

灰毛豆 *Tephrosia purpurea* 中查尔酮类化合物的分离鉴定 Study on Chalcones from *Tephrosia purpurea*

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摘要:【目的】为探讨灰毛豆属植物 *Tephrosia purpurea* 化学成分中的查尔酮类化合物, 对其枝、叶部位进行化学成分分离及结构解析。【方法】采用乙醇提取、溶剂萃取、柱层析分离方法得到多种灰毛豆属 *T. purpurea* 化学成分, 并用波谱分析手段对其进行结构表征。【结果】从灰毛豆属 *T. purpurea* 中共分离鉴定出 11 个查尔酮类化合物, 分别为 elatadihydrochalcone (1), spinochalcone A (2), 3',5'-diisopentenyl-2',4'-dihydroxychalcone (3), candidachalcone (4), *O*-methylpongamol (5), (+)-tephrosone (6), spinochalcone C (7), epoxyobovatachalcone (8), tunicatachalcone (9), crassichalcone (10), (*S*)-elatadihydrochalcone (11)。【结论】以上化合物均为查尔酮, 且均属首次从该植物中分离得到。

关键词: 灰毛豆 查尔酮类化合物 分离 结构鉴定

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Abstract:【Objective】There is a rich resource of *Tephrosia purpurea* in China. The chemical components from different parts of *T. purpurea* were investigated in order to explore their structures.【Methods】The chalcones were isolated and purified by column chromatography on silica gel, and their structures were identified by spectral analyses and compared with literature.【Results】Eleven chalcones chemical components were obtained from the branches and leaves of *T. purpurea* and characterized as: elatadihydrochalcone (1), spinochalcone A (2), 3',5'-diisopentenyl-2',4'-dihydroxychalcone (3), candidachalcone (4), *O*-methylpongamol (5), (+)-tephrosone (6), spinochalcone C (7), epoxyobovatachalcone (8), tunicatachalcone (9), crassichalcone (10) and (*S*)-elatadihydrochalcone (11).【Conclusion】All the compounds mentioned above belong to chalcones, which are isolated from *T. purpurea* for the first time.

Key words: *Tephrosia purpurea*, chalcones chemical component, isolation, structure identification

0 引言

【研究意义】灰毛豆属是一类大量分布于热带的豆科植物, 已超过 350 多种, 在台湾、海南、福建、广东、广西、云南等地区皆有分布^[1,2]。它具有重要的传统用途^[3,4], 其提取物还具有多种生物活性, 如抗

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病毒^[5], 抗原生动物^[6], 细胞毒素^[7]和抗疟原虫^[8]等活性。深入研究灰毛豆属植物 *Tephrosia purpurea* 的化学成分, 有利于丰富灰毛豆属植物的化学成分数据库, 强化其化学分类学意义。【前人研究进展】关于灰毛豆属植物化学成分的研究始于 1910 年^[9], 目前报道的主要化合物类型有黄酮、查尔酮、鱼藤酮、甾体、异黄酮等^[6, 8, 10~14]。【本研究切入点】目前国内外对灰毛豆属植物的研究主要集中在根、茎部位, 着重探究其杀虫及医学活性, 而其化学成分分离及其结构解析鲜见报道。【拟解决的关键问题】对灰毛豆属植物 *Tephrosia purpurea* 进行系统的化学成分分离及其结构解析, 并在后期进行查尔酮类化合物的筛选。

1 材料与方法

1.1 材料

超导核磁共振波谱仪: Bruker DRX-400 及 AM-500 型(Bruker, 德国); 质谱仪: 美国 PE 公司 GC-MS API 2000 型(ESI-MS); 红外光谱仪: WQF-401FT-IR(Beifen-Ruili, 中国); 紫外分光光度计: PE Lamda 25(Wellesley, 美国); 旋转蒸发仪: N-1100V-W(日本东京理化株式会社)。

