

[成分分析]

洋金花叶化学成分及其抗炎活性研究

刘 艳¹, 荣晓惠¹, 谭金燕², 潘 娟¹, 管 伟¹, 匡海学¹, 杨炳友^{1*}

(1. 黑龙江中医药大学, 教育部北药基础与应用研究重点实验室, 黑龙江 哈尔滨 150040; 2. 山西中医药大学, 山西 晋中 030619)

摘要: 目的 研究洋金花 *Datura metel* L. 叶化学成分及其抗炎活性。方法 采用大孔吸附树脂、硅胶、ODS、HPLC 法对洋金花叶 70% 乙醇提取物进行分离纯化, 根据理化性质及波谱数据鉴定所得化合物的结构, 采用 RAW264.7 模型评价其体外抗炎活性。结果 从中分离得到 28 个化合物, 分别鉴定为 7, 27-dihydroxy-1-oxowitha-2, 5, 24-trienolide (1)、acnistoferin (2)、baimantuoluoline K (3)、daturafoliside G (4)、daturafoliside H (5)、daturafoliside I (6)、daturafoliside R (7)、daturafoliside S (8)、daturametelin A (9)、daturametelin I (10)、daturametelin J (11)、daturataturin A (12)、baimantuoluoside H (13)、(22R)-27-hydroxy-7 α -methoxy-1-oxowitha-3, 5, 24-trienolide (14)、柯里拉京 (15)、desmethylagrimonolide 6-O- β -D-glucopyranoside (16)、仙鹤草内酯-6-O- β -D-吡喃葡萄糖苷 (17)、丁香脂素 (18)、丁香脂素-4-O- β -D-吡喃葡萄糖苷 (19)、1, 6-di-O-coumaroyl glucopyranoside (20)、滨蒿内酯 (21)、东莨菪内酯 (22)、野蔷薇亭 (23)、(-)-loliolide (24)、(+)-isololiolide (25)、邻苯二甲酸二丁酯 (26)、对羟基苯甲醛 (27)、没食子酸乙酯 (28)。化合物 9、20 能抑制 LPS 诱导细胞释放 NO, IC₅₀ 分别为 25.14、16.26 μ mol/L。结论 化合物 15~17、20、23、28 为首次从茄科植物中分离得到, 化合物 2、19、24、27 首次从曼陀罗属植物中分离得到。化合物 9、20 具有较强的抗炎活性。

关键词: 洋金花; 叶; 化学成分; 分离鉴定; 抗炎活性

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Chemical constituents from the leaves of *Datura metel* and their anti-inflammatory activities

LIU Yan¹, RONG Xiao-hui¹, TAN Jin-yan², PAN Juan¹, GUAN Wei¹, KUANG Hai-xue¹, YANG Bing-you^{1*}

(1. Ministry of Education Key Laboratory for Basic and Applied Research of Northern Medicines, Heilongjiang University of Chinese Medicine, Harbin 150040, China; 2. Shanxi University of Chinese Medicine, Jinzhong 030619, China)

ABSTRACT: **AIM** To study the chemical constituents from the leaves of *Datura metel* L. and their anti-inflammatory activities. **METHODS** The 70% ethanol extract of the leaves of *D. metel* was isolated and purified by macroporous resin, silica gel, ODS and HPLC, then the structures of obtained compounds were identified by physicochemical properties and spectral data. Their anti-inflammatory activities *in vitro* were evaluated by RAW264.7 model. **RESULT** Twenty eight compounds were isolated and identified as 7, 27-dihydroxy-1-oxowitha-2, 5, 24-trienolide (1), acnistoferin (2), baimantuoluoline K (3), daturafoliside G (4), daturafoliside H (5), daturafoliside I (6), daturafoliside R (7), daturafoliside S (8), daturametelin A (9), daturametelin I (10), daturametelin J (11), daturataturin A (12), baimantuoluoside H (13), (22R)-27-hydroxy-7 α -methoxy-1-oxowitha-3, 5, 24-trienolide (14), corilagin (15), desmethylagrimonolide 6-O- β -D-

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作者简介: 刘 艳(1987—), 女, 副教授, 研究方向为中药及复方的药效物质基础。Tel: (0451) 87266871, E-mail: lifeliuyan@163.com

* **通信作者:** 杨炳友(1970—), 博士, 教授, 研究方向为中药及复方的药效物质基础。Tel: (0451) 82193007, E-mail: ybywater@163.com

glucopyranoside (16), agrimonolide-6-*O*- β -*D*-glucopyranoside (17), syringaresinol (18), syringaresinol-4-*O*- β -*D*-glucopyranoside (19), 1, 6-di-*O*-coumaroyl-glucopyranoside (20), scoparone (21), scopoletin (22), rosamultin (23), (–)-loliolide (24), (+)-isololiolide (25), dibutyl phthalate (26), *p*-hydroxybenzaldehyde (27), ethyl gallate (28). Compounds 9, 20 effectively inhibited the production of NO by LPS-induced cells with the IC₅₀ value of 25.14, 16.26 μ mol/L, respectively. **CONCLUSION** Compounds 15-17, 20, 23, 28 are first isolated from family Solanaceae, compounds 2, 19, 24, 27 are isolated from genus *Datura* for the first time. Compounds 9 and 20 show strong anti-inflammatory activities.

KEY WORDS: *Datura metel* L.; leaves; chemical constituents; isolation and identification; anti-inflammatory activity

洋金花 *Datura metel* L. 为茄科曼陀罗属植物, 始载于《本草纲目》, 其花入药, 具有定喘, 祛风, 麻醉止痛的功效^[1], 药用历史悠久, 临床应用广泛, 但其生长周期长、产量低造成资源紧张。洋金花叶具有抗炎^[2]、抗菌、抗氧化^[3]、抑制免疫^[4]等多种生物活性, 并含可治疗银屑病的醉茄内酯类成分, 其含量约为洋金花中的 3.5 倍^[5]。为了充分利用洋金花植物资源, 本实验对洋金花叶化学成分及抗炎活性进行研究, 共分离鉴定出 28 个化合物, 其中化合物 15~17、20、23、28 为首次从该茄科植物中分离得到, 2、19、24、27 首次从曼陀罗属植物中分离得到, 9、20 表现出抗炎活性。

1 材料

Waters 2695-2998-2414 分析高效液相色谱仪 (美国 Waters 公司); CBM-20A 半制备 HPLC 色谱仪 (日本岛津公司); Bruker-400/600 超导核磁共振光谱仪 (德国 Bruker 公司); UHPLC-Orbitrap-MS 质谱系统 (美国 Thermo Fisher Scientific 公司); 旋转蒸发器 (日本东京理化器械株式会社); Sepacore 型中压液相色谱仪 (瑞士 Buchi 公司)。GF254 型薄层硅胶 (烟台江友硅胶开发有限公司); 柱层色谱用硅胶 (80~100、200~300 目, 青岛海洋化工厂); 柱用 ODS (12 nm, s-50 μ m, 日本 YMC 公司)。RAW264.7 细胞系 (武汉大学细胞保藏中心); FBS 胎牛血清 (以色列 BI 公司); LPS (美国 Sigma 公司)。洋金花叶于 2017 年 9 月采收于哈尔滨双城区, 经黑龙江中医药大学药学院药用植物教研室樊锐锋教授鉴定为茄科曼陀罗属植物白花曼陀罗 *Datura metel* L. 的叶, 标本 (20170901) 保存于黑龙江中医药大学中药化学教研室。色谱层析用分析纯试剂 (天津试剂一厂); 色谱纯甲醇 (德国 Merck 公司)。

