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黄桐枝叶的化学成分研究

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摘 要: 为研究黄桐(Endospermum chinense Benth.) 的化学成分 运用柱层析等分离纯化方法从黄桐枝叶中分离得到 13 个化合物: pubinernoid A(1)、(E) -linalool-1-oic acid(2)、(+)-去氢催吐萝芙木醇(3)、3 α -hydroxy-5 β -epoxy-7-megastigmen-9-one(4)、齐墩果酸(5)、3-羰基齐墩果酸(6)、3-oleana-9⁽¹¹⁾,12-dien-28-oic acid(7)、甘五酸(8)、altissimanin C(9)、7-羟基 β -谷甾醇(10)、丁香脂素(11)、ficusesquilignan A(12)、ficusesquilignan B(13)。其中化合物 7 为新天然产物,化合物 1 ~ 4、8 ~ 13 为首次从该属植物中分离得到。

关键词: 黄桐; 大戟科; 化学成分; 结构鉴定

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Chemical Constituents from the Twigs and Leaves of Endospermum chinense

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Abstract: To study chemical constituents of *Endospermum chinense*, thirteen known compounds were isolated from the 95% ethanol extract of *E. chinense* with different chromatographic methods. Their structures were identified as publiner-noid A (1), (E) \dashv inalool \dashv -oic acid (2), (6S) \dashv dehydrovomifoliol (3), 3α -hydroxy-5, 6-epoxy-7-megastigmen-9-one (4) ρ leanolic acid (5), 3-ketooleanolic acid (6), 3-oleana-9⁽¹¹⁾, 12-dien-28-oic acid (7), mangiferonic acid (8), altissimanin C (9), 7β -hydroxy- β -sitosterol (10), syringaresinol (11), ficusesquilignan A (12), ficusesquilignan B (13). Compounds 1-4 and 7-13 were obtained from the genus *Endospermum* for the first time.

Key words: Endospermum chinense; Euphorbiaceae; chemical constituents; structural identification

黄桐(Endospermum chinense Benth.) 为大戟科 (Euphorbiaceae) 黄桐属乔木。分布于印度东北部、缅甸、泰国、越南以及我国的福建南部、广东、广西、海南和云南南部 [1]。据记载 [2],黄桐树皮具有治疗伤寒发狂、跌仆伤损等功效,其树叶可用于手足肿浮、痈疽发背等。为了进一步阐明黄桐的化学物质基础,为以后寻找其生理活性以及开发应用提供依据,本课题较为系统地对黄桐枝叶进行了化学成分研究,共分离得到 13 个化合物,通过波谱数据分析分别鉴定为 pubinernoid A(1)、(E) -linalool-1-oic acid(2)、(+) -去氢催吐萝芙木醇(3)、3 α -hydroxy-5 β -epoxy-7-megastigmen-9-one(4)、齐墩果酸(5)、3-羰基齐墩果酸(6)、3-oleana-9 [11],12-dien-28-oic acid (7)、甘五酸(8)、altissimanin C(9)、7-羟基 β -谷甾

醇(10)、丁香脂素(11)、ficusesquilignan A(12)、

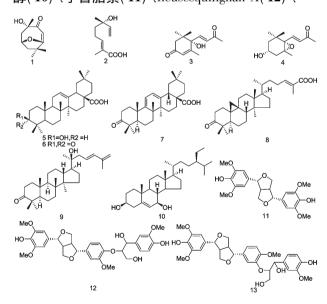


图1 化合物1~13的化学结构

Fig. 1 Chemical structures of compounds **1-43**

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ficusesquilignan B(13)。其中化合物 7 为新天然产物 化合物 $1 \sim 4 \times 8 \sim 13$ 为首次从该属植物中分离得到。