材料: 正相硅胶(100~200 目, 200~300 目)均为青岛海洋所化工厂产品; Sephadex LH-20 为 Pharmacia 公司产品; 实验所用其它试剂均为分析纯及化学纯, 由北京化工厂生产。 *T. purpurea* 枝叶部位, 采摘于广东省湛江市雷州市, 经华南植物鉴定中心鉴定为灰毛豆 *T. purpurea*。

1.2 分离纯化

T. purpurea 枝、叶(共 10 kg)经干燥粉碎后用 5 倍量 95% 的工业乙醇提取 3 次, 每次过夜。提取液合并, 减压回收溶剂后得到总浸膏。浸膏以水混悬, 依次用正己烷、乙酸乙酯、正丁醇进行萃取, 得到正己烷萃取物(A)180 g, 乙酸乙酯萃取物(E) 220 g, 正丁醇萃取物(T) 480 g, 水部位(W)约 600 g。实验主要进行乙酸乙酯萃取物(E)和正丁醇萃取物(T)的细化分离。

1.3 化合物的结构鉴定

运用 25℃, 内标法(NMR)、电喷雾电离质谱(MS)、KBr 压片法(IR)、分光光度法(UV)等方法测定获得的单体化合物 1~11 相关光谱数据, 与文献对比相关数据, 以确定获得的单体化合物 1~11 的结构。

2 结果与分析

2.1 化合物的分离纯化

化合物 1~7 制备: *T. purpurea* 枝叶的乙酸乙酯萃取物(220 g)经正相硅胶(100~200 目)柱层析, 用正己烷-乙酸乙酯(99:1; 49:1; 20:1; 10:1; 5:1; 2:1, V:V)进行洗脱, 得到 E1~E6 流分; 然后再分别经正相硅胶(100~200 目)柱层析, 用正己烷-乙酸乙酯-甲醇(200:100:1; 200:100:2; 200:100:5; 200:100:10; 200:100:20; 200:100:50, V:V:V)进行洗脱, 得到 E1-1~E1-6, E2-1~E2-6, E3-1~E3-6, E4-1~E4-6, E5-1~E5-6, E6-1~E6-6 等共 36 个流分。流分 E1-3 经硅胶柱色谱分离, 氯仿-甲醇(5:1, V:V)洗脱、正己烷-氯仿-甲醇(1:1:2, V:V:V)洗脱、Sephadex LH-20 甲醇洗脱, 得化合物 1(15 mg); 流分 E2-3 经硅胶柱层析, 以氯仿-甲醇(8:1, V:V)洗脱得到 E2-3-1~E2-3-5, E2-3-3 再经 Sephadex LH-20 用甲醇洗脱从第 3 和 4 亚流分得化合物 2(11 mg)和化合物 3(10 mg); E2-4 经 Sephadex LH-20 用甲醇洗脱得到 E2-4-1~E2-4-6, E2-4-4 经硅胶(氯仿-甲醇, 6:1, V:V)柱层析, 得化合物 4(9 mg)和化合物 5(21 mg); E3-1 经 Sephadex LH-20 用氯仿-甲醇(1:2, V:V)洗脱从第 3 亚流分得化合物 6(15 mg); E3-2 经多次 Sephadex LH-20 用甲醇洗脱得化合物 7(7 mg)。

化合物 8~11 制备: *T. purpurea* 枝叶的正丁醇萃取物(480 g)经正相硅胶(100~200 目)柱层析, 用正己烷-乙酸乙酯(99:1; 49:1; 20:1; 5:1; 2:1, V:V)进行洗脱, 得到 T1~T5 流分。

T1-1 经多次 Sephadex LH-20 用甲醇洗脱, 第 2 和 4 亚流分分别经硅胶(氯仿-甲醇, 6:1, V:V)柱层析后得化合物 8(13 mg)和化合物 9(11 mg)。T2 经硅胶柱色谱以正己烷-氯仿-甲醇(1:1:2, V:V:V)洗脱, 亚流分 T2-3 再经硅胶柱色谱以氯仿-甲醇(8:1, V:V)洗脱得到化合物 10(4 mg); 亚流分 T2-4 经 2 次 Sephadex LH-20 甲醇洗脱得到化合物 11(8 mg)。

