C-24), 29. 24 (C-25), 19. 94 (C-26), 19. 14 (C-27), 23. 16 (C-28), 12. 06 (C-29), 199. 76 (C=O)。以上数据与文献[11]报道基本一致,故鉴定化合物 2 为 β -sitostenone。

化合物 3 白色粉末,ESI-MS m/z 414 [M + H] $^+$ 。 $^1\text{H-NMR}$ (600 MHz, CDCl_3) δ : 5. 35 (1H, dd, $J = 5. 07, 1. 66$ Hz, H-6), 3. 52 (1H, ddd, $J = 20. 91, 10. 43, 4. 81$ Hz, H-3), 1. 01 (3H, s, 3H, CH_3 -19), 0. 92 (3H, d, $J = 6. 53$ Hz, CH_3 -21), 0. 85 (3H, dd, $J = 6. 84, 3. 40$ Hz, CH_3 -29), 0. 82 (3H, m, CH_3 -26), 0. 79 (3H, dd, $J = 7. 00, 1. 81$ Hz, CH_3 -27), 0. 69 (3H, d, CH_3 -18); $^{13}\text{C-NMR}$ (151 MHz, CDCl_3) δ : 32. 39 (C-1), 31. 79 (C-2), 71. 95 (C-3), 42. 46 (C-4), 140. 88 (C-5), 121. 86 (C-6), 32. 04 (C-7), 32. 06 (C-8), 50. 29 (C-9), 36. 65 (C-10), 21. 21 (C-11), 39. 82 (C-12), 42. 35 (C-13), 56. 90 (C-

14), 24.45 (C-15), 28.40 (C-16), 57.18 (C-17), 12.01 (C-18), 19.55 (C-19), 36.65 (C-20), 18.92 (C-21), 34.08 (C-22), 26.19 (C-23), 45.96 (C-24), 29.28 (C-25), 18.92 (C-26), 19.13 (C-27), 23.20 (C-28), 12.01 (C-29)。以上数据与文献[12]报道基本一致,故鉴定化合物**3**为 β -sitosterol。

化合物**4** 白色粉末,ESI-MS m/z 417 [M + H]⁺。¹H-NMR (600 MHz, CDCl₃) δ : 3.52 (1H, ddd, $J = 20.91, 10.43, 4.80$ Hz, H-3), 5.35 (1H, dd, $J = 5.07, 1.66$ Hz, H-6), 5.15 (1H, dd, $J = 15.15, 8.69$ Hz, H-22), 5.01 (1H, dd, $J = 15.17, 8.75$ Hz, H-23); ¹³C-NMR (151 MHz, CDCl₃) δ : 32.39 (C-1), 31.79 (C-2), 71.95 (C-3), 42.43 (C-4), 140.88 (C-5), 121.86 (C-6), 31.79 (C-7), 32.04 (C-8), 50.26 (C-9), 36.65 (C-10), 21.21 (C-11), 39.82 (C-12), 42.35 (C-13), 56.08 (C-14), 24.45 (C-15), 29.07 (C-16), 56.90 (C-17), 12.01 (C-18), 19.55 (C-19), 40.65 (C-20), 21.21 (C-21), 138.46 (C-22), 129.39 (C-23), 51.38 (C-24), 32.04 (C-25), 18.92 (C-26), 21.23 (C-27), 25.96 (C-28), 12.41 (C-29)。以上数据与文献[13]报道基本一致,故鉴定化合物**4**为stigmasterol。

