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金樱根化学成分的研究

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摘 要: 金樱根为三金片的主要成分,但目前对于金樱根的化学成分和药理作用研究甚少。为了阐明金樱根的物质基础和生物活性,该研究采用硅胶、Sephadex LH-20、MCI gel CHP 20P 等柱色谱以及 HPLC 半制备等方法,对金樱根(*Rosa laevigata*)的化学成分进行研究。结果表明:从中共分离得到 9 个化合物,经过波谱数据分析结合文献对照分别鉴定为儿茶素(**1**), 表儿茶素(**2**), rosamultin (**3**), sericoside (**4**), 2α, 3α, 19α, 23-tetrahydroxy-urs-12-en-28-oic acid-3-O-β-D-glucopyranosyl ester(**5**), kaji-ichigoside F1(**6**), β-D-Glucopyranosyl 3β, 19α-dihydroxy-2-oxo-urs-12-en-28-oate(**7**), 胡萝卜苷(**8**), β-谷甾醇(**9**), 其中化合物 **2**、**4**、**5**、**7** 为首次从该植物中分离得到。该研究结果为金樱根在功能医药领域的开发利用提供了理论依据。

关键词: 金樱根, 化学成分, 结构鉴定
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Chemical constituents from the roots of *Rosa laevigata*

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Abstract: The roots of *Rosa laevigata* were the main ingredients of the Sanjin tablet, at present, the studies on the chemical constituents and pharmacological of the *R. laevigata* were relatively insufficient. In order to elucidate the material basis and the activity of *R. laevigata*, the constituents of *R. laevigata* were isolated by silica gel, Sephadex LH-20, MCI gel CHP 20P column chromatography and semi-preparative HPLC. Their structures were elucidated by anlalyzing their spectral data and comparing with the previously reported literatures. Nine compounds: (+)-catechin (**1**), (-)-epicatechin (**2**), rosamultin (**3**), sericoside (**4**), 2α, 3α, 19α, 23-tetrahydroxy-urs-12-en-28-oic acid-3-O-β-D-glucopyranosyl ester (**5**), kaji-ichigoside F1 (**6**), β-D-Glucopyranosy 3β, 19α-dihydroxy-2-oxo-urs-12-en-28-oate (**7**), daucosterol (**8**), β-sitosterol (**9**) were obtained. Compounds (**2**), (**4**), (**5**) and (**7**) were reported from the plant for the first time. The results provide scientific information for exploitation and medicine utilization of *R. laevigata*.

Key words: roots of *Rosa laevigata*, chemical constituents, structure identification

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中药金樱子(*Rosa laevigata*)为蔷薇科蔷薇属灌木植物,主要分布于我国华东、中南、西南等地。《本草纲目》中记载:金樱子,性酸、涩、平、无毒;主治脾泻下痢、止小便利、涩精气。研究表明,金樱子具有抗氧化、保护肾脏肝脏、降低血糖血脂、抗菌抗病毒、增强抗炎以及增强免疫力的作用。

国内外学者从金樱子的果实中和叶中已经分离纯化得到了甾体及甾体皂苷类、三萜及三萜皂苷类、木脂素、黄酮、可水解鞣质、多糖等多种化学成分。但对于金樱地下部分的物质基础研究不多,为了更全面地掌握金樱根的药效物质基础,本研究运用现代分离手段和鉴定技术,从金樱根 60%乙醇提取物乙酸乙酯萃取部位分离得到化合物 9 个。

1 材料与方法

1.1 仪器与材料

仪器:瑞士 Bruker DRX-500 MHz 超导核磁共振仪;N-1100 旋转蒸发仪;CF810C 冷却循环水;硅胶薄层板 F_{254}(0.2 mm thick Merck KGaA Darmstadt, Germany); MCI gel CHP 20P (70~150 μm; Mitsubishi Chemical; Tokyo, Japan); Sephadex LH-20(25~100 μm, GE Healtheare Bio-science AB, Uppsala, Sweden);所有试剂均为分析纯。