2 提取与分离

将 20 kg 干燥洋金花叶以 8 倍量 70% 乙醇回流

提取 3 次, 每次 2 h, 滤过, 合并提取液, 减压浓缩得 70% 乙醇总提物 4.82 kg, 经 HP-20 大孔吸附树脂, 分别用水、30% 乙醇、95% 乙醇洗脱后减压浓缩, 分别得水洗脱组分 930 g、30% 乙醇洗脱组分 1 195 g、95% 乙醇洗脱组分 1 894 g。

取 30% 乙醇洗脱组分 400 g, 经硅胶柱 [二氯甲烷-甲醇 (1:0~0:1)] 梯度洗脱, 洗脱液浓缩后经 TLC 薄层检识, 得到 Fr. A~Fr. I 流分。其中, Fr. B 经硅胶柱、半制备型 HPLC (甲醇-水, 58:42, 3 mL/min) 得化合物 15 (21.8 mg, t_R = 43.1 min); Fr. D 经硅胶柱、ODS 柱、半制备型 HPLC (甲醇-水, 60:40, 3 mL/min) 得化合物 16 (3.1 mg, t_R = 18.8 min)、23 (2.1 mg, t_R = 19.3 min); Fr. F 经硅胶柱、半制备型 HPLC (甲醇-水, 40:60, 3 mL/min) 得化合物 25 (2.6 mg, t_R = 15.4 min)、27 (3.3 mg, t_R = 14.8 min)、28 (4.3 mg, t_R = 15.2 min); Fr. G 经硅胶柱、ODS 柱、半制备型 HPLC (甲醇-水, 35:65, 3 mL/min) 得化合物 24 (2.4 mg, t_R = 16.4 min); Fr. H 经硅胶柱、半制备型 HPLC (甲醇-水, 50:50, 3 mL/min) 得化合物 22 (1.9 mg, t_R = 15.6 min)。

取 95% 乙醇洗脱组分 400 g, 经硅胶柱 [二氯甲烷-甲醇 (100:1~0:1)] 梯度洗脱, 洗脱液浓缩后经 TLC 薄层检识, 得到 Fr. A~Fr. F 流分。其中, Fr. B 经硅胶柱、半制备型 HPLC (甲醇-水, 60:40, 3 mL/min) 得化合物 1 (20.2 mg, t_R = 45.2 min)、2 (2.5 mg, t_R = 40.8 min); Fr. D 经硅胶柱、ODS 柱、半制备型 HPLC (甲醇-水, 65:35, 3 mL/min) 得化合物 3 (30.1 mg, t_R = 45.9 min)、8 (2.5 mg, t_R = 40.5 min)、10 (12.9 mg, t_R = 47.2 min)、11 (14.8 mg, t_R = 41.9 min)、12 (22.6 mg, t_R = 43.6 min)、14 (16.8 mg, t_R = 49.6 min)、18 (2.8 mg, t_R = 38.3 min)、20 (2.2 mg, t_R = 36.2 min)、21 (15.0 mg, t_R = 34.6 min);

Fr. E 经硅胶柱、ODS 柱、半制备型 HPLC (甲醇-水, 60 : 40, 3 mL/min) 得化合物 **4** (7.0 mg, t_R = 44.4 min)、**6** (4.1 mg, t_R = 45.9 min)、**7** (2.3 mg, t_R = 48.0 min)、**13** (7.8 mg, t_R = 49.9 min)、**17** (2.1 mg, t_R = 42.3 min)、**19** (1.0 mg, t_R = 38.1 min); Fr. F 经硅胶柱、半制备型 HPLC (甲醇-水, 65 : 35, 3 mL/min) 得化合物 **5** (4.1 mg, t_R = 44.3 min)、**9** (3.2 mg, t_R = 49.4 min)、**26** (0.7 mg, t_R = 50.4 min)。

3 结构鉴定

化合物 **1**: 白色无定形粉末 (甲醇), 分子式 $C_{28}H_{38}O_5$; HR-ESI-MS m/z 477.260 6 $[M+Na]^+$ 。 1H -NMR (CD_3OD , 400 MHz) δ : 6.92 (1H, ddd, J = 10.4, 5.2, 2.8 Hz, H-3), 5.85 (1H, m, H-2), 5.79 (1H, dd, J = 6.0, 2.0 Hz, H-6), 4.47 (1H, dt, J = 13.2, 3.2 Hz, H-22), 4.38 (1H, d, J = 11.6 Hz, H-27a), 4.31 (1H, d, J = 11.6 Hz, H-27b), 3.79 (1H, t, J = 4.4 Hz, H-7), 3.41 (1H, br. d, J = 21.2 Hz, H-4a), 2.94 (1H, dd, J = 21.2, 5.2 Hz, H-4b), 2.56 (1H, dd, J = 18.0, 13.2 Hz, H-23a), 2.23 (1H, m, H-11a), 2.20 (1H, dd, J = 18.0, 3.2 Hz, H-23b), 2.10 (3H, s, H-28), 1.99 (3H, m, H-9, 12a, 20), 1.81 (2H, m, H-15a, 16a), 1.60 (1H, m, H-11b), 1.45 (1H, m, H-8), 1.40 (1H, m, H-16b), 1.35 (1H, m, H-12b), 1.30 (2H, m, H-14, 17), 1.25 (3H, s, H-19), 1.19 (1H, m, H-15b), 1.06 (3H, d, J = 6.8 Hz, H-21), 0.79 (3H, s, H-18); ^{13}C -NMR (CD_3OD , 100 MHz) δ : 205.6 (C-1), 168.6 (C-26), 157.9 (C-24), 147.6 (C-3), 141.6 (C-5), 128.4 (C-2), 127.5 (C-6), 126.4 (C-25), 80.2 (C-22), 64.8 (C-7), 56.4 (C-27), 53.2 (C-17), 52.3 (C-10), 51.0 (C-14), 43.5 (C-13), 40.8 (C-12), 40.5 (C-20), 39.5 (C-8), 36.4 (C-9), 34.4 (C-4), 30.7 (C-23), 28.2 (C-16), 25.0 (C-15), 24.6 (C-11), 20.2 (C-28), 18.7 (C-19), 13.7 (C-21), 12.1 (C-18)。与文献 [6] 报道一致, 故鉴定为 **7, 27-dihydroxy-1-oxowitha-2, 5, 24-trienolide**。

化合物 **2**: 白色无定形粉末 (甲醇), 分子式 $C_{28}H_{40}O_6$; HR-ESI-MS m/z 495.271 5 $[M+Na]^+$ 。 1H -NMR (CD_3OD , 400 MHz) δ : 6.65 (1H, ddd, J = 10.4, 5.2, 2.4 Hz, H-3), 5.77 (1H, dd, J = 10.4, 2.4 Hz, H-2), 4.46 (1H, dt, J = 13.2, 3.2 Hz, H-