1 仪器与材料

JASCO P-1020 全自动数字旋光仪(测比旋度); Shimadzu UV-2401 分光光度仪(测 UV); Bruker Tensor-27 傅立叶变换中红外光谱仪(测 IR); Bruker AM-400 和 DRX-500 核磁共振仪(测定¹H 和¹³C NMR 谱),以 TMS 作为内标;液相-离子阱色谱质谱联用仪 Bruker HCT/Esquire(ESI-MS); 三扇型双聚焦磁质谱仪(HR-EI-MS 质谱); 高效液相为 Agilent 1100、1200(美国 Agilent 公司); Sephadex LH-20(Pharmacia 公司); 硅胶和薄层色谱硅胶(青岛海洋化工厂);反相材料 Lichroprep RP-18 gel(德国 Merck 公司); MCI gel(日本三菱化学公司); 5% H₂SO4乙醇溶液(显色剂)。

黄桐于 2010 年 12 月采自海南昌江霸王岭,由中科院昆明植物研究所胡光万博士鉴定,标本保存在中科院昆明植物研究所植物化学与西部植物资源持续利用国家重点实验室郝小江研究组(标本号: No. H20101204)。

2 提取与分离

黄桐干燥枝叶 12 kg ,粉碎后用 95% 乙醇加热回流提取三次 (4 h/x) ,然后用乙酸乙酯 5 L 萃取三次。乙酸乙酯部分 (300 g) 用硅胶 $(100 \sim 200 \text{ H})$ 柱层析 $(V_{Glabel}: V_{Phill} = 9:1 \sim 5:5 \text{ J}, V_{safe}: V_{Phill} = 9:1$ 和 7:3 分为 7 个组分 $(A \sim G)$ 。组分 $C \sim D \sim E \sim G$ 用 MCI 脱色素后 ,经反相 RP-18、正相硅胶、Sephadex LH-20、HPLC 等分离纯化 ,得到化合物 $1(7 \text{ mg}) \sim 2(7 \text{ mg}) \sim 3(5 \text{ mg}) \sim 4(3 \text{ mg}) \sim 5(10 \text{ mg}) \sim 6(22 \text{ mg}) \sim 7(5 \text{ mg}) \sim 8(11 \text{ mg}) \sim 9(6 \text{ mg}) \sim 10(11 \text{ mg}) \sim 11(8 \text{ mg}) \sim 12(16 \text{ mg}) \sim 13(17 \text{ mg})$ 。

3 结构鉴定

化合物 1 白色粉末, C_{11} H_{16} O_3 , 1 H 1 1 H 2 H H 2 H

化合物 2 无色油状 $C_{10}H_{16}O_3$, $[\alpha]_D^{25}+6.4$ (c 0.30 ,CHCl₃), ¹H NMR (CDCl₃, 400 MHz) δ: 6.89 (1H $_{\rm f}$ $_{\rm J}$ = 7.2 Hz ,H-3) 2.24 (2H ,m ,H-4) ,1.66 (2H ,m ,H-5) 5.90 (1H ,dd , $_{\rm J}$ = 17.3 ,10.7 Hz ,H-7) 5.09 (1H ,d $_{\rm J}$ = 10.7 Hz ,H-8α) 5.23 (1H ,d $_{\rm J}$ = 17.3 Hz ,H-8β) ,1.82 (3H $_{\rm F}$,H-9) ,1.31 (3H $_{\rm F}$,H-10); ¹³C NMR (CDCl₃,100 MHz) δ: 173.1 (C-1) ,127.1 (C-2) ,144.5 (C-3) ,23.6 (C-4) ,40.4 (C-5) ,73.1 (C-6) ,144.3 (C-7) ,112.3 (C-8) ,12.0 (C-9) 27.9 (C-10) 。以上数据与文献 [4] 报道数据基本一致 故鉴定该化合物为($_{\rm E}$) -linalool-1-oicacid。

