

文章编号: 1001-6880(2009)04-0604-04

牡丹籽的化学成分研究

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摘要: 从毛茛科芍药属牡丹 *Paeonia suffruticosa* Andr 种子甲醇提取物中分离得到 13 个化合物, 通过理化性质及波谱分析鉴定为齐墩果酸 (oleanolic acid, 1)、12, 13-dehydromicromeric acid (2)、常春藤皂甙元 (hederagenin, 3)、山奈酚 (kaempferol, 4)、木犀草素 (luteolin, 5)、芹菜素 (apigenin, 6)、柯伊利素 (chrysoeriol, 7)、反式葡萄糖素 (trans-viniferin, 8)、顺式葡萄糖素 (cis-viniferin, 9)、反式白藜芦醇 (trans-resveratrol, 10)、谷甾醇 (-sitosterol, 11)、豆甾醇 (stigmasterol, 12) 和 胡萝卜苷 (-daucosterol, 13)。所有化合物均首次从该植物的种子部位中得到。

关键词: 化学成分; 结构鉴定; 牡丹; 牡丹籽; 芍药属

中图分类号: R284.1; Q946.91

文献标识码: A

Studies on Chemical Constituents from Seeds of *Paeonia suffruticosa* Andr.

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Abstract: Thirteen compounds were isolated from seeds of *Paeonia suffruticosa* Andr. Comparison of the physico-chemical and NMR properties with the reported data allowed for their structures to be identified as oleanolic acid (1), 12, 13-dehydromicromeric acid (2), hederagenin (3), kaempferol (4), luteolin (5), apigenin (6), chrysoeriol (7), trans-viniferin (8), cis-viniferin (9), trans-resveratrol (10), -sitosterol (11), stigmasterol (12), and -daucosterol (13). All compounds were isolated from seeds of *Paeonia suffruticosa* Andr. for the first time.

Key words: chemical constituents; structural identification; *Paeonia suffruticosa*; seeds; *Paeonia*

牡丹 (*Paeonia suffruticosa* Andr.) 属毛茛科芍药属灌木, 在我国各地均有广泛种植。其干燥根皮 (又名丹皮) 为一种常用的中草药, 味辛苦, 性凉, 功能清热凉血, 活血化瘀, 具有抗炎、抗心肌缺血、保肝、降血糖和调节免疫细胞等作用功效^[1,2]。丹皮含有牡丹皮原苷 (酶解后生成丹皮酚和丹皮酚苷)、芍药苷、芍药酚、挥发油、甾醇生物碱以及植物甾醇等。吴少华等^[3]从丹皮中分离出白桦脂酸、白桦脂醇、齐墩果酸、芍药苷元、丹皮酚、6-羟基香豆素、没食子酸等 9 个化合物。赵帆平等^[4-6]又从牡丹皮中分离出丹皮多糖, 筛选出了降血糖的有效成分 PSM2b。牡丹籽在民间常用于治疗腰腿疼痛, 具有

抗氧化、消炎止痛作用^[7], 作为潜在的药用资源, 有待深入开发利用。目前牡丹籽多用于育种, 并没有开发其药用价值, 而牡丹籽的化学成分研究目前也无文献报道。本实验对牡丹籽甲醇提取物进行分离, 经鉴定得到 13 个化合物, 13 个化合物均首次从该植物种子部位中得到。

1 仪器与材料

Bruker AM-400M 和 Bruker DRX-500M 核磁共振仪 (TMS 作内标); 质谱仪 (VG AUTO Spec-3000 或 Finnigan MAT90); Agilent 1100 高效液相色谱仪 (美国 Agilent 公司); 显微熔点测定仪 (X-4, 北京泰克仪器有限公司); 薄层色谱用硅胶 GF₂₅₄ (青岛海洋化工厂); 200~300 目柱层析硅胶 (青岛海洋化工厂); Sephadex LH-20 (美国 GE 公司); MCI gel CHP-20P 和 RP-18 (日本三菱化工公司)。其余试剂均为

收稿日期: 2008-11-20

接受日期: 2009-02-05

基金项目: 河南省洛阳市科技发展计划项目 (0602042-5)

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分析纯、工业试剂重蒸后用。

本试验用牡丹籽于 2007 年 8 月中旬采摘于河南省洛阳市国家牡丹园, 经河南科技大学食品与生物工程学院朱文学教授鉴定为凤丹牡丹 *Paeonia suffruticosa* Andr 种子。