2.2 化合物的结构鉴定

化合物 1: UV λ_{\max} (MeOH) nm: 277, 295, 309; ESI-MS m/z 355. 15 [M+1]⁺. ¹H-NMR (600 MHz, CDCl₃) δ_{H} : 13. 87 (1H, s, OH-2'), 7. 25 - 7. 46 (5H, m, H-2/6, 3/5, 4), 6. 70 (1H, d, J = 10. 1 Hz, H-4''), 5. 88 (1H, s, H-5'), 5. 43 (1H, d, J = 10. 1 Hz, H-3''), 5. 30 (1H, dd, J = 3. 0, 9. 0 Hz, H- β), 3. 75 (3H, s, OMe-6'), 3. 47 (2H,

dd, $J = 3.0, 18.0$ Hz, H- α), 1.48 (6H, s, 2Me-2''); $^{13}\text{C-NMR}$ (150 MHz, CDCl_3) δ_{c} : 204.4 (C-CO), 163.1 (C-6'), 161.68 (C-2'), 160.8 (C-4'), 143.3 (C-1), 128.4 (C-3,5), 127.6 (C-4), 125.8 (C-2,6), 125.4 (C-3''), 115.9 (C-4''), 105.5 (C-1'), 102.7 (C-3'), 91.3 (C-5'), 78.3 (C-2''), 70.4 (C- β), 55.5 (OMe-6'), 52.7 (C- α), 28.5 (2Me-2''). 以上数据与文献[18]对照基本一致, 因此将化合物**1**鉴定为 elatadihydrochalcone。

化合物**2**: UV λ_{max} (MeOH) nm: 262, 358; IR ν_{max} (KBr) cm^{-1} : 3351, 1642, 1532, 1381. $^1\text{H-NMR}$ (600 MHz, CDCl_3): 13.44 (1H, s, OH-2'), 7.94 (1H, d, $J = 16$ Hz, H- β), 7.71 (1H, d, $J = 16$ Hz, H- α), 7.63 (1H, s, H-6'), 7.35-7.56 (5H, m, H-2,3,4,5,6), 6.09 (1H, s, OH-4'), 5.26 (2H, t, $J = 7.2$ Hz, H-2'',2'''), 3.45 (2H, d, $J = 7$ Hz, H-1''), 3.36 (2H, d, $J = 7$ Hz, H-1'''), 1.84 (6H, s, 2Me-3''), 1.76 (6H, s, 2Me-3'''); $^{13}\text{C-NMR}$ (150 MHz, CD_3OD) δ_{c} : 192.81 (C-CO), 162.19 (C-2'), 161.63 (C-4'), 145.1 (C- β), 135.2 (C-1), 131.8 (C-3'',3'''), 128.6 (C-3,5), 128.5 (C-2,6), 127.9 (C-4), 123.9 (C-6'), 123.1 (C-2'',2'''), 120.0 (C-5'), 116.6 (C-3'), 24.6 (C-4'',4'''), 18.6 (C-5'',5'''). 以上数据与文献[15]对照基本一致, 因此将化合物**2**鉴定为 spinochalcone A。

化合物**3**: UV λ_{max} (MeOH) nm: 230, 370; IR ν_{max} (KBr) cm^{-1} : 3340, 1450, 980. $^1\text{H-NMR}$ (600 MHz, CDCl_3) δ_{H} : 13.81 (1H, s, H-2'), 7.83 (1H, d, $J = 15$ Hz, H- β), 7.74 (1H, d, $J = 15$ Hz, H- α), 7.34-7.56 (5H, m, H-2,3,4,5,6), 6.72 (1H, s, H-6'), 3.47 (2H, d, $J = 3$ Hz, H-1''), 3.38 (2H, d, $J = 3$ Hz, H-1'''), 1.79 (12H, s, 4Me-3'',3'''); $^{13}\text{C-NMR}$ (150 MHz, CD_3OD) δ_{c} : 191.75 (C-CO), 162.65 (C-4'), 160.25 (C-2'), 143.87 (C- β), 134.79 (C-6'), 130.41 (C-4,3''), 129.31 (C-3,5), 128.90 (C-2''), 128.47 (C-2'''), 128.35 (C-2,6), 118.83 (C- α), 114.43 (C-3'), 113.70 (C-1',5'), 29.21 (C-4''), 25.92 (C-4'''), 21.99 (C-1'',1'''), 18.33 (C-5'',5'''). 以上数据与文献[16]对照基本一致, 因此将化合物**4**鉴定为 3',5'-diisopentenyl-2',4'-dihydroxychalcone。