化合物 3 黄色油状, C_{13} H_{18} O_{3} , H NMR (CDCl₃ $\not=$ 400 MHz) δ : 2. 50 (1H ,d ,J = 17. 2 Hz ,H-2 α) 2. 36 (1H ,d ,J = 17. 2 Hz ,H-2 β) 5. 96 (1H $\not=$ 5, H-4) δ . 83 (1H ,d ,J = 16. 0 Hz ,H-7) δ . 47 (1H ,d ,J = 16. 0 Hz ,H-8) 2. 31 (3H $\not=$,H-40) ,1. 11 (3H $\not=$ 5, H-41) ,1. 02 (3H $\not=$,H-42) ,1. 89 (3H $\not=$,H-43); f CNMR (CDCl₃ ,100 MHz) δ : 41. 4 (C-1) ,49. 5 (C-2) ,197. 0 (C-3) ,127. 7 (C-4) ,160. 4 (C-5) ,79. 2 (C-6) ,145. 0 (C-7) ,130. 3 (C-8) ,197. 5 (C-9) ,28. 4 (C-10) 22. 9 (C-11) 24. 3 (C-12) ,18. 7 (C-13) $\not=$ 以上数据与文献 f 报道数据基本一致,故鉴定该化合物为(+) 去氢催吐萝芙木醇 (dehydrovomifoliol) $\not=$

化合物 4 黄色油状, C_{13} H_{20} O_{3} , 1 H 1 H 1 H 2 H $^{$

化合物 5 白色粉末, C₃₀ H₄₈ O₃, H NMR (CDCl₃, 500 MHz) δ: 5. 28 (1H, br s, H-12), 2. 82 (1H, dd, J = 14. 0, 4. 0 Hz, H-18), 0. 90 (3H, s, H-18)

白色粉末,C₃₀ H₄₆ O₃, H NMR 化合物 6 (CDCl₃ 400 MHz) δ: 5. 29 (1H ,br s ,H-12) ,2. 83 (1 H, dd, J = 13.3, 3.4 Hz, H + 18), (1.04, 3 H, s, H + 18)23) 0.79 (3H ,s ,H-24) 0.92 (3H ,s ,H-25) ,1.07 (3H s ,H-26) ,1. 13 (3H s ,H-27) ,0. 90 (3H s ,H-29) ,1. 02 (3H ,s ,H-30); ¹³ C NMR (CDCl₃ ,100 MHz) δ : 39. 2 (C-1) ,33. 9 (C-2) ,217. 9 (C-3) , 47. 4 (C-4) 55. 2 (C-5) ,19. 6 (C-6) 32. 2 (C-7) , 39. 0 (C-8) ,46. 8 (C-9) ,36. 7 (C-10) ,22. 9 (C-11) ,122.3 (C-12) ,143.1 (C-13) ,41.7 (C-14) , 27.6 (C-15) 23.7 (C-16) 46.5 (C-17) 41.0 (C-18) 46.0 (C-19) 30.6 (C-20) 34.1 (C-21) 32.2 (C-22) 26.4 (C-23) 21.4 (C-24) ,15.0 (C-25) , 17. 0 (C-26) 25. 8 (C-27) 184. 2 (C-28) 33. 0 (C-29) 23.5 (C-30)。以上数据与文献^[7]报道数据基 tooleanolic acid) o

化合物7 白色粉末,ESI-MS(m/z): 475 [M + Na] + ,HR-EI-MS(m/z): 452. 3298 (C_{30} H₄₄ O₃ ,calcd for 452. 3290); [α] + 225. 3 (c 0. 18 ,MeOH); IR (KBr) ν_{max} 3443 ,2945 ,2867 ,1705 ,1632 ,1463 and 1384 cm +; UV (MeOH) λ_{max} 283 nm (0. 48) 201 nm (0. 17); H NMR (CDCl₃ ,400 MHz) δ : 5. 58 (1H , d ,J = 5. 7 Hz ,H-1) .0. 90 ρ . 94 ,1. 00 ,1. 03 ,1. 06 ,1. 10 ,1. 23 (each 3H ρ ,7 × CH₃); C NMR (CDCl₃ ,100 MHz) δ : 37. 6 (C-1) ,34. 4 (C-2) ,217. 9 (C-3) ,47. 2 (C-4) ,51. 7 (C-5) ,19. 5 (C-6) ,31. 2 (C-7) ,42. 5 (C-8) ,152. 6 (C-9) ,38. 2 (C-10) ,117. 4 (C-11) ,120. 4 (C-12) ,145. 4 (C-13) ,40. 6 (C-14) ,26. 9