2 提取与分离

牡丹籽 1.0 kg, 在 45 烘箱中干燥至质量恒定, 用粉碎机破碎至一定粒度, 用甲醇回流提取 3 次, 每次 3 h。提取液过滤后减压浓缩成浸膏, 回收甲醇得到牡丹籽提取物 (123.2 g)。用 80% 甲醇溶解, 然后用正己烷回流 2 次, 每次 3 h, 去除牡丹籽中油脂成分, 80% 甲醇提取物得 84.8 g。然后加入 10% 甲醇溶液使之成悬浊液, 再用乙醚萃取 3 次, 乙醚部份 (24.6 g) 以硅胶柱层析, 用氯仿-甲醇梯度洗脱 (10:1:4:1) 得到 7 个流分 (Fr₁ ~ Fr₇)。流分₁ 经硅胶柱层析 (石油醚-丙酮 = 8:1), 然后再用丙酮重结晶得化合物 11 (116 mg) 和 12 (29 mg)。流分₂ 和₃ 合并后上硅胶柱层析 (氯仿-甲醇 = 8:1), 继而上 Sephadex LH-20 色谱柱反复用甲醇洗脱得化合物 3 (36 mg), 4 (24 mg), 10 (12 mg)。流分₄ 经 Sephadex LH-20 (甲醇) 得化合物 5 (10 mg), 6 (8 mg), 7 (14 mg)。流分₅ 经硅胶柱层析 (氯仿-甲醇 = 9:1:8:1) 和 Sephadex LH-20 (甲醇) 得化合物 1 (14 mg), 2 (11 mg) 和 13 (230 mg)。流分₆ 经硅胶柱层析 (氯仿:甲醇 = 5:1)、Sephadex LH-20 (甲醇)、MCI-gel CHP-20P 和 RP-18 反相硅胶柱层析反复纯化得化合物 8 (106 mg) 和 9 (6 mg)。

3 结构鉴定

化合物 1 白色粉末。 $C_{30}H_{48}O_3$, mp. 312 ~ 315 , EIMS m/z : 456 [M]⁺。¹H NMR (400 MHz, CDCl₃) : 5.49 (H, brs, H-12), 3.46 (H, dd, J = 11.2, 4.6 Hz, H-3), 1.24, 1.19, 1.02, 1.01, 0.96, 0.89, 0.82 (each 3H, s, H-23, 24, 25, 26, 27, 29, 30); ¹³C NMR (100 MHz, CDCl₃) : 39.1 (t, C-1), 28.2 (t, C-2), 78.2 (d, C-3), 39.5 (s, C-4), 55.9 (d, C-5), 18.8 (t, C-6), 33.4 (t, C-7), 39.8 (s, C-8), 48.2 (d, C-9), 37.5 (s, C-10), 23.8 (t, C-11), 122.7 (d, C-12), 144.9 (s, C-13), 42.3 (s, C-14), 28.3 (t, C-15), 23.9 (t, C-16), 48.2 (s, C-17), 42.1 (d, C-18), 46.8 (t, C-19), 31.1 (s, C-20), 34.6 (t, C-21), 33.3 (t, C-22), 28.9 (q, C-23), 16.7 (q, C-24), 15.7 (q, C-25), 17.5

(q, C-26), 26.3 (q, C-27), 180.3 (s, C-28), 33.4 (q, C-29), 23.9 (q, C-30)。以上波谱数据与文献报道^[8]一致, 故鉴定为齐墩果酸 (Oleanolic acid)。

化合物 2 白色针晶, $C_{30}H_{48}O_3$, mp. 327 ~ 329 , EIMS m/z : 456 [M]⁺ (35), 189 [M]⁺ (100)。¹H NMR (400 MHz, CDCl₃) : 5.12 (1H, d, J = 10.8 Hz, H-30a), 4.68 (1H, d, J = 10.8 Hz, H-30b), 3.28 (1H, dd, J = 11.0, 4.6 Hz, H-3), 1.79, 1.22, 1.06, 1.04, 1.00, 0.81 (each 3H, s, H-23, 24, 25, 26, 27, 29); ¹³C NMR (100 MHz, CDCl₃) : 39.4 (t, C-1), 28.0 (t, C-2), 78.3 (d, C-3), 39.7 (s, C-4), 56.1 (d, C-5), 18.9 (t, C-6), 31.3 (t, C-7), 41.2 (s, C-8), 47.9 (d, C-9), 37.6 (s, C-10), 21.3 (t, C-11), 37.6 (t, C-12), 51.1 (d, C-13), 43.0 (s, C-14), 30.4 (t, C-15), 26.2 (t, C-16), 56.8 (s, C-17), 38.7 (d, C-18), 49.9 (d, C-19), 151.5 (s, C-20), 35.0 (t, C-21), 33.0 (t, C-22), 28.8 (q, C-23), 15.1 (q, C-24), 16.5 (q, C-25), 16.6 (q, C-26), 16.6 (q, C-27), 179.1 (s, C-28), 19.6 (q, C-29), 110.2 (t, C-30)。以上波谱数据与文献报道^[9]一致, 故鉴定为 12, 13-dehydromicromeric acid。