材料:金樱根药材由桂林三金股份有限公司周艳林博士提供并鉴定。

1.2 提取与分离

干燥的金樱根 8.5 kg,用 60%的乙醇浸提 2 次,提取液浓缩得到浸膏,浸膏依次经过石油醚、乙酸乙酯、正丁醇萃取。将乙酸乙酯萃取部位(292 g)经硅胶(200~300 目)柱色谱,分别用氯仿,氯仿∶甲醇(98∶2,95∶5,9∶1,8∶2,7∶3,5∶5),纯甲醇洗脱。经 TLC 检测合并得到 7 个流份。流分 5(10.1 g)经反复 Sephadex LH-20 柱色谱、MCI gel CHP 20P 柱色谱以及半制备 HPLC 方法分离纯化,得化合物 **1**(27 mg)、**2**(18 mg)、**3**(167 mg)、**4**(46 mg)、**5**(40 mg)、**6**(18 mg)、**7**(63mg)、**8**(30 mg)、**9**(16 mg)。

2 结构鉴定

化合物 **1** 黄色无晶型粉末,分子式 $C_{15}H_{14}O_6$。^{1}H-NMR (500 MHz, methanol-d_4) δ: 2.51(1H, dd, J=8.1, 16.2 Hz, H-4a), 2.85(1H, dd, J=5.3, 16.1 Hz, H-4b), 3.98 (1H, m, H-3), 4.57(1H, d, J=7.5,H-2), 5.86 (1H, d, J=2.2 Hz, H-6), 5.93 (1H, d, J=2.2 Hz, H-8), 6.72 (1H, d, J=8.1 Hz, H-5′), 6.77 (1H, dd, J=1.8, 8.1 Hz, H-6′), 6.84 (1H, d, J=1.8 Hz, H-2′); ^{13}C-NMR (125 MHz, methanol-d_4) δ: 27.8 (C-4), 66.1 (C-3), 78.5 (C-2), 94.5 (C-6), 95.1 (C-8), 98.7 (C-1), 113.9 (C-2′), 114.5 (C-6′), 118.0 (C-5′), 130.9 (C-1′), 44.4 (C-3′), 144.5 (C-4′), 155.9 (C-9), 156.3 (C-7), 156.6 (C-5)。上述波谱数据与关小丽等(2014)报道一致,故鉴定 **1** 为儿茶素。

化合物 **2** 黄色无晶型粉末,分子式 $C_{15}H_{14}O_6$。^{1}H-NMR (500 MHz, methanol-d_4) δ: 2.74 (1H, dd, J=2.8, 16.8 Hz, H-4ax), 2.86 (1H, dd, J=4.6, 16.7Hz, H-4eq), 4.18 (1H, s, H-3), 4.81 (1H, d, J=4.5Hz, H-2), 5.94 (1H, d, J=2.0 Hz, H-6), 6.04 (1H, d, J=2.0 Hz, H-8), 6.77 (1H, d, J=8.2 Hz, H-5′), 6.80 (1H, d, J=8.2 Hz, H-6′), 6.98 (1H, d, J=1.6 Hz, H-2′); ^{13}C-NMR(125 MHz, methanol-d4) δ: 27.8 (C-4), 65.9 (C-3), 78.0 (C-2), 94.3 (C-8), 94.7 (C-6), 98.3 (C-1), 114.0(C-5′), 114.4 (C-2′), 117.8 (C-6′), 131.9 (C-1′), 144.9(C-3′), 145.0 (C-4′), 155.8 (C-5), 156.4 (C-9), 156.6 (C-7)。上述波谱数据与张朝凤等(2003)报道一致,故鉴定 **2** 为表儿茶素。