(C-15) 23.5 (C-16) 45.7 (C-17) 39.4 (C-18) , 45.8 (C-19) 30.6 (C-20) 33.6 (C-21) 32.0 (C-22) 26.8 (C-23) 21.2 (C-24) 25.1 (C-25) ,19.9 (C-26) 20.0 (C-27) ,182.9 (C-28) 23.5 (C-29) , 32.9 (C-30) 。结合文献^[7-9]所报道的数据 ,确定该化合物为 3-oleana-9⁽¹¹⁾ ,12-diene-28-oic acid ,该化合物在文献中为合成的三萜衍生物 ,并修正了其 1D-NMR 数据。

白色粉末,C₃₀ H₄₆ O₃, H NMR 化合物 8 (CDCl₃ 400 MHz) δ : 0. 58 (1H ,d ,J = 4. 0 Hz ,H- 19α) 0.78 (1H ,d J = 4.0 Hz ,H-49 β) 6.90 (1H , t J = 7.2 Hz, H-24) ,0. 99 ,1. 84 ,1. 04 ,1. 09 ,0. 90 (each 3H ,s ,H-18 ,27 \sim 30); ¹³ C NMR (CDCl₃ ,100 MHz) δ : 33.4 (C-1),37.5 (C-2),216.7 (C-3), 50. 2 (C-4) 48. 4 (C-5) 21. 5 (C-6) 25. 8 (C-7) , 47.8 (C-8) ,21.0 (C-9) ,25.9 (C-10) ,26.6 (C-11) 32.7 (C-12) 45.3 (C-13) 48.7 (C-14) 35.5 (C-45) 28.1 (C-46) 52.2 (C-47) ,18.1 (C-48) , 29.5 (C-49) 35.9 (C-20) ,18.1 (C-21) 25.9 (C-22) 34.7 (C-23) ,145.8 (C-24) ,126.6 (C-25) , 173. 2 (C-26) ,11. 9 (C-27) ,22. 1 (C-28) ,20. 7 (C-29) 19.3 (C-30)。以上数据与文献[10] 报道数据基本 一致 故鉴定该化合物为甘五酸(mangiferonic acid)。

白色粉末,C₃₀ H₄₈ O₂, H NMR 化合物 9 (CDCl₃ A00 MHz) δ : 5. 70 (1H ,dt ,J = 15.6 ,7. 6 Hz ,H-23) 6.20 (1H ,d ,J = 15.6 Hz ,H-24) 4.90(2H ,brs ,H-26) ,1.00 ,0.94 ,1.14 ,1.85 ,1.07 ,1.03 , 0. 88 (each 3H ,s ,H-18 ,19 ,21 ,27 ~ 30); ¹³ C NMR (CDCl₃, 100 MHz) δ: 39. 9 (C-1) 34. 1 (C-2) 218. 0 (C-3) 47.3 (C-4) 55.4 (C-5) ,19.6 (C-6) 34.5 (C-7) 40.3 (C-8) 50.0 (C-9) 36.8 (C-10) 22.0 (C-11) ,27.5 (C-12) ,42.5 (C-13) ,50.2 (C-14) , 31. 1 (C-15) ,24. 9 (C-16) ,50. 0 (C-17) ,15. 2 (C-18) ,16. 0 (C-19) ,75. 2 (C-20) ,26. 0 (C-2) ,43. 9 (C-22) ,125.7 (C-23) ,136.5 (C-24) ,141.9 (C-25) ,115. 2 (C-26) ,18. 6 (C-27) , 26. 7 (C-28) ,21. 0 (C-29),16.3 (C-30)。以上数据与文献[11]报道数

化合物 10 白色粉末, C_{29} H_{50} O_{2} , H NMR (CDC1₃,400 MHz) δ : 3.52 (1H, m, H-3),5.28 (1H, m, H-6), 3.84 (1H, m, H-7), 0.69 (3H, s, H-18), 1.04 (3H, s, H-19), 0.92 (3H, d, J=6.4 Hz, H-21), 0.82 (3H, m, H-26), 0.80 (3H, m, H-28),