化合物 3 白色晶体, $C_{30}H_{48}O_4$, mp. 345 ~ 347 , EIMS m/z : 472 [M]⁺。¹H NMR (500 MHz, CDCl₃) : 5.83 (H, brs, H-12), 4.22 (H, d, J = 10.8 Hz, H-3), 4.19 (H, d, J = 10.2 Hz, H-23a), 3.73 (H, d, J = 10.2 Hz, H-23b), 3.31 (H, dd, J = 4.2, 11.2 Hz, H-5), 1.22, 1.04, 1.04, 0.98, 0.95, 0.91 (each 3H, s, H-24, 25, 26, 27, 29, 30); ¹³C NMR (125 MHz, CDCl₃) : 38.8 (t, C-1), 27.7 (t, C-2), 73.5 (d, C-3), 42.9 (s, C-4), 48.8 (d, C-5), 18.6 (t, C-6), 33.0 (t, C-7), 39.8 (s, C-8), 48.2 (d, C-9), 37.3 (s, C-10), 23.9 (t, C-11), 123.4 (d, C-12), 145.0 (s, C-13), 42.2 (s, C-14), 28.4 (t, C-15), 23.8 (t, C-16), 46.7 (s, C-17), 42.1 (d, C-18), 46.5 (t, C-19), 31.0 (s, C-20), 34.3 (t, C-21), 33.3 (t, C-22), 68.0 (t, C-23), 13.2 (q, C-24), 16.0 (q, C-25), 17.5 (q, C-26), 26.2 (q, C-27), 180.2 (s, C-28), 33.3 (q, C-29), 23.7 (q, C-30)。以上波谱数据与文献报道^[10]一致, 故鉴定为常春藤皂甙元 (Hederagenin)。

化合物 4 黄色晶体, $C_{15}H_{10}O_6$, mp. 278 ~ 280 , EIMS m/z (%): 286 [M]⁺ (100), 285 (22), 258 [M-CO]⁺ (10), 121 (21); ¹H NMR (C_5D_5N , 500 Hz) : 6.70 (1H, s, H-6), 6.79 (1H, s, H-8), 8.44 (2H,

d, *J* = 8.2 Hz, H-2, 6), 7.28 (2H, *d*, *J* = 8.2 Hz, H-3, 5); ¹³C NMR (C₅D₅N, 100 Hz) : 146.7 (s, C-2), 135.4 (s, C-3), 176.1 (s, C-4), 160.4 (s, C-5), 98.0 (d, C-6), 164.0 (s, C-7), 92.8 (d, C-8), 156.1 (s, C-9), 102.9 (s, C-10), 121.8 (s, C-1), 129.1 (×2, d, C-2, 6), 115.3 (×2, d, C-3, 5), 159.2 (s, C-4)。以上波谱数据与文献报道^[8]一致,故鉴定为山柰酚(Kaempferol)。

化合物 5 黄色粉末, C₁₅H₁₀O₆, mp. 333 ~ 335 , FAB⁻MS *m/z* 285 [M-1]⁻。¹H NMR (400 MHz, C₅D₅N) : 7.91 (H, *d*, *J* = 2.0 Hz, H-2), 7.55 (H, dd, *J* = 2.0, 8.4 Hz, H-6), 7.29 (H, *d*, *J* = 8.4 Hz, H-5), 7.20 (1H, s, H-8), 6.93 (1H, s, H-3), 6.72 (1H, s, H-6); ¹³C NMR (100 MHz, C₅D₅N) : 164.9 (s, C-2), 104.0 (d, C-3), 182.9 (s, C-4), 158.6 (s, C-5), 100.0 (d, C-6), 165.9 (s, C-7), 94.9 (d, C-8), 163.2 (s, C-9), 105.0 (s, C-10), 123.0 (s, C-1), 114.7 (d, C-2), 147.9 (s, C-3), 148.8 (s, C-4), 116.9 (d, C-5), 119.6 (d, C-6)。以上波谱数据与文献报道^[11]一致,故鉴定为木犀草素(Luteolin)。