22), 4.38 (1H, d, J = 11.6 Hz, H-27a), 4.31 (1H, d, J = 11.6 Hz, H-27b), 3.52 (1H, t, J = 2.8 Hz, H-6), 3.26 (1H, dt, J = 20.0, 2.8 Hz, H-4a), 2.55 (1H, dd, J = 17.6, 13.6 Hz, H-23a), 2.20 (1H, dd, J = 17.6, 2.8 Hz, H-23b), 2.20 (1H, overlap, H-11a), 2.10 (3H, s, H-28), 2.04 (1H, dd, J = 20.0, 5.0 Hz, H-4b), 1.97 (2H, m, H-12a, 20), 1.78 (3H, m, H-9, 15a, 16a), 1.67 (2H, m, H-7a, 8), 1.54 (1H, m, H-7b), 1.40 (1H, m, H-15b), 1.38 (1H, m, H-16b), 1.36 (2H, m, H-11b, 12b), 1.30 (3H, s, H-19), 1.27 (1H, m, H-17), 1.20 (1H, m, H-14), 1.04 (3H, d, J = 6.8 Hz, H-21), 0.80 (3H, s, H-18); ^{13}C -NMR (CD_3OD , 100 MHz) δ : 207.6 (C-1), 168.6 (C-26), 157.9 (C-24), 143.8 (C-3), 129.0 (C-2), 126.4 (C-25), 80.2 (C-22), 78.3 (C-5), 75.3 (C-6), 57.1 (C-14), 56.4 (C-27), 53.4 (C-17), 53.0 (C-10), 44.2 (C-13), 42.6 (C-9), 41.5 (C-12), 40.5 (C-20), 36.6 (C-4), 34.1 (C-7), 31.4 (C-8), 30.7 (C-23), 28.2 (C-16), 25.4 (C-15), 24.5 (C-11), 20.2 (C-28), 16.2 (C-19), 13.7 (C-21), 12.7 (C-18)。与文献 [7] 报道一致, 故鉴定为 **acnistoferin**。

化合物 **3**: 白色无定形粉末 (甲醇), 分子式 $C_{28}H_{42}O_6$; HR-ESI-MS m/z 497.287 1 $[M+Na]^+$ 。 1H -NMR (CD_3OD , 400 MHz) δ : 5.71 (1H, d, J = 5.3 Hz, H-6), 4.47 (1H, dt, J = 13.2, 3.4 Hz, H-22), 4.38 (1H, d, J = 11.7 Hz, H-27a), 4.31 (1H, d, J = 11.7 Hz, H-27b), 3.91 (1H, m, H-3), 3.82 (1H, s, H-1), 3.72 (1H, t, J = 3.8 Hz, H-7), 2.54 (1H, dd, J = 17.9, 13.4 Hz, H-23a), 2.33 (2H, m, H-4), 2.20 (1H, dd, J = 17.9, 3.4 Hz, H-23b), 2.10 (3H, s, H-28), 2.00 (3H, m, H-2a, 12a, 20), 1.90 (1H, m, H-8), 1.82 (2H, m, H-15a, 16a), 1.72 (1H, m, H-2b), 1.57 (2H, m, H-11a, 14), 1.53 (1H, m, H-11b), 1.45 (1H, m, H-9), 1.41 (1H, m, H-16b), 1.25 (2H, m, H-12b, 15b), 1.19 (1H, m, H-17), 1.04 (3H, d, J = 6.6 Hz, H-21), 1.01 (3H, s, H-19), 0.78 (3H, s, H-18); ^{13}C -NMR (CD_3OD , 100 MHz) δ : 168.6 (C-26), 157.9 (C-24), 144.3 (C-5), 127.3 (C-6), 126.4 (C-25), 80.2 (C-22), 73.5 (C-1), 66.7 (C-3), 65.9 (C-7), 56.4 (C-27), 53.2 (C-17), 50.7 (C-14), 43.7 (C-13), 43.3 (C-10),

42.4 (C-4), 40.5 (C-12), 40.4 (C-20), 39.1 (C-2), 38.9 (C-9), 35.1 (C-8), 30.7 (C-23), 28.3 (C-16), 25.1 (C-15), 21.0 (C-11), 20.2 (C-28), 18.8 (C-19), 13.8 (C-21), 12.0 (C-18)。与文献[4]报道一致,故鉴定为 baimantuoluoline K。

化合物 4: 白色无定形粉末 (甲醇), Molish 反应呈阳性, 分子式 $C_{35}H_{52}O_{11}$; HR-ESI-MS m/z 671.340 0 $[M+Na]^+$ 。 1H -NMR (CD_3OD , 600 MHz) δ : 5.72 (1H, d, $J=4.3$ Hz, H-6), 4.62 (1H, d, $J=11.2$ Hz, H-27a), 4.49 (1H, dt, $J=13.4, 3.4$ Hz, H-22), 4.46 (1H, d, $J=11.2$ Hz, H-27b), 4.32 (1H, d, $J=7.8$ Hz, H-1'), 3.85 (2H, m, H-3, 6'a), 3.72 (1H, m, H-7), 3.67 (1H, dd, $J=11.9, 5.5$ Hz, H-6'b), 3.32 (1H, m, H-3'), 3.27 (1H, m, H-4'), 3.28 (3H, s, 3-OCH₃), 3.24 (1H, m, H-5'), 3.16 (1H, t, $J=8.1$ Hz, H-2'), 2.99 (1H, dd, $J=14.0, 3.5$ Hz, H-2a), 2.87 (1H, brd, $J=14.9$ Hz, H-4a), 2.57 (1H, dd, $J=18.0, 13.6$ Hz, H-23a), 2.51 (2H, m, H-2b, 4b), 2.22 (1H, dd, $J=18.0, 3.2$ Hz, H-23b), 2.13 (3H, s, H-28), 2.00 (1H, m, H-9), 1.98 (2H, m, H-12a, 20), 1.84 (3H, m, H-11a, 15a, 16a), 1.55 (1H, m, H-14), 1.45 (1H, m, H-11b), 1.41 (1H, m, H-8), 1.38 (1H, m, H-16b), 1.30 (3H, s, H-19), 1.28 (2H, m, H-12b, 17), 1.19 (1H, m, H-15b), 1.04 (3H, d, $J=6.6$ Hz, H-21), 0.77 (3H, s, H-18); ^{13}C -NMR (CD_3OD , 150 MHz) δ : 212.9 (C-1), 168.6 (C-26), 160.4 (C-24), 142.9 (C-5), 128.4 (C-6), 123.6 (C-25), 104.0 (C-1'), 80.2 (C-22), 78.3 (C-3), 78.0 (C-3', 5'), 75.0 (C-2'), 71.6 (C-4'), 65.2 (C-7), 63.6 (C-27), 62.7 (C-6'), 56.2 (3-OCH₃), 55.4 (C-10), 53.2 (C-17), 50.8 (C-14), 43.7 (C-2, 13), 40.6 (C-12), 40.4 (C-20), 38.7 (C-8), 36.7 (C-4), 36.0 (C-9), 30.8 (C-23), 28.2 (C-16), 25.0 (C-15), 23.5 (C-11), 20.7 (C-28), 19.2 (C-19), 13.7 (C-21), 12.1 (C-18)。与文献[2]报道一致,故鉴定为 daturafoliside G。

化合物 5: 白色无定形粉末 (甲醇), Molish 反应呈阳性, 分子式 $C_{38}H_{58}O_{11}$; HR-ESI-MS m/z 713.387 1 $[M+Na]^+$ 。 1H -NMR (CD_3OD , 600 MHz) δ : 5.73 (1H, d, $J=5.6$ Hz, H-6), 4.63 (1H, d,