化合物**4**: 黄色粉末; IR ν_{max} (KBr) cm^{-1} : 3450, 1650, 1555, 1513, 1462, 1420, 1262, 1144 cm^{-1} ; ESI-MS m/z 370.16 $[\text{M}]^+$; $^1\text{H-NMR}$ (600 MHz, CDCl_3) δ_{H} : 14.62 (1H, brs, OH), 7.77 (2H, s, H- α,β), 7.53 (2H, d, $J = 8.0$ Hz, H-1,6), 6.85 (2H, d, $J = 8.0$ Hz, H-3,5), 6.69 (1H, d, $J = 9.5$ Hz, H-1''), 5.91 (1H, s, H-5'), 5.44 (d, $J = 9.5$ Hz, H-2''), 3.93 (3H, s, OMe-6'), 1.45 (6H, s, 2Me-3''); $^{13}\text{C-NMR}$ (150 MHz, CD_3OD) δ_{c} : 192.6 (C-CO), 162.5 (C-2'), 162.5 (C-4'), 160.2 (C-6'), 157.4 (C-4), 142.2 (CH- β), 130.3 (C-6), 130.34 (C-2), 125.3 (C- α), 128.5 (C-1), 125.3 (C-2''), 116.1 (C-1''), 115.8 (C-3,5), 107.1 (C-1'), 106.0 (C-3'), 91.5 (C-5'), 78.19 (C-3''), 55.9 (C-OMe), 28.4 (C-2Me)。以上数据与文献[17]对照基本一致, 因此将化合物**4**鉴定为 candidachalcone。

化合物**5**: $^1\text{H-NMR}$ (600 MHz, CDCl_3) δ_{H} : 6.17 (3H, s, $\text{OCH}_3-\beta$), 5.94 (3H, s, OMe-6'), 3.74 (1H, s, H- α), 2.31-3.22 (2H, m, H-2,6); $^{13}\text{C-NMR}$ (150 MHz, CD_3OD) δ_{c} : 191.1 (C-CO), 170.6 (C- β), 158.3 (C-2'), 152.9 (C-4'), 144.6 (C-8'), 135.5 (C-1), 129.6 (C-4), 129.0 (C-2,6), 127.6 (C-3,5), 126.7 (C-6'), 119.1 (C-1'), 106.5 (C-7'), 105.1 (C-5'), 103.2 (C- α), 61.3 (OMe-6'), 56.4 (OMe- β)。以上数据与文献[10]对照基本一致, 因此将化合物**5**鉴定为 *O*-methylpongamol。

化合物**6**: IR ν_{max} (KBr) cm^{-1} : 3435, 1635. $^1\text{H-NMR}$ (600 MHz, CDCl_3) δ_{H} : 13.62 (1H, s, OH-6'), 7.91 (1H, d, $J = 15.5$ Hz, H- β), 7.85 (1H, d, $J = 8$ Hz, H-2'), 7.64 (2H, m, H-2,6), 7.56 (1H, d, $J = 15.5$ Hz, H- α), 7.45 (3H, m, H-3,4,5), 6.59 (1H, d, $J = 6.4$ Hz, H-2''), 6.48 (1H, d, $J = 8.7$ Hz, H-3'), 4.36 (1H, s, H-4''), 4.09 (1H, d, $J = 6.4$ Hz, H-3''), 1.06 / 1.42 (6H, s, 2Me-5''); $^{13}\text{C-NMR}$ (150 MHz, CDCl_3) δ_{c} : 192.7 (C-CO), 165.9 (C-4'), 161.8 (C-6'), 145.3 (C- β), 135.1 (C-1), 132.8 (C-2'), 130.9 (C-4), 129.5 (C-6), 128.9 (C-2,6), 120.7 (C- α), 115.5 (C-5'), 114.2 (C-1'), 113.2 (C-2''), 102.8 (C-3'), 88.6 (C-5''), 80.4 (C-4''), 55.6 (C-3''), 23.5 / 27.9 (2Me-5'')。以上数据与文献[18]对照基本一致, 因此将化合物**6**鉴定为 (+)-tephrosone。