化合物**5** 白色粉末,ESI-MS m/z 468 [M + H]⁺。¹H-NMR (600 MHz, CDCl₃) δ : 7.36 (2H, t, H-3', 5'), 7.26 (3H, m, H-2', 6', 4'), 6.60 (1H, s, H-8), 6.49 (1H, s, H-5), 6.34 (1H, ddd, $J = 6.5, 10.2, 17.0$ Hz, H-3), 5.34 (1H, dt, $J = 1.4, 10.2$ Hz, H-2b), 5.06 (1H, dt, $J = 1.5, 17.2$ Hz, H-2a), 4.87 (1H, $J = 6.5$ Hz, H-4), 3.86 (3H, s, 9-OCH₃), 3.78 (3H, s, 6-OCH₃); ¹³C-NMR (151 MHz, CDCl₃) δ : 171.08 (C = O), 21.33 (CH₃C = O), 39.27 (C-1), 23.52 (C-2), 80.94 (C-3), 37.69 (C-4), 55.28 (C-5), 18.17 (C-6), 32.51 (C-7), 40.89 (C-8), 47.54 (C-9), 38.05 (C-10), 22.86 (C-11), 122.54 (C-12), 143.59 (C-13), 41.54 (C-14), 27.66 (C-15), 25.92 (C-16), 32.43 (C-17), 46.55 (C-18), 45.82 (C-19), 30.67 (C-20), 33.78 (C-21), 36.98 (C-22), 28.04 (C-23), 17.13 (C-24), 15.39 (C-25), 16.67 (C-26), 23.59 (C-27), 29.72 (C-28), 33.07 (C-29), 23.39 (C-30)。以上数据与文献[14]报道基本一致,故鉴定化合物**5**为 β -amyrin acetate。

化合物**6** 白色粉末,ESI-MS m/z 498 [M + H]⁺。¹H-NMR (600 MHz, CDCl₃) δ : 0.84 (3H, s, H-

23), 0.86 (3H, s, H-24), 0.87 (3H, s, H-25), 0.90 (3H, s, H-26), 0.92 (3H, s, H-27), 0.93 (3H, s, H-28), 1.12 (3H, s, H-29), 1.18 (3H, d, $J = 22.51$ Hz, H-30), 2.33 (3H, t, $J = 7.46$ Hz, H-2'), 4.49 (1H, m, H-3), 5.26 (1H, s, H-12); ¹³C-NMR (151 MHz, CDCl₃) δ : 39.62 (C-1), 23.73 (C-2), 81.32 (C-3), 38.40 (C-4), 55.62 (C-5), 18.53 (C-6), 32.81 (C-7), 41.26 (C-8), 47.88 (C-9), 37.29 (C-10), 23.73 (C-11), 122.86 (C-12), 143.88 (C-13), 41.91 (C-14), 26.25 (C-15), 27.50 (C-16), 32.70 (C-17), 46.91 (C-18), 46.14 (C-19), 31.00 (C-20), 34.43 (C-21), 38.02 (C-22), 27.97 (C-23), 17.00 (C-24), 15.73 (C-25), 17.00 (C-26), 25.77 (C-27), 28.37 (C-28), 33.40 (C-29), 23.85 (C-30), 171.49 (C-1'), 34.12 (C-2'), 32.27 (C-14'), 21.65 (C-15'), 14.47 (C-16'), 29.40 (C-3'-13')。以上数据与文献[15]报道基本一致,故鉴定化合物**6**为 β -amyrin-3-palmitate。

化合物**7** 白色针状结晶,ESI-MS m/z 470 [M + H]⁺。¹H-NMR (600 MHz, CDCl₃) δ : 5.27 (1H, t, $J = 3.55$ Hz, H-12), 4.49 (1H, dd, $J = 9.43, 6.55$ Hz, H-3), 2.80 (1H, dd, $J = 13.74, 4.18$ Hz, H-18), 2.04 (3H, s, COCH₃), 1.12 (3H, s, H-27), 0.93 (3H, s, H-25), 0.92 (3H, s, H-30), 0.90 (3H, s, H-29), 0.86 (3H, s, H-24), 0.84 (3H, s, H-23), 0.74 (3H, s, H-26); ¹³C-NMR (151 MHz, CDCl₃) δ : 40.85 (C-1), 23.45 (C-2), 80.86 (C-3), 37.99 (C-4), 55.21 (C-5), 18.10 (C-6), 33.71 (C-7), 39.20 (C-8), 47.48 (C-9), 37.62 (C-10), 23.32 (C-11), 122.48 (C-12), 143.52 (C-13), 41.48 (C-14), 27.59 (C-15), 23.51 (C-16), 46.47 (C-17), 41.48 (C-18), 45.76 (C-19), 30.60 (C-20), 36.92 (C-21), 32.37 (C-22), 27.97 (C-23), 16.59 (C-24), 15.32 (C-25), 17.10 (C-26), 25.84 (C-27), 183.70 (C-28), 33.71 (C-29), 23.45 (C-30), 170.98 (COCH₃), 21.26 (COCH₃)。以上数据与文献[16]报道基本一致,故鉴定化合物**7**为3-acetoxy-oleanoic acid。

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