$J=11.2$ Hz, H-27a), 4.52 (1H, m, H-22), 4.47 (1H, d, $J=11.2$ Hz, H-27b), 4.33 (1H, d, $J=7.8$ Hz, H-1'), 3.94 (1H, t, $J=2.9$ Hz, H-3), 3.86 (1H, dd, $J=11.9, 1.7$ Hz, H-6'a), 3.74 (1H, t, $J=3.8$ Hz, H-7), 3.68 (1H, dd, $J=11.9, 5.4$ Hz, H-6'b), 3.43 (1H, m, H-1'a), 3.40 (1H, m, H-3'), 3.34 (1H, m, H-1'b), 3.28 (2H, m, H-4', 5'), 3.17 (1H, t, $J=7.8$ Hz, H-2'), 2.98 (1H, dd, $J=13.9, 3.7$ Hz, H-2a), 2.87 (1H, br. d, $J=14.8$ Hz, H-4a), 2.58 (1H, dd, $J=17.8, 13.8$ Hz, H-23a), 2.48 (2H, m, H-2b, 4b), 2.23 (1H, dd, $J=17.8, 2.9$ Hz, H-23b), 2.14 (3H, s, H-28), 2.03 (1H, m, H-9), 2.00 (1H, m, H-20), 1.98 (1H, m, H-12a), 1.83 (3H, m, H-11a, 15a, 16a), 1.56 (1H, m, H-14), 1.49 (1H, m, H-2'a), 1.46 (1H, m, H-11b), 1.45 (2H, m, H-8, 2'b), 1.40 (1H, m, H-16b), 1.36 (1H, m, H-3'a), 1.32 (2H, m, H-3'b, 12b), 1.31 (3H, s, H-19), 1.25 (1H, m, H-17), 1.20 (1H, m, H-15b), 1.05 (3H, d, $J=6.6$ Hz, H-21), 0.92 (3H, t, $J=7.4$ Hz, H-4''), 0.78 (3H, s, H-18); ^{13}C -NMR (CD_3OD , 150 MHz) δ : 213.2 (C-1), 168.6 (C-26), 160.4 (C-24), 143.0 (C-5), 128.3 (C-6), 123.6 (C-25), 104.0 (C-1'), 80.2 (C-22), 78.0 (C-3', 5'), 76.8 (C-3), 75.0 (C-2'), 71.6 (C-4'), 69.5 (C-1''), 65.3 (C-7), 63.6 (C-27), 62.7 (C-6'), 55.4 (C-10), 53.1 (C-17), 50.8 (C-14), 44.2 (C-2), 43.7 (C-13), 40.5 (C-12), 40.4 (C-20), 38.6 (C-8), 37.1 (C-4), 36.1 (C-9), 32.8 (C-2''), 30.8 (C-23), 28.2 (C-16), 25.0 (C-15), 23.4 (C-11), 20.7 (C-28), 20.3 (C-3''), 19.1 (C-19), 14.2 (C-4''), 13.7 (C-21), 12.1 (C-18)。与文献[2]报道一致,故鉴定为 daturafoliside H。

化合物 6: 白色无定形粉末 (甲醇), Molish 反应呈阳性, 分子式 $C_{34}H_{48}O_{10}$; HR-ESI-MS m/z 639.313 9 $[M+Na]^+$ 。 1H -NMR (CD_3OD , 600 MHz) δ : 6.08 (1H, dd, $J=9.7, 1.9$ Hz, H-4), 5.78 (1H, m, H-3), 5.52 (1H, d, $J=2.2$ Hz, H-6), 4.62 (1H, d, $J=11.2$ Hz, H-27a), 4.50 (1H, dt, $J=13.3, 3.4$ Hz, H-22), 4.46 (1H, d, $J=11.2$ Hz, H-27b), 4.32 (1H, d, $J=8.0$ Hz, H-1'), 3.85 (1H, dd, $J=11.9, 2.0$ Hz, H-6'a), 3.79 (1H, d, $J=8.3$ Hz, H-7), 3.67 (1H, dd, $J=$

11.9, 5.4 Hz, H-6'b), 3.41 (1H, m, H-3'), 3.26 (2H, m, H-4', 5'), 3.16 (1H, t, $J=8.0$ Hz, H-2'), 2.68 (1H, dd, $J=20.0, 4.7$ Hz, H-2a), 2.57 (1H, dd, $J=18.0, 13.3$ Hz, H-23a), 2.21 (1H, dd, $J=18.0, 3.4$ Hz, H-23b), 2.13 (3H, s, H-28), 1.98 (2H, m, H-12a, 20), 1.96 (1H, m, H-2b), 1.88 (3H, m, H-11, 15a), 1.81 (2H, m, H-9, 16a), 1.52 (2H, m, H-8, 11b), 1.42 (3H, s, H-19), 1.40 (2H, m, H-15b, 16b), 1.34 (1H, m, H-12b), 1.29 (1H, m, H-14), 1.24 (1H, m, H-17), 1.04 (3H, d, $J=6.6$ Hz, H-21), 0.80 (3H, s, H-18); $^{13}\text{C-NMR}$ (CD_3OD , 150 MHz) δ : 212.1 (C-1), 168.6 (C-26), 160.3 (C-24), 143.4 (C-5), 131.7 (C-6), 129.5 (C-4), 125.6 (C-3), 123.7 (C-25), 104.0 (C-1'), 80.2 (C-22), 78.0 (C-3', 5'), 75.0 (C-2'), 72.4 (C-7), 71.6 (C-4'), 63.6 (C-27), 62.7 (C-6'), 57.1 (C-14), 53.2 (C-10), 52.7 (C-17), 44.7 (C-13), 41.4 (C-8), 41.1 (C-9), 40.8 (C-2), 40.7 (C-12), 40.4 (C-20), 30.8 (C-23), 28.5 (C-16), 27.4 (C-15), 23.8 (C-11), 20.8 (C-28), 20.5 (C-19), 13.8 (C-21), 12.4 (C-18)。与文献 [2] 报道一致, 故鉴定为 daturafoliside I。

化合物 7: 白色无定形粉末 (甲醇), Molish 反应呈阳性, 分子式 $\text{C}_{35}\text{H}_{50}\text{O}_{10}$; HR-ESI-MS m/z 653.332 7 [$\text{M}+\text{Na}$] $^+$ 。 $^1\text{H-NMR}$ (CD_3OD , 600 MHz) δ : 6.08 (1H, dd, $J=9.9, 2.5$ Hz, H-7), 5.71 (1H, br. s, H-6), 5.69 (1H, br. s, H-4), 4.63 (1H, d, $J=11.3$ Hz, H-27a), 4.49 (1H, m, H-22), 4.46 (1H, d, $J=11.3$ Hz, H-27b), 4.32 (1H, d, $J=7.8$ Hz, H-1'), 4.31 (1H, m, H-3), 3.86 (1H, dd, $J=12.0, 2.1$ Hz, H-6'a), 3.67 (1H, dd, $J=12.0, 5.2$ Hz, H-6'b), 3.35 (3H, s, 3-OCH₃), 3.34 (1H, m, H-3'), 3.28 (1H, m, H-4'), 3.24 (1H, m, H-5'), 3.15 (1H, t, $J=7.8$ Hz, H-2'), 3.01 (1H, dd, $J=13.4, 5.8$ Hz, H-2a), 2.56 (1H, m, H-23a), 2.55 (1H, m, H-2b), 2.23 (1H, m, H-23b), 2.15 (1H, m, H-8), 2.12 (3H, s, H-28), 2.01 (1H, m, H-12a), 2.00 (1H, m, H-20), 1.83 (2H, m, H-15a, 16a), 1.67 (1H, m, H-11a), 1.52 (1H, m, H-9), 1.44 (1H, m, H-16b), 1.43 (1H, m, H-11b), 1.33 (1H, m, H-15b), 1.29 (1H, m, H-14), 1.28 (1H, m, H-17), 1.26 (1H, m, H-12b), 1.19 (3H, s, H-19),