化合物**7**: 黄色油状; UV λ_{max} (MeOH) nm: 263, 392; IR ν_{max} (KBr) cm^{-1} : 3405, 1635, 1545, 1385; ESI-MS m/z 374.17 $[\text{M}]^+$; $^1\text{H-NMR}$ (600 MHz, CDCl_3) δ_{H} : 12.17 (1H, s, OH-2'), 7.92 (1H, d, $J = 16$ Hz, H- β), 7.70 (2H, m, H-2,6), 7.61 (1H, d, $J = 16$ Hz, H- α), 7.53 (2H, d, $J = 8.0$ Hz, H-1,6), 6.85 (2H, d, $J = 8.0$ Hz, H-3,5), 6.69 (1H, d, $J = 9.5$ Hz, H-1''), 5.91 (1H, s, H-5'), 5.44 (d, $J = 9.5$ Hz, H-2''), 3.93 (3H, s, OMe-6'), 1.45 (6H, s, 2Me-3''); $^{13}\text{C-NMR}$ (150 MHz, CD_3OD) δ_{c} : 192.6 (C-CO), 162.5 (C-2'), 162.5 (C-4'), 160.2 (C-6'), 157.4 (C-4), 142.2 (CH- β), 130.3 (C-6), 130.34 (C-2), 125.3 (C- α), 128.5 (C-1), 125.3 (C-2''), 116.1 (C-1''), 115.8 (C-3,5), 107.1 (C-1'), 106.0 (C-3'), 91.5 (C-5'), 78.19 (C-3''), 55.9 (C-OMe), 28.4 (C-2Me)。以上数据与文献[17]对照基本一致, 因此将化合物**4**鉴定为 candidachalcone。

化合物**7**: 黄色油状; UV λ_{max} (MeOH) nm: 263, 392; IR ν_{max} (KBr) cm^{-1} : 3405, 1635, 1545, 1385; ESI-MS m/z 374.17 $[\text{M}]^+$; $^1\text{H-NMR}$ (600 MHz, CDCl_3) δ_{H} : 12.17 (1H, s, OH-2'), 7.92 (1H, d, $J = 16$ Hz, H- β), 7.70 (2H, m, H-2,6), 7.61 (1H, d, $J = 16$ Hz, H- α), 7.53 (2H, d, $J = 8.0$ Hz, H-1,6), 6.85 (2H, d, $J = 8.0$ Hz, H-3,5), 6.69 (1H, d, $J = 9.5$ Hz, H-1''), 5.91 (1H, s, H-5'), 5.44 (d, $J = 9.5$ Hz, H-2''), 3.93 (3H, s, OMe-6'), 1.45 (6H, s, 2Me-3''); $^{13}\text{C-NMR}$ (150 MHz, CD_3OD) δ_{c} : 192.6 (C-CO), 162.5 (C-2'), 162.5 (C-4'), 160.2 (C-6'), 157.4 (C-4), 142.2 (CH- β), 130.3 (C-6), 130.34 (C-2), 125.3 (C- α), 128.5 (C-1), 125.3 (C-2''), 116.1 (C-1''), 115.8 (C-3,5), 107.1 (C-1'), 106.0 (C-3'), 91.5 (C-5'), 78.19 (C-3''), 55.9 (C-OMe), 28.4 (C-2Me)。以上数据与文献[17]对照基本一致, 因此将化合物**4**鉴定为 candidachalcone。