化合物 6 黄色粉末, C₁₅H₁₀O₅, mp. 339 ~ 341 , FAB⁻MS *m/z* 269 [M-1]⁻。¹H NMR (500 MHz, C₅D₅N) : 7.65 (2H, *d*, *J* = 8.3 Hz, H-2, 6), 7.28 (2H, *d*, *J* = 8.3 Hz, H-3, 5), 7.20 (1H, s, H-8), 6.86 (H, *d*, *J* = 1.6 Hz, H-8), 6.75 (1H, s, H-3), 6.75 (1H, *d*, *J* = 1.6 Hz, H-6); ¹³C NMR (125 MHz, C₅D₅N) : 164.6 (s, C-2), 104.0 (d, C-3), 182.8 (s, C-4), 158.5 (s, C-5), 100.0 (d, C-6), 163.2 (s, C-7), 94.9 (d, C-8), 162.7 (s, C-9), 104.3 (s, C-10), 121.4 (s, C-1), 128.9 (×2, d, C-2, 6), 162.0 (s, C-4), 116.9 (×2, d, C-3, 5)。以上波谱数据与文献报道^[11]一致,故鉴定为芹菜素(Apigenin)。

化合物 7 黄色粉末, C₁₆H₁₂O₅, mp. 326 ~ 328 , EIMS *m/z* (%): 300 [M]⁺。¹H NMR (400 MHz, C₅D₅N) : 7.65 (H, dd, *J* = 2.0, 8.2 Hz, H-6), 7.61 (H, *d*, *J* = 2.0 Hz, H-2), 7.28 (H, *d*, *J* = 8.2 Hz, H-5), 7.20 (1H, s, H-3), 6.86 (1H, *d*, *J* = 2.0 Hz, H-8), 6.76 (1H, *d*, *J* = 2.0 Hz, H-6), 3.81 (3H, s, OCH₃); ¹³C NMR (100 MHz, C₅D₅N) : 164.6 (s, C-2), 104.2 (d, C-3), 182.8 (s, C-4), 158.6 (s, C-5), 100.0 (d, C-6), 166.0 (s, C-7), 95.0 (d, C-8), 163.2 (s, C-9), 105.0 (s, C-10), 122.6 (s, C-1), 110.3 (d, C-2), 149.0 (s, C-3), 152.5 (s, C-4), 117.0 (d, C-

5), 121.4 (d, C-6), 56.1 (q, OCH₃)。以上波谱数据与文献报道^[11]一致,故鉴定为柯伊利素(Chrysoeriol)。

化合物 8 黄色固体, C₂₈H₂₂O₆, mp. 152 ~ 155 , FAB⁻MS *m/z*: 453 [M-1]⁻。¹H NMR (500 MHz, CD₃OD) : 7.16 (2H, *d*, *J* = 8.5 Hz, H-2, 6), 7.05 (2H, *d*, *J* = 8.5 Hz, H-2, 6), 6.85 (H, *d*, *J* = 16.3 Hz, H-7), 6.78 (2H, *d*, *J* = 8.5 Hz, H-3, 5), 6.67 (2H, *d*, *J* = 8.5 Hz, H-3, 5), 6.65 (1H, *t*, *J* = 2.0 Hz, H-12), 6.59 (H, *d*, *J* = 16.3 Hz, H-8), 6.26 (H, *d*, *J* = 2.0 Hz, H-14), 6.20 (H, *d*, *J* = 2.0 Hz, H-12), 6.17 (2H, *d*, *J* = 2.0 Hz, H-10, 14), 5.38 (1H, *d*, *J* = 6.6 Hz, H-7), 4.36 (1H, *d*, *J* = 6.6 Hz, H-8); ¹³C NMR (125 MHz, CD₃OD) : 133.9 (s, C-1), 128.8 (×2, *d*, C-2, 6), 116.4 (×2, *d*, C-3, 5), 159.7 (s, C-4), 94.8 (d, C-7), 58.3 (d, C-8), 147.4 (s, C-9), 107.5 (×2, *d*, C-10, 14), 160.0 (×2, s, C-11, 13), 102.2 (d, C-12), 130.4 (s, C-1), 128.8 (×2, *d*, C-2, 6), 116.3 (×2, *d*, C-3, 5), 158.5 (s, C-4), 130.3 (d, C-7), 123.7 (d, C-8), 136.9 (s, C-9), 120.1 (s, C-10), 162.7 (s, C-11), 96.9 (d, C-12), 159.7 (s, C-13), 104.3 (d, C-14)。以上波谱数据与文献报道^[12]一致,故鉴定为反式葡萄素(trans-Viniferin)。