1.02 (3H, d, $J=6.6$ Hz, H-21), 0.81 (3H, s, H-18); $^{13}\text{C-NMR}$ (CD_3OD , 150 MHz) δ : 214.1 (C-1), 168.6 (C-26), 160.3 (C-24), 144.9 (C-5), 132.7 (C-6), 128.3 (C-7), 125.8 (C-4), 123.6 (C-25), 104.0 (C-1'), 80.0 (C-22), 78.0 (C-3', 5'), 77.1 (C-3), 75.0 (C-2'), 71.6 (C-4'), 63.6 (C-27), 62.7 (C-6'), 56.2 (3-OMe), 55.1 (C-14), 53.0 (C-17), 50.4 (C-10), 47.0 (C-9), 44.9 (C-13), 44.7 (C-2), 40.8 (C-12), 40.4 (C-20), 38.4 (C-8), 30.8 (C-23), 28.2 (C-16), 25.1 (C-15), 23.8 (C-11), 20.7 (C-28), 18.1 (C-19), 13.6 (C-21), 12.3 (C-18)。与文献 [5] 报道一致, 故鉴定为 daturafoliside R。

化合物 8: 白色无定形粉末 (甲醇), 分子式 $\text{C}_{28}\text{H}_{38}\text{O}_6$; HR-ESI-MS m/z 493.255 8 [$\text{M}+\text{Na}$] $^+$ 。 $^1\text{H-NMR}$ (CD_3OD , 600 MHz) δ : 7.07 (1H, dd, $J=9.6, 6.0$ Hz, H-3), 6.23 (1H, $J=6.0$ Hz, H-4), 5.96 (1H, d, $J=9.6$ Hz, H-2), 4.45 (1H, dt, $J=13.4, 3.4$ Hz, H-22), 4.37 (1H, d, $J=11.8$ Hz, H-27a), 4.30 (1H, d, $J=11.8$ Hz, H-27b), 4.22 (1H, d, $J=3.2$ Hz, H-6), 3.73 (1H, t, $J=2.8$ Hz, H-7), 2.52 (1H, dd, $J=18.0, 13.4$ Hz, H-23a), 2.16 (1H, dd, $J=18.0, 3.4$ Hz, H-23b), 2.08 (3H, s, H-28), 2.06 (1H, m, H-8), 2.00 (1H, m, H-12a), 1.96 (2H, m, H-11a, 20), 1.79 (2H, m, H-15a, 16a), 1.57 (3H, m, H-9, 11b, 14), 1.43 (3H, s, H-19), 1.43 (1H, overlap, H-16b), 1.28 (1H, m, H-15b), 1.26 (1H, m, H-17), 1.14 (1H, m, H-12b), 1.03 (3H, d, $J=6.7$ Hz, H-21), 0.84 (3H, s, H-18); $^{13}\text{C-NMR}$ (CD_3OD , 150 MHz) δ : 208.7 (C-1), 168.5 (C-26), 157.8 (C-24), 157.4 (C-5), 142.4 (C-3), 126.7 (C-2), 126.4 (C-25), 122.1 (C-4), 80.1 (C-22), 79.2 (C-6), 73.7 (C-7), 56.4 (C-27), 55.2 (C-10), 53.1 (C-17), 51.3 (C-14), 43.8 (C-13), 42.6 (C-9), 40.6 (C-12), 40.4 (C-20), 36.4 (C-8), 30.7 (C-23), 28.2 (C-16), 24.8 (C-15), 22.5 (C-11), 20.2 (C-28), 19.9 (C-19), 13.6 (C-21), 12.1 (C-18)。与文献 [5] 报道一致, 故鉴定为 daturafoliside S。

化合物 9: 白色无定形粉末 (甲醇), Molish 反应呈阳性, 分子式 $\text{C}_{34}\text{H}_{48}\text{O}_9$; HR-ESI-MS m/z 623.319 2 [$\text{M}+\text{Na}$] $^+$ 。 $^1\text{H-NMR}$ (CD_3OD , 600 MHz) δ : 6.91 (1H, ddd, $J=10.0, 4.9, 2.4$ Hz, H-3),

5.83 (1H, dd, $J=10.0, 2.4$ Hz, H-2), 5.62 (1H, d, $J=5.8$ Hz, H-6), 4.63 (1H, d, $J=11.2$ Hz, H-27a), 4.49 (1H, m, H-22), 4.47 (1H, d, $J=11.2$ Hz, H-27b), 4.33 (1H, d, $J=7.8$ Hz, H-1'), 3.86 (1H, dd, $J=12.0, 2.0$ Hz, H-6'a), 3.68 (1H, dd, $J=12.0, 5.4$ Hz, H-6'b), 3.35 (2H, m, H-4a, 3'), 3.27 (2H, m, H-4', 5'), 3.17 (1H, t, $J=8.1$ Hz, H-2'), 2.88 (1H, dd, $J=21.4, 4.9$ Hz, H-4b), 2.58 (1H, dd, $J=18.1, 13.6$ Hz, H-23a), 2.23 (1H, dd, $J=18.1, 3.1$ Hz, H-23b), 2.18 (1H, m, H-11a), 2.14 (3H, s, H-28), 2.03 (1H, m, H-12a), 2.02 (1H, m, H-7a), 1.97 (1H, m, H-20), 1.80 (2H, m, H-9, 16a), 1.68 (1H, m, H-15a), 1.59 (1H, m, H-11b), 1.58 (1H, m, H-7b), 1.47 (1H, m, H-8), 1.38 (2H, m, H-12b, 16b), 1.28 (1H, m, H-14), 1.26 (3H, s, H-19), 1.17 (2H, m, H-15b, 17), 1.05 (3H, d, $J=6.6$ Hz, H-21), 0.80 (3H, s, H-18); $^{13}\text{C-NMR}$ (CD_3OD , 150 MHz) δ : 206.7 (C-1), 168.6 (C-26), 160.3 (C-24), 148.1 (C-3), 137.4 (C-5), 128.3 (C-2), 125.7 (C-6), 123.6 (C-25), 104.0 (C-1'), 80.1 (C-22), 78.0 (C-3', 5'), 75.0 (C-2'), 71.6 (C-4'), 63.6 (C-27), 62.8 (C-6'), 57.6 (C-14), 53.3 (C-17), 51.8 (C-10), 44.5 (C-9), 43.8 (C-13), 41.0 (C-12), 40.4 (C-20), 34.5 (C-4), 34.4 (C-8), 31.9 (C-7), 30.8 (C-23), 28.2 (C-16), 25.4 (C-15), 24.9 (C-11), 20.8 (C-28), 19.5 (C-19), 13.8 (C-21), 12.4 (C-18)。与文献 [8] 报道一致, 故鉴定为 daturametelin A。