$J = 16$ Hz, H- α), 7.59 (1H, d, $J = 16$ Hz, H-6'), 7.47 (3H, m, H-3,4,5), 6.75 (1H, d, $J = 10$ Hz, H-4''), 5.403 (1H, d, $J = 10$ Hz, H-3''), 5.25 (1H, t, $J = 7.2$ Hz, H-2'''), 3.42 (2H, d, $J = 7$ Hz, H-1'''), 1.84/1.72 (6H, s, 2Me-3'''), 1.56 (6H, s, 2Me-2''); $^{13}\text{C-NMR}$ (150 MHz, CDCl_3) δ_{C} : 192.7 (C-CO), 166.7 (C-4'), 163.6 (C-2'), 145.3 (C- β), 135.3 (C-1), 132.8 (C-3'''), 128.6 (C-3,5), 128.5 (C-2,6), 128.2 (C-3''), 127.9 (C-4), 124.1 (C-6'), 123.2 (C-2'''), 122.2 (C-4''), 119.6 (C-3'), 118.8 (C- α), 114.8 (C-5'), 111.2 (C-1'), 85.5 (C-2''), 28.5 (2Me-2''), 24.7/18.9 (2Me-3'''), 22.6 (C-1'''). 以上数据与文献[19]对照基本一致,因此将化合物**7**鉴定为 spinochalcone C。

化合物**8**: 黄色油状; UV λ_{max} (MeOH) nm: 352, 298; IR ν_{max} (KBr) cm^{-1} : 3436, 1620, 1585; ESI-MS m/z 352.12 $[\text{M}]^+$; $^1\text{H-NMR}$ (600 MHz, CDCl_3) δ_{H} : 15.32 (1H, s, OH-2'), 7.91 (1H, d, $J = 16$ Hz, H- β), 7.83 (1H, d, $J = 16$ Hz, H- α), 7.65 (2H, m, H-2,6), 7.49 (3H, m, H-3,4,5), 5.95 (1H, s, H-5'), 4.87 (1H, d, $J = 7.2$ Hz, H-7'), 3.96 (3H, s, OMe-6'), 3.79 (1H, d, $J = 7.2$ Hz, H-8'), 1.43 (6H, s, 2Me-9'); $^{13}\text{C-NMR}$ (150 MHz, CDCl_3) δ_{C} : 192.7 (C-CO), 160.9 (C-6'), 158.9 (C-2'), 157.8 (C-4'), 145.6 (C- β), 134.7 (C-1), 127.7 (C-3,5), 127.6 (C-2,6), 126.9 (C-4), 125.7 (C- α), 107.5 (C-3'), 104.9 (C-1'), 92.4 (C-5'), 84.7 (C-9'), 69.9 (C-8'), 54.5 (OMe-6'), 46.8 (C-7'), 25.9 (2Me-9')。以上数据与文献[20]对照基本一致,因此将化合物**8**鉴定为 epoxyobovatachalcone。

化合物**9**: ESI-MS m/z 406.16 $[\text{M}]^+$; $^1\text{H-NMR}$ (600 MHz, CDCl_3) δ_{H} : 15.18 (1H, s, OH-9), 7.87 (1H, d, $J = 16.7$ Hz, H-7), 7.74 (1H, d, $J = 16.7$ Hz, H-8), 7.57-7.60 (2H, m, H-2,6), 7.40-7.42 (3H, m, H-3,4,5), 5.55 (1H, s, H-5'), 4.88 (2H, d, $J = 7.4$ Hz, H-2',2''), 3.93 (3H, s, OMe-6'), 2.65 (4H, d, $J = 7.4$ Hz, H-1',1''), 1.58 (12H, brs, $J = 3$ Hz, H-4'',4''',5'',5'''); $^{13}\text{C-NMR}$ (150 MHz, CDCl_3) δ_{C} : 205.8 (C-2'), 198.4 (C-4'), 178.9 (C-9), 171.3 (C-6'), 143.5 (C-7), 134.9 (C-1), 134.9 (C-3'',3'''),