化合物 **3** 白色针晶,分子式 $C_{36}H_{58}O_{10}$, ESI-MS m/z: 649 [M - H]$^-$, 673 [M + Na]$^+$。^{1}H-NMR (500 MHz, methanol-d_4) δ: 0.79, 0.82, 1.03, 1.03, 1.22, 1.34(each 3H, s, CH_3), 0.94(3H, d, J=6.6 Hz, CH_3), 2.48 (1H, s, H-18), 2.93 (1H, d, J=9.7 Hz, H-3), 3.32~3.70 (m), 3.82 (1H, dd, J=2.0, 11.9 Hz, H-2), 5.32 (1H, s, H-12), 5.34 (1H, d, J=8.2, H-1′); ^{13}C-NMR(125 MHz, methanol-d_4), δ: 15.3 (q, C-30), 15.8 (q, C-26), 16.1 (q, C-24), 16.3 (q, C-25), 18.3 (t, C-6),23.3 (t, C-11),23.4 (q, C-27),25.2 (t, C-16), 25.7 (t, C-21), 25.8 (t, C-29), 28.0 (t, C-15), 28.3 (q, C-23), 32.7 (t, C-7), 36.9 (t, C-22), 37.8 (s, C-4), 39.1 (s, C-10), 39.9 (s, C-8), 41.3 (s, C-14), 41.5 (d, C-20), 46.8 (t, C-1), 47.2 (d, C-9), 48.1 (t, C-17), 53.6 (d, C-18), 55.3 (d, C-5), 61.1 (t, C-6′), 68.2 (d, C-2), 69.7 (d, C-4′), 72.3 (s, C-19), 72.5 (d, C-2′), 76.9 (d, C-