0.84 (3H ,m ,H-29); ¹³C NMR (CDCl₃ ,100 MHz) δ: 36.9 (C-l) 31.5 (C-2) ,71.4 (C-3) ,41.7 (C-4) ,143.4 (C-5) ,125.4 (C-6) ,73.3 (C-7) ,40.8 (C-8) ,48.2 (C-9) ,36.4 (C-10) ,21.0 (C-11) ,39.5 (C-12) ,42.8 (C-13) ,55.3 (C-14) ,26.3 (C-15) ,28.5 (C-16) ,55.9 (C-17) ,11.8 (C-18) ,19.1 (C-19) ,36.1 (C-20) ,18.8 (C-21) ,33.9 (C-22) ,26.0 (C-23) ,45.8 (C-24) ,29.1 (C-25) ,19.8 (C-26) ,19.0 (C-27) ,23.0 (C-28) ,11.9 (C-29) 。以上数据与文献^[12]报道数据基本一致 ,故鉴定该化合物为7-羟基β-谷甾醇(7β-hydroxy-β-sitosterol)。

无色油状 ,C31 H36 O11 ,ESIMS(m/ 化合物 12 z):583 [M-H]⁻¹, H NMR (CDCl₃, A00 MHz) δ : 6.63 (2H s ,H-2 ,6) A. 76 (2H ,m ,H-7 ,7') 3. 13 (2H , m ,H-8 β ') A. 12 (2H ,m ,H α -9 β ') A. 30 (2H ,m , $H\beta - 9, 9'$), 6.73 (1H, dd, J = 8.2, 1.9 Hz, H-6''), 4. 99 (1H ,d ,J = 3.3 Hz ,H-7") ,3. 88 ,3. 89 ,3. 90 (12H s $4 \times OCH_3$); ¹³C NMR (CDCl₃, 100 MHz) δ : 131.1 (C-1),102.7 (C-2,6),153.4 (C-3,5), 134. 2 (C-4) ,86. 0 (C-7) ,54. 5 (C-8) ,72. 1 (C-9) ,137.7 (C-1') ,118.7 (C-2') ,146.6 (C-3') , 144. 8 (C-4') ,108. 3 (C-5') ,114. 2 (C-6') ,85. 7 (C-7') 54.0 (C-8') 71.5 (C-9') ,132.6 (C-1'') , 108. 6 (C-2") ,146. 7 (C-3") ,145. 3 (C-4") ,114. 3 (C-5''), 118. 9 (C-6''), 72. 4 (C-7''), 87. 0 (C-8''), 60.5 (C-9") 56.2 55.9 (2×OCH₃)。以上数据与 文献[14] 报道数据基本一致,故鉴定该化合物为 ficusesquilignan A.

 $H\beta$ -9 β ′) ,6. 83 (1H ,dd ,J = 8.1 ,1.8 Hz ,H-6") ,5. 02 (1H ,d ,J = 9.0 Hz ,H-7") ,3. 88 ,3. 89 ,3. 90 (12H β β × OCH $_3$) ; 13 C NMR (CDCl $_3$,100 MHz) δ : 132. 6 (C-1) ,102. 7 (C-2 ,6) ,153. 1 (C-3 ,5) ,134. 6 (C-4) ,85. 7 (C-7) ,54. 0 (C-8) ,71. 5 (C-9) ,137. 7 (C-1′) ,114. 3 (C-2′) ,145. 3 (C-3′) ,146. 6 (C-4′) ,108. 6 (C-5′) ,118. 9 (C-6′) ,85. 9 (C-7′) 54. 5 (C-8′) ,72. 1 (C-9′) ,131. 1 (C-1″) ,109. 8 (C-2″) ,146. 7 (C-3″) ,145. 3 (C-4″) ,114. 2 (C-5″) ,120. 3 (C-6″) ,74. 0 (C-7″) ,89. 0 (C-8″) ,60. 5 (C-9″) β 6. 2 β 5. 9 (2 × OCH $_3$) 。以上数据与文献 $^{[14]}$ 报道数据基本一致 ,故鉴定该化合物为 ficusesquilignan B。

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