化合物 10: 白色无定形粉末 (甲醇), Molish 反应呈阳性, 分子式 $\text{C}_{34}\text{H}_{48}\text{O}_{10}$; HR-ESI-MS m/z 639.314 1 $[\text{M}+\text{Na}]^+$ 。 $^1\text{H-NMR}$ (CD_3OD , 400 MHz) δ : 6.14 (1H, dd, $J=10.0, 1.9$ Hz, H-4), 5.83 (1H, m, H-3), 5.79 (1H, d, $J=5.5$ Hz, H-6), 4.63 (1H, d, $J=11.2$ Hz, H-27a), 4.50 (1H, m, H-22), 4.47 (1H, d, $J=11.2$ Hz, H-27b), 4.32 (1H, d, $J=7.8$ Hz, H-1'), 3.90 (1H, m, H-7), 3.85 (1H, dd, $J=11.9, 2.0$ Hz, H-6'a), 3.67 (1H, dd, $J=11.9, 5.2$ Hz, H-6'b), 3.40 (1H, m, H-5'), 3.28 (2H, m, H-3', 4'), 3.16 (1H, t, $J=7.8$ Hz, H-2'), 2.71 (1H, dd, $J=20.4, 4.3$ Hz, H-2), 2.58 (1H, dd, $J=18.0, 13.5$ Hz, H-23a), 2.23 (1H, dd, $J=18.0, 3.2$ Hz, H-23b), 2.14

(3H, s, H-28), 2.12 (1H, m, H-9), 1.99 (1H, m, H-20), 1.98 (1H, m, H-8), 1.95 (1H, m, H-12a), 1.85 (2H, m, H-15a, 16a), 1.81 (1H, m, H-11a), 1.60 (1H, m, H-16b), 1.57 (1H, m, H-11b), 1.40 (1H, m, H-14), 1.37 (3H, s, H-19), 1.33 (1H, m, H-15b), 1.31 (1H, m, H-12b), 1.22 (1H, m, H-17), 1.05 (3H, d, $J=6.6$ Hz, H-21), 0.80 (3H, s, H-18); $^{13}\text{C-NMR}$ (CD_3OD , 100 MHz) δ : 211.6 (C-1), 168.6 (C-26), 160.3 (C-24), 145.2 (C-5), 130.2 (C-4), 128.7 (C-6), 126.2 (C-3), 123.7 (C-25), 104.0 (C-1'), 80.2 (C-22), 78.0 (C-3', 5'), 75.0 (C-2'), 71.6 (C-4'), 65.0 (C-7), 63.6 (C-27), 62.8 (C-6'), 54.0 (C-10), 53.2 (C-17), 50.6 (C-14), 43.8 (C-13), 40.6 (C-12), 40.5 (C-2, 20), 38.4 (C-8), 35.0 (C-9), 30.8 (C-23), 28.2 (C-16), 25.0 (C-15), 23.3 (C-11), 20.8 (C-28), 20.0 (C-19), 13.8 (C-21), 12.2 (C-18)。与文献 [6] 报道一致, 故鉴定为 daturametelin I。

化合物 11: 白色无定形粉末 (甲醇), Molish 反应呈阳性。分子式 $\text{C}_{34}\text{H}_{48}\text{O}_{11}$; HR-ESI-MS m/z 655.308 8 $[\text{M}+\text{Na}]^+$ 。 $^1\text{H-NMR}$ (CD_3OD , 400 MHz) δ : 7.08 (1H, dd, $J=9.7, 5.8$ Hz, H-3), 6.23 (1H, d, $J=5.8$ Hz, H-4), 5.97 (1H, d, $J=9.7$ Hz, H-2), 4.62 (1H, d, $J=11.2$ Hz, H-27a), 4.48 (1H, m, H-22), 4.46 (1H, d, $J=11.2$ Hz, H-27b), 4.32 (1H, d, $J=7.8$ Hz, H-1'), 4.22 (1H, d, $J=3.3$ Hz, H-6), 3.85 (1H, dd, $J=12.0, 2.0$ Hz, H-6'a), 3.74 (1H, t, $J=2.8$ Hz, H-7), 3.67 (1H, m, H-6'b), 3.34 (1H, overlap, H-5'), 3.28 (1H, m, H-4'), 3.26 (1H, m, H-3'), 3.15 (1H, m, H-2'), 2.56 (1H, dd, $J=18.0, 13.4$ Hz, H-23a), 2.18 (1H, dd, $J=18.0, 3.2$ Hz, H-23b), 2.12 (3H, s, H-28), 2.10 (1H, m, H-8), 1.98 (3H, m, H-11a, 12a, 20), 1.80 (2H, m, H-15a, 16a), 1.58 (2H, m, H-9, 11b), 1.46 (1H, m, H-16b), 1.43 (3H, s, H-19), 1.42 (1H, m, H-14), 1.28 (2H, m, H-15b, 17), 1.15 (1H, m, H-12b), 1.03 (3H, d, $J=6.6$ Hz, H-21), 0.84 (3H, s, H-18); $^{13}\text{C-NMR}$ (CD_3OD , 100 MHz) δ : 208.7 (C-1), 168.6 (C-26), 160.2 (C-24), 157.4 (C-5), 142.4 (C-3), 126.7 (C-2), 123.7 (C-25), 122.0 (C-4), 104.0 (C-1'), 80.1 (C-22), 79.2 (C-6), 78.0 (C-3', 5'), 75.0 (C-

2'), 73.7 (C-7), 71.6 (C-4'), 63.6 (C-27), 62.8 (C-6'), 55.2 (C-10), 53.1 (C-17), 51.3 (C-14), 43.8 (C-13), 42.6 (C-9), 40.6 (C-12), 40.5 (C-20), 36.4 (C-8), 30.8 (C-23), 28.2 (C-16), 24.9 (C-15), 22.5 (C-11), 20.7 (C-28), 19.9 (C-19), 13.6 (C-21), 12.2 (C-18)。与文献 [6] 报道一致, 故鉴定为 daturametelin J。

化合物 **12**: 白色无定形粉末 (甲醇), Molish 反应呈阳性, 分子式 $C_{34}H_{48}O_{10}$; HR-ESI-MS m/z 639.313 8 $[M+Na]^+$ 。 1H -NMR (CD_3OD , 400 MHz) δ : 6.92 (1H, ddd, $J=10.0, 4.9, 2.4$ Hz, H-3), 5.85 (1H, dd, $J=10.0, 2.4$ Hz, H-2), 5.79 (1H, dd, $J=5.9, 1.7$ Hz, H-6), 4.63 (1H, d, $J=11.2$ Hz, H-27a), 4.50 (1H, m, H-22), 4.47 (1H, d, $J=11.2$ Hz, H-27b), 4.32 (1H, d, $J=7.8$ Hz, H-1'), 3.85 (1H, dd, $J=12.0, 2.2$ Hz, H-6'a), 3.79 (1H, t, $J=4.2$ Hz, H-7), 3.67 (1H, dd, $J=12.0, 5.2$ Hz, H-6'b), 3.41 (1H, m, H-4a), 3.34 (1H, m, H-3'), 3.28 (2H, m, H-4', 5'), 3.16 (1H, t, $J=8.1$ Hz, H-2'), 2.94 (1H, dd, $J=21.4, 4.9$ Hz, H-4b), 2.58 (1H, dd, $J=17.9, 13.5$ Hz, H-23a), 2.22 (1H, m, 23b), 2.14 (3H, s, H-28), 1.99 (4H, m, H-8, 9, 12a, 20), 1.82 (3H, m, H-11, 16a), 1.60 (1H, m, H-15a), 1.44 (1H, m, H-16b), 1.40 (1H, m, H-14), 1.35 (1H, m, H-15b), 1.30 (1H, m, H-12b), 1.25 (3H, s, H-19), 1.20 (1H, m, H-17), 1.06 (1H, d, $J=6.6$ Hz, H-21), 0.79 (3H, s, H-18); ^{13}C -NMR (CD_3OD , 100 MHz) δ : 205.6 (C-1), 168.6 (C-26), 160.3 (C-24), 147.6 (C-3), 141.6 (C-5), 128.4 (C-6), 127.5 (C-2), 123.6 (C-25), 104.0 (C-1'), 80.2 (C-22), 78.0 (C-3', 5'), 75.0 (C-2'), 71.6 (C-4'), 64.8 (C-7), 63.6 (C-27), 62.7 (C-6'), 53.2 (C-14), 52.2 (C-17), 51.0 (C-10), 43.5 (C-13), 40.8 (C-12), 40.5 (C-9), 39.5 (C-20), 36.4 (C-8), 34.4 (C-4), 30.8 (C-23), 28.2 (C-16), 25.0 (C-15), 24.6 (C-11), 20.7 (C-28), 18.7 (C-19), 13.7 (C-21), 12.1 (C-18)。与文献 [8] 报道一致, 故鉴定为 daturaturin A。