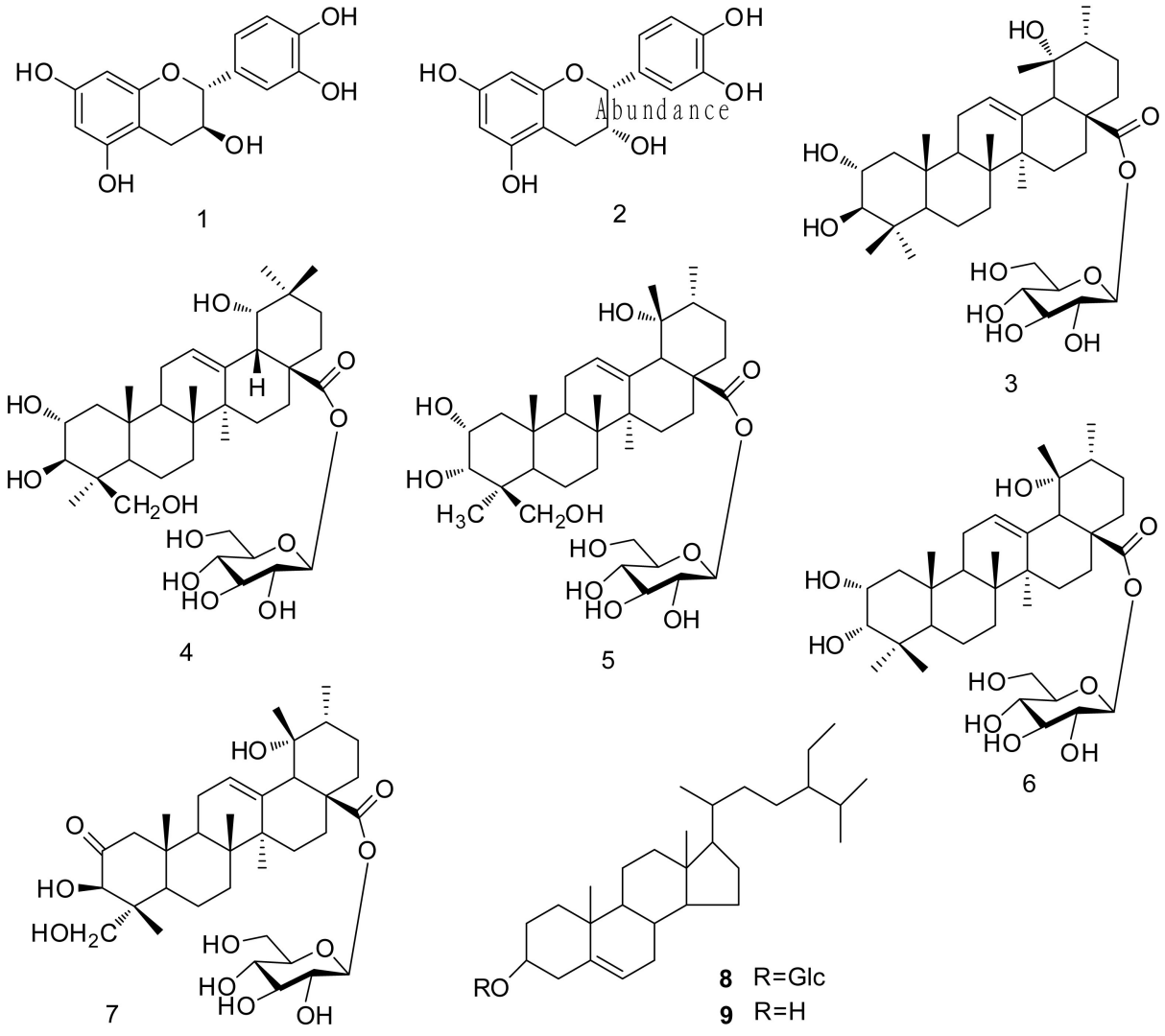

图 1　化合物 **1**–**9** 的结构式

Fig. 1　Chemical structures of compounds **1**–**9**

6′), 77.1 (d, C-3′), 83.2 (d, C-3), 94.4 (d, C-1′), 128.1 (d, C-12), 138.3 (s, C-13), 177.1 (s, C-28)。上述数据与吴小鹏等(2014)报道一致,故鉴定 **3** 为 rosamltin。

化合物 **4**　白色晶体,分子式 $C_{36}H_{58}O_{11}$,ESI-MS *m/z*: 665 [M− H]$^-$, 689 [M + Na]$^+$。^{1}H-NMR (500 MHz, methanol-d_4) δ: 0.74, 0.96, 0.97, 1.00, 1.25, 1.30 (each 3H, s, CH_3), 4.05 (1H, d, *J* = 11.2, H-24b), 3.83 (1H, d, *J* = 11.2, H-24a), 5.27 (1H, s, H-1′), 5.38 (1H, d, *J* = 8.2, H-12); ^{13}C-NMR (125MHz, methanol-d_4), δ: 17.4 (q, C-25), 17.7 (q, C-26), 20.0 (t, C-6), 23.8 (q, C-23), 25.0 (q, C-27), 25.1 (t, C-11), 25.2 (q, C-30), 28.4 (t, C-16), 28.6 (q, C-29), 29.4 (t, C-21), 29.5 (t, C-15), 33.2 (t, C-22), 34.1 (t, C-7), 35.9 (s, C-20), 39.2 (s, C-10), 40.8 (s, C-8), 42.6 (s, C-14), 44.3 (s, C-4), 45.0 (d, C-18), 47.1 (s, C-17), 47.7 (t, C-1), 49.2 (d, C-9), 57.2 (d, C-5), 62.4 (t, C-6′), 66.1 (t, C-24), 69.6 (d,C-2), 71.0 (d, C-4′), 73.8 (d, C-2′), 78.2 (d, C-3′), 78.6 (d, C-5′), 82.4 (d, C-19), 85.9 (d, C-3), 95.7 (d, C-1′), 124.7 (d, C-12), 144.3 (s, C-13), 178.5 (s, C-28)。以上波谱数据与李延芳等(2003)报道一致,故鉴定 **4** 为 sericoside。

化合物 **5**　白色晶体,分子式 $C_{36}H_{58}O_{11}$, ESI-MS *m/z*: 665 [M - H]$^-$, 689 [M + Na]$^+$。^{1}H-NMR