化合物 9 黄色固体, C₂₈H₂₂O₆, mp. 171 ~ 173 , FAB⁻MS *m/z* (%): 453 [M-1]⁻ (100)。¹H NMR (500 MHz, CD₃OD) : 7.14 (2H, *d*, *J* = 8.5 Hz, H-2, 6), 7.03 (2H, *d*, *J* = 8.5 Hz, H-2, 6), 6.75 (2H, *d*, *J* = 8.5 Hz, H-3, 5), 6.64 (2H, *d*, *J* = 8.5 Hz, H-3, 5), 6.24 (H, *d*, *J* = 2.0 Hz, H-14), 6.21 (H, *d*, *J* = 2.0 Hz, H-12), 6.18 (H, *d*, *J* = 12.0 Hz, H-7), 6.11 (1H, *t*, *J* = 2.0 Hz, H-12), 6.08 (H, *d*, *J* = 12.0 Hz, H-8), 5.94 (2H, *d*, *J* = 2.0 Hz, H-10, 14), 5.16 (1H, *d*, *J* = 6.6 Hz, H-7), 3.81 (1H, *d*, *J* = 6.6 Hz, H-8); ¹³C NMR (125 MHz, CD₃OD) : 133.7 (s, C-1), 128.9 (×2, *d*, C-2, 6), 116.2 (×2, *d*, C-3, 5), 157.8 (s, C-4), 94.6 (d, C-7), 57.5 (d, C-8), 147.1 (s, C-9), 106.9 (×2, *d*, C-10, 14), 159.8 (×2, s, C-11, 13), 101.6 (d, C-12), 129.8 (s, C-1), 129.1 (×2, *d*, C-2, 6), 116.1 (×2, *d*, C-3, 5), 158.2 (s, C-4), 131.0 (d, C-7), 126.5 (d, C-8), 137.1 (s, C-9), 120.2 (s, C-10), 162.8 (s, C-11), 96.6 (d, C-12), 159.5 (s, C-13), 108.7 (d, C-14)。以上波谱数据

与文献报道^[12]一致,故鉴定为顺式葡萄糖(Viniferin)。

化合物 10 淡黄色晶体,mp. 288~291, EI-MS m/z (%): 228 [M]⁺; ¹H NMR (400 MHz, CD₃OD) : 7.31 (2H, d, J = 8.4 Hz, H-2, 6), 6.90 (1H, d, J = 16.4 Hz, H-7), 6.72 (1H, d, J = 16.4 Hz, H-8), 6.69 (2H, d, J = 8.4 Hz, H-3, 5), 6.39 (2H, d, J = 2.0 Hz, H-2, 6), 6.07 (1H, t, J = 2.0 Hz, H-4); ¹³C NMR (100 MHz, CD₃OD) : 140.5 (s, C-1), 105.2 (d, C-2), 159.8 (x2, s, C-3, 5), 102.1 (d, C-4), 108.0 (d, C-6), 131.1 (s, C-1), 129.2 (d, C-2), 116.1 (d, C-3), 158.2 (s, C-4), 116.3 (d, C-5), 128.9 (d, C-6), 130.5 (d, C-7), 127.1 (d, C-8)。以上波谱数据与文献报道^[12]一致,故鉴定为反式白藜芦醇(trans-Resveratrol)。

化合物 11 无色结晶, C₂₉H₅₀O, 在 TLC 上多种溶剂系统展开时化合物与标准品 谷甾醇的 R_f 值相同,确认化合物为 谷甾醇(β -Sitosterol)。

化合物 12 白色粉末, C₂₉H₄₈O, 在 TLC 上多种溶剂系统展开时化合物与豆甾醇的 R_f 值相同,确认化合物为豆甾醇(Stigmasterol)。

化合物 13 白色粉末, C₃₅H₆₀O₆, EI-MS m/z : 456 [M]⁺。在 TLC 上多种溶剂系统展开时化合物与 胡萝卜苷的 R_f 值相同,确认化合物为 胡萝卜苷(β -Daucosterol)。

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