130.6 (C-4), 129.3 (C-3,5), 128.7 (C-2,6), 122.9 (C-8), 118.5 (C-2'',2'''), 105.8 (C-1'), 98.9 (C-5'), 62.4 (C-3'), 56.5 (OMe-6'), 38.7 (C-1'',1'''), 25.9 (C-5'',5'''), 17.8 (C-4'',4''')。以上数据与文献[21]对照基本一致,因此将化合物**9**鉴定为 tunicatachalcone。

化合物**10**: 黄色油状; UV λ_{max} (MeOH) nm: 348, 21; IR ν_{max} (KBr) cm^{-1} : 3025, 1650, 1605, 1130, 1110; ESI-MS m/z 336.16 $[\text{M}]^+$; $^1\text{H-NMR}$ (600 MHz, CDCl_3) δ_{H} : 7.86 (1H, d, $J = 15.8$ Hz, H- β), 7.75 (1H, d, $J = 15.8$ Hz, H- α), 7.58 (2H, m, H-2,6), 7.37 (3H, m, H-3,4,5), 5.98 (1H, s, H-5'), 5.30 (1H, dd, $J = 9.9, 7.5$ Hz, H-8'), 5.06 (1H, s, H-10'b), 4.91 (1H, s, H-10'a), 3.92 (3H, s, OMe), 3.30 (1H, dd, $J = 9.9, 15$ Hz, H-7'eq), 2.94 (1H, dd, $J = 7.5, 15$ Hz, H-7'ax), 1.78 (3H, s, H-11'); $^{13}\text{C-NMR}$ (150 MHz, CDCl_3) δ_{C} : 193.9 (C-CO), 169.40 (C-4'), 162.9 (C-6'), 160.9 (C-2'), 145.5 (C- β), 143.3 (C-9'), 135.5 (C-1), 126.8 (C-3,5), 126.9 (C-2,6), 125.5 (C-4), 124.8 (C- α), 112.8 (C-11'), 107.4 (C-3'), 105.8 (C-1'), 92.9 (C-5'), 88.6 (C-8'), 55.4 (OMe-6'), 30.9 (C-7), 17.5 (C-10')。以上数据与文献[22]对照基本一致,因此将化合物**10**鉴定为 crassichalcone。

化合物**11**: 黄色晶体; ESI-MS m/z 350.42 $[\text{M}]^+$; $^1\text{H-NMR}$ (600 MHz, CDCl_3) δ_{H} : 7.52 (2H, m, H-2,6), 7.41 (1H, m, $J = 16.1$ Hz, H- α), 7.36 (3H, m, H-3,4,5), 6.60 (1H, m, $J = 16.1$ Hz, H- β), 6.55 (1H, m, $J = 10.2$ Hz, H-4''), 6.27 (1H, s, H-3'), 5.56 (1H, m, $J = 10.2$ Hz, H-3''), 3.76 (6H, s, OMe-2',6'), 1.48 (6H, s, 2Me-2''); $^{13}\text{C-NMR}$ (150 MHz, CDCl_3) δ_{C} : 194.5 (C-CO), 158.5 (C-2'), 156.5 (C-6'), 154.9 (C-4'), 144.9 (C- α), 134.96 (C-1), 130.5 (C-3''), 129.5 (C-2,6), 128.9 (C- β), 128.7 (C-3,5), 127.9 (C-4), 116.8 (C-4''), 116.5 (C-5'), 108.4 (C-1'), 96.4 (C-3'), 78.5 (C-3''), 63.6 (OMe-6'), 55.9 (OMe-2'), 27.9 (2Me-2'')。以上数据与文献[23]对照基本一致,因此将化合物**11**鉴定为 (S)-elatadihydrochalcone。