(500 MHz, methanol-d_4) δ: 0.79, 0.90, 1.03, 1.22, 1.36 (each 3H, s, CH_3), 0.94 (3H, d, J = 9.5 Hz), 2.55 (1H, s, H-18), 5.27 (1H, s, H-1′), 5.36 (1H, d, J=8.2, H-12); ^{13}C-NMR (125 MHz, methanol-d_4) δ: 16.6 (q, C-30), 17.2 (q, C-24), 17.3 (q, C-26), 17.7 (q, C-25), 19.0 (t, C-6), 24.6 (q, C-27), 24.7 (t, C-11), 24.8 (t, C-16), 26.4 (t, C-21), 27.1 (t, C-29), 29.5 (t, C-15), 33.5 (t, C-7), 38.1 (t, C-22), 39.0 (s, C-10), 41.2 (s, C-8), 42.2 (d, C-20), 42.4 (s, C-14), 42.6 (s, C-4), 42.8 (t, C-1), 44.1 (d, C-5), 48.2 (d, C-9), 49.3 (t, C-17), 55.8 (d, C-18), 62.4 (t, C-6′), 67.1 (d, C-2), 71.0 (d, C-4′), 71.2 (q, C-23), 73.6 (s, C-19), 73.7 (d, C-2′), 78.1 (d, C-5′), 78.3 (d, C-3), 78.5 (d, C-3′), 95.6 (d, C-1′), 129.4 (d, C-12), 139.5 (d, C-13), 178.4 (s,C-28)。以上数据与刘岱琳等(2010)报道基本一致,故鉴定 **5** 为 2α,3α,19α,23-tetrahydroxy-urs-12-en-28-oic acid-3-O-β-D-glucopyranosyl ester。

化合物 **6** 白色晶体,分子式 $C_{36}H_{58}O_{10}$, ESI-MS m/z: 649 [M - H]$^-$, 673 [M + Na]$^+$。^{1}H-NMR (500 MHz, methanol-d_4) δ: 0.79, 0.80, 1.04, 1.22, 1.30, 136 (each 3H, s, CH_3), 0.94 (3H, d, J = 6.6, CH_3), 5.30 (1H, s, H-12), 5.32 (1H, d, J= 8.2Hz, H-1′); ^{13}C-NMR (125 MHz, methanol-d_4) δ: 13.0(q, C-30) , 16.6(q, C-25), 17.9(q, C-26) ,19.5 (t, C-6) , 22.4 (q, C-24), 24.8 (t, C-11) , 24.8 (q, C-27) , 26.5 (t, C-16) ,27.1 (q, C-29), 27.2 (t, C-21), 29.0 (q,C-23) , 29.7 (t, C-15) , 34.2 (t, C-7), 38.3 (t,C-22), 39.0 (s,C-10), 39.4 (s,C-4), 41.4 (s, C-8), 42.0(t, C-1), 42.6 (s, C-14), 42.9 (d,C-20), 48.2 (d, C-9), 49.3 (d, C-5), 49.5 (s, C-17), 54.9 (d, C-18), 62.5 (t, C-6′), 67.2 (d, C-2), 71.1(d, C-4′), 73.6 (s, C-19), 73.8 (d, C-2′), 78.3 (d, C-5′), 78.5 (d, C-3′), 80.7 (d, C-3), 95.8 (d, C-l′), 130.9 (d, C-12), 138.4 (s, C-13), 178.7 (s, C-28)。以上数据与左国营等(2008)报道基本一致,故鉴定 **6** 为 kaji-ichigoside F1。

化合物 **7** 白色晶体,分子式 $C_{36}H_{56}O_{11}$,ESI-MS m/z: 663 [M - H]$^-$, 687 [M + Na]$^+$。^{1}H-NMR (500MHz, methanol-d_4) δ: 0.59, 0.80, 0.93, 1.22, 1.41(each 3H, s, CH_3), 0.96(3H, d, J=7.6Hz), 2.18 (1H, d, J=12.0Hz, αH-1), 2.37 (1H, d, J= 12.0Hz, βH-1), 2.55 (1H, s, H-18), 2.64 (1H, t, H-18), 3.68 (1H, dd, J = 12.1, 4.5Hz, Ha-6′), 3.81 (1H, dd, J=12.1, 2.0Hz, Hb-6′), 4.40 (1H, s, H-3), 5.31 (1H, s, H-12), 5.33 (1H, d, J= 8.2Hz, H-1′); ^{13}C-NMR (125MHz, methanol-d_4) δ: 13.5 (q, C-24), 16.6 (q, C-30), 17.2 (q, C-26), 17.2 (q, C-25), 19.5 (t, C-6), 24.6 (q, C-27), 24.7 (t, C-11), 26.5 (t, C-21), 27.1 (q, C-29), 27.2 (t, C-16), 29.7 (t, C-15), 33.3 (t, C-7), 38.2 (s, C-10), 38.2 (t, C-22), 41.6 (s, C-8), 42.8 (d, C-20), 42.8 (s, C-14), 44.4 (s, C-4), 47.2 (d, C-9), 48.2 (d, C-5), 49.5 (s, C-17), 54.3 (t, C-1), 54.9 (d, C-18), 62.5 (t, C-6′), 65.4 (t, C-23), 71.1 (d, C-4′), 73.6 (s, C-19), 73.9 (d, C-2′), 77.9 (d, C-3′), 78.3 (d,C-5′), 78.5 (d, C-3), 95.8 (d, C-1′), 129.0 (d, C-12), 140.0 (s, C-13), 178.5 (s, C-28), 213.9 (s, C-2)。以上数据与 Germain et al(2009)报道基本一致,故鉴定 **7** 为 β-D-glucopyranosyl 3β, 19α-dihydroxy-2-oxo-urs-12-en-28-oate。