化合物 **13**: 白色无定形粉末 (甲醇), Molish 反应呈阳性, 分子式 $C_{34}H_{46}O_9$; HR-ESI-MS m/z 621.303 3 $[M+Na]^+$ 。 1H -NMR (CD_3OD , 400 MHz) δ : 7.11 (1H, dd, $J=10.0, 6.2$ Hz, H-3), 6.20

(1H, dd, $J=10.0, 2.6$ Hz, H-6), 6.03 (1H, d, $J=6.2$ Hz, H-4), 5.88 (2H, d, $J=10.0$ Hz, H-2, 7), 4.62 (1H, d, $J=11.2$ Hz, H-27a), 4.48 (1H, m, H-22), 4.46 (1H, overlap, H-27b), 4.32 (1H, d, $J=7.8$ Hz, H-1'), 3.85 (1H, dd, $J=12.0, 2.1$ Hz, H-6'a), 3.67 (1H, m, H-6'b), 3.33 (1H, m, H-3'), 3.26 (2H, m, H-4', 5'), 3.15 (1H, t, $J=8.4$ Hz, H-2'), 2.55 (1H, dd, $J=17.9, 13.2$ Hz, H-23a), 2.37 (1H, t, $J=10.0$ Hz, H-8), 2.18 (2H, m, H-15a, 23b), 2.12 (3H, s, H-28), 2.06 (1H, m, H-12a), 1.97 (1H, m, H-20), 1.83 (2H, m, H-11a, 16a), 1.69 (1H, m, H-15b), 1.56 (1H, m, H-9), 1.45 (1H, m, H-16b), 1.35 (1H, m, H-11b), 1.28 (2H, m, H-14, 17), 1.24 (3H, s, H-19), 1.19 (1H, m, H-12b), 1.03 (3H, d, $J=6.6$ Hz, H-21), 0.86 (3H, s, H-18); ^{13}C -NMR (CD_3OD , 100 MHz) δ : 207.2 (C-1), 168.6 (C-26), 160.3 (C-24), 157.5 (C-5), 143.2 (C-3), 136.2 (C-7), 128.5 (C-6), 125.8 (C-2), 123.6 (C-25), 118.4 (C-4), 104.0 (C-1'), 80.0 (C-22), 78.0 (C-3', 5'), 75.0 (C-2'), 71.5 (C-4'), 63.6 (C-27), 62.7 (C-6'), 55.1 (C-14), 53.0 (C-17), 52.3 (C-10), 48.8 (C-9), 45.0 (C-13), 41.0 (C-12), 40.4 (C-20), 39.3 (C-8), 30.7 (C-23), 28.1 (C-16), 25.0 (C-11), 23.7 (C-15), 20.8 (C-28), 20.5 (C-19), 13.6 (C-21), 12.2 (C-18)。与文献 [8] 报道一致, 故鉴定为 baimantuoluoside H。

化合物 **14**: 白色无定形粉末 (甲醇), Molish 反应呈阳性, 分子式 $C_{35}H_{50}O_{10}$; HR-ESI-MS m/z 653.329 4 $[M+Na]^+$ 。 1H -NMR (CD_3OD , 400 MHz) δ : 6.16 (1H, dd, $J=10.0, 1.6$ Hz, H-4), 6.02 (1H, d, $J=5.2$ Hz, H-6), 5.84 (1H, m, H-3), 4.62 (1H, d, $J=11.2$ Hz, H-27a), 4.49 (1H, m, H-22), 4.46 (1H, overlap, H-27b), 4.32 (1H, d, $J=7.8$ Hz, H-1'), 3.85 (1H, dd, $J=12.0, 2.0$ Hz, H-6'a), 3.67 (1H, dd, $J=12.0, 5.2$ Hz, H-6'b), 3.48 (1H, m, H-7), 3.44 (1H, m, H-2b), 3.35 (1H, m, H-3'), 3.30 (3H, overlap, 7-OCH₃), 3.26 (2H, m, H-4', 5'), 3.16 (1H, t, $J=8.4$ Hz, H-2'), 2.72 (1H, dd, $J=20.3, 5.0$ Hz, H-2a), 2.57 (1H, m, H-23a), 2.22 (1H, dd, $J=18.1, 3.2$ Hz, H-23b), 2.13 (3H, s, H-28), 2.10 (1H, m, H-9), 1.97 (2H, m, H-12a, 20),

1.81 (2H, m, H-11a, 16a), 1.74 (2H, m, H-11b, 15a), 1.61 (2H, m, H-8, 14), 1.42 (1H, m, H-16b), 1.36 (3H, s, H-19), 1.28 (3H, m, H-12b, 15b, 17), 1.04 (3H, d, $J=6.6$ Hz, H-21), 0.78 (3H, s, H-18); $^{13}\text{C-NMR}$ (CD_3OD , 100 MHz) δ : 211.5 (C-1), 168.6 (C-26), 160.4 (C-24), 146.6 (C-5), 130.2 (C-4), 126.4 (C-3), 125.6 (C-6), 123.6 (C-25), 104.0 (C-1'), 80.1 (C-22), 78.0 (C-3', 5'), 75.0 (C-2'), 74.0 (C-7), 71.6 (C-4'), 63.6 (C-27), 62.7 (C-6'), 56.8 (7-OCH₃), 54.1 (C-10), 53.2 (C-17), 50.4 (C-14), 43.8 (C-13), 40.5 (C-2, 20), 40.4 (C-12), 38.0 (C-8), 35.7 (C-9), 30.7 (C-23), 28.2 (C-16), 25.1 (C-15), 23.4 (C-11), 20.7 (C-28), 20.1 (C-19), 13.7 (C-21), 11.9 (C-18)。与文献 [4] 报道一致, 故鉴定为 (22*R*)-27-hydroxy-7 α -methoxy-1-oxowitha-3, 5, 24-trienolide。

化合物 15: 白色无定形粉末 (甲醇), Molish 反应呈阳性, 分子式 $\text{C}_{27}\text{H}_{22}\text{O}_{18}$; HR-ESI-MS m/z 657.068 5 $[\text{M}+\text{Na}]^+$ 。 $^1\text{H-NMR}$ (CD_3OD , 600 MHz) δ : 7.06 (2H, s, H-23, 27), 6.69 (1H, s, H-3), 6.66 (1H, s, H-12), 6.37 (1H, d, $J=1.6$ Hz, H-20), 4.96 (2H, m, H-16, 18), 4.52 (1H, m, H-15a), 4.47 (1H, m, H-17), 4.16 (1H, dd, $J=11.0, 8.4$ Hz, H-15b), 3.99 (1H, d, $J=1.0$ Hz, H-19); $^{13}\text{C-NMR}$ (CD_3OD , 150 MHz) δ : 170.1 (C-14), 168.5 (C-1), 166.7 (C-21), 146.4 (C-24, 26), 146.0 (C-11), 145.6 (C-9), 145.3 (C-6), 145.2 (C-4), 140.4 (C-25), 138.2 (C-5), 137.7 (C-10), 125.5 (C-13), 125.4 (C-2), 120.6 (C-22), 117.2 (C-7), 116.7 (C-8), 110.2 (C-3), 111.0 (C-23, 27), 108.3 (C-12), 95.0 (C-20), 76.2 (C-18), 71.6 (C-16), 69.5 (C-19), 65.0 (C-15), 62.5 (C-17)。与文献 [9] 报道一致, 故鉴定为柯里拉京。

化合物 16: 黄色无定形粉末 (甲醇), Molish 反应呈阳性, 分子式 $\text{C}_{23}\text{H}_{26}\text{O}_{10}$; HR-ESI-MS m/z 485.123 5 $[\text{M}+\text{Na}]^+$ 。 $^1\text{H-NMR}$ (CD_3OD , 400 MHz) δ : 7.04 (2H, d, $J=8.4$ Hz, H-2', 6'), 6.70 (2H, d, $J=8.4$ Hz, H-3', 5'), 6.52 (1H, d, $J=2.2$ Hz, H-7), 6.50 (1H, br. s, H-5), 4.98 (1H, d, $J=7.3$ Hz, H-1'''), 4.50 (1H, m, H-3), 3.89 (1H, dd, $J=12.2, 2.2$ Hz, H-6'''a), 3.68 (1H, dd, $J=12.2, 5.7$ Hz, H-6'''b), 3.31~3.50 (4H, m, H-

2''', 3''', 4''', 5'''), 2.95 (1H, m, H-4a), 2.89 (1H, m, H-4b), 2.78 (1H, m, H-2''a), 2.69 (1H, m, H-2''b), 2.07 (1H, m, H-1''a), 1.96 (1H, m, H-1''b); $^{13}\text{C-NMR}$ (CD_3OD , 100 MHz) δ : 171.4 (C-1), 165.1 (C-6, 8), 156.7 (C-4'), 143.4 (C-10), 133.1 (C-1'), 130.4 (C-2', 6'), 116.3 (C-3', 5'), 108.2 (C-5), 104.0 (C-9), 103.5 (C-7), 101.3 (C-1'''), 80.1 (C-3), 78.3 (C-5'''), 77.8 (C-3'''), 74.7 (C-2'''), 71.2 (C-4'''), 62.4 (C-6'''), 37.9 (C-1''), 33.9 (C-4), 31.1 (C-2'')。与文献 [10] 报道一致, 故鉴定为 desmethylogrimonolide 6-*O*- β -*D*-glucopyranoside。

化合物 17: 黄色无定形粉末 (甲醇), Molish 反应呈阳性。分子式 $\text{C}_{24}\text{H}_{28}\text{O}_{10}$; HR-ESI-MS m/z 499.157 0 $[\text{M}+\text{Na}]^+$ 。 $^1\text{H-NMR}$ (CD_3OD , 600 MHz) δ : 7.14 (2H, d, $J=8.6$ Hz, H-2', 6'), 6.83 (2H, d, $J=8.6$ Hz, H-3', 5'), 6.52 (1H, d, $J=2.1$ Hz, H-7), 6.50 (1H, br. s, H-5), 4.97 (1H, d, $J=7.3$ Hz, H-1'''), 4.50 (1H, m, H-3), 3.89 (1H, dd, $J=12.1, 2.0$ Hz, H-6'''a), 3.74 (3H, s, 4'-OCH₃), 3.68 (1H, dd, $J=12.1, 5.8$ Hz, H-6'''b), 3.34~3.49 (4H, m, H-2''', 3''', 4''', 5'''), 2.96 (1H, m, H-4a), 2.92 (1H, m, H-4b), 2.82 (1H, m, H-2''a), 2.72 (1H, m, H-2''b), 2.09 (1H, m, H-1''a), 1.98 (1H, m, H-1''b); $^{13}\text{C-NMR}$ (CD_3OD , 150 MHz) δ : 171.3 (C-1), 165.2 (C-6), 165.1 (C-8), 159.6 (C-4'), 143.4 (C-10), 134.3 (C-1'), 130.4 (C-2', 6'), 115.0 (C-3', 5'), 108.3 (C-5), 104.0 (C-9), 103.5 (C-7), 101.4 (C-1'''), 80.1 (C-3), 78.4 (C-5'''), 77.9 (C-3'''), 74.7 (C-2'''), 71.2 (C-4'''), 62.4 (C-6'''), 55.7 (4'-OCH₃), 37.8 (C-1''), 33.9 (C-4), 31.1 (C-2'')。与文献 [11] 报道一致, 故鉴定为仙鹤草内酯-6-*O*- β -*D*-葡萄糖吡喃糖苷。

化合物 18: 无色方晶 (甲醇), 分子式为 $\text{C}_{22}\text{H}_{26}\text{O}_8$; HR-ESI-MS m/z 441.151 7 $[\text{M}+\text{Na}]^+$ 。 $^1\text{H-NMR}$ (CD_3OD , 400 MHz) δ : 6.65 (4H, s, H-2, 2', 6, 6'), 4.71 (2H, d, $J=4.3$ Hz, H-7, 7'), 4.26 (2H, dd, $J=9.0, 6.9$ Hz, H-9a, 9'a), 3.88 (2H, dd, $J=9.0, 3.6$ Hz, H-9b, 9'b), 3.84 (12H, s, 3, 3', 5, 5'-OCH₃), 3.13 (2H, m, H-8, 8'); $^{13}\text{C-NMR}$ (CD_3OD , 100 MHz) δ : 149.4 (C-3, 3', 5, 5'), 136.2 (C-4, 4'), 133.2 (C-1, 1'), 104.6 (C-2, 2', 6, 6'), 87.6 (C-7, 7'), 72.8 (C-

9, 9'), 56.8 (3, 3', 5, 5'-OCH₃), 55.5 (C-8, 8')。与文献[12]报道一致,故鉴定为丁香脂素。

化合物 **19**: 白色无定形粉末 (甲醇), Molish 反应呈阳性, 分子式 C₂₈H₃₆O₁₃; HR-ESI-MS m/z 603.204 7 [M+Na]⁺。¹H-NMR (CD₃OD, 400 MHz) δ : 6.71 (2H, s, H-2, 6), 6.65 (2H, s, H-2', 6'), 4.86 (1H, d, $J=7.5$ Hz, H-1''), 4.76 (1H, d, $J=4.0$ Hz, H-7), 4.71 (1H, d, $J=4.3$ Hz, H-7'), 4.28 (2H, m, H-9a, 9'a), 3.91 (2H, dd, $J=9.2, 3.0$ Hz, H-9b, 9'b), 3.85 (6H, s, 3, 5-OCH₃), 3.84 (6H, s, 3', 5'-OCH₃), 3.77 (1H, dd, $J=12.0, 2.3$ Hz, H-6''a), 3.65 (1H, dd, $J=12.0, 5.1$ Hz, H