化合物**1~11**的化学结构如图1所示。

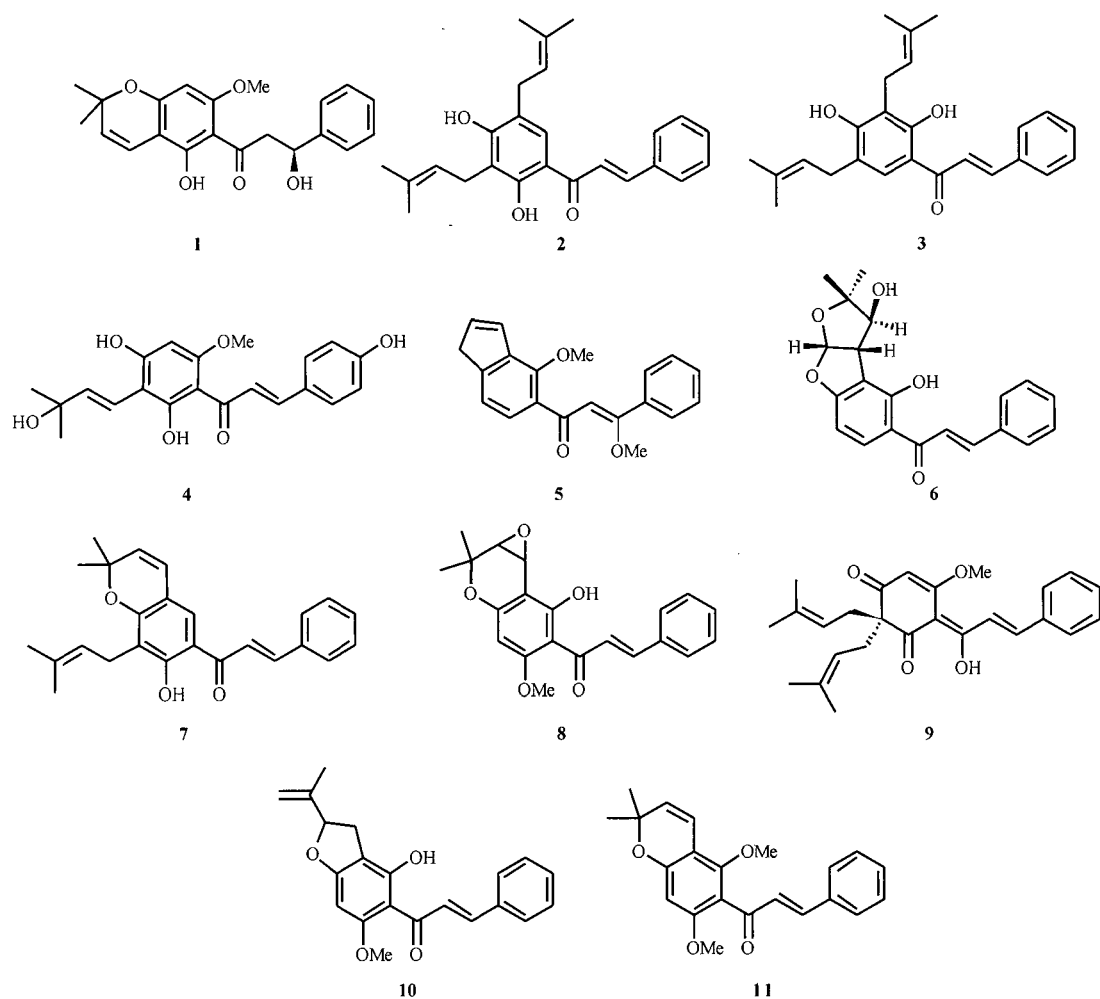


图1 化合物1~11结构

Fig.1 Structures of compounds 1~11

3 结论

本研究对采自湛江雷州的灰毛豆属植物 *T. purpurea* 的枝、叶部位进行化学成分分离及其结构解析,从中共分离鉴定出 11 个查尔酮类化合物,分别为 elatadihydrochalcone (1), spinochalcone A (2), 3',5'-diisopentenyl-2',4'-dihydroxychalcone (3), candidachalcone (4), *O*-methylpongamol (5), (+)-tephrosone (6), spinochalcone C (7), epoxybovat-achalcone (8), tunicatachalcone (9), crassichalcone (10), (*S*)-elatadihydrochalcone (11)。以上化合物均为首次从 *T. purpurea* 中分离得到,丰富了灰毛豆属植物的化学成分数据库。

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