化合物 **8** 白色晶体,分子式 $C_{35}H_{60}O_6$,在 5%硫酸乙醇溶液中显紫红色,与胡萝卜苷标准品 TLC 检测 Rf 值一致,且混合后熔点不降低。^{13}C-NMR (125 MHz, $CDCl_3$) δ: 12.1 (C-18), 13.0 (C-29), 17.0 (C-26), 18.3 (C-21), 19.0 (C-27), 19.2 (C-19), 19.8 (C-11), 22.0 (C-28), 22.1 (C-15), 25.7(C-10), 27.1 (C-12), 29.0 (C-25), 29.5 (C-2), 30.4 (C-8), 31.7 (C-7), 34.4 (C-22), 34.7 (C-20), 37.1 (C-23), 37.8 (C-1), 40.6 (C-4), 41.4 (C-16), 42.1 (C-13), 45.8 (C-24), 48.2 (C-9), 55.8 (C-17), 56.0 (C-14), 61.2 (C-6′), 69.3 (C-4′), 73.4 (C-2′), 74.4 (C-5′), 75.8 (C-3′), 76.8 (C-3), 104.0 (C-1′), 122.1 (C-6), 146.4 (C-5)。以上数据与黄建猷等(2015)报道基本一致,故鉴定 **8** 为胡萝卜苷。

化合物 **9** 白色针晶,分子式 $C_{29}H_{50}O$,在 5%硫酸乙醇溶液中显紫红色,与 β-谷甾醇标准品 TLC 检测 Rf 值一致,且混合后熔点不降低。^{13}C-NMR (125 MHz, $CDCl_3$) δ: 12.0 (C-18), 12.1 (C-29), 18.8 (C-19), 19.1 (C-26), 19.2 (C-21), 19.8 (C-27), 21.1 (C-11), 23.1 (C-28), 24.3 (C-15), 26.1 (C-23), 28.2 (C-16), 29.1 (C-27), 29.2 (C-1), 29.3

(C-25), 31.7 (C-2), 31.9 (C-8), 34.1 (C-22), 36.4 (C-10), 36.5 (C-20), 38.3 (C-12), 39.8 (C-4), 42.3 (C-11), 45.8 (C-24), 50.1 (C-9), 56.1 (C-17), 56.8 (C-14), 71.8 (C-3), 121.7 (C-6), 140.8 (C-5)。以上数据与张洪财等(2016)报道基本一致,故鉴定 **9** 为β-谷甾醇。

3　结论

本研究从金樱根乙醇浸膏的乙酸乙酯萃取部位分离得到 9 个化合物,其中化合物 **2**、**4**、**5**、**7** 为首次从该植物中分离得到,这些分离得到的化合物多为儿茶素类化合物和五环三萜类化合物,且多以同分异构体的形式存在。儿茶素类化合物大多具有抗氧化、抗菌等活性,三萜类化合物大多具有抗肿瘤、抗菌、抗病毒、抗炎等活性。因此我们将进一步对所分离的得到的化合物进行生物活性的研究,从而为该药用植物的充分利用提供科学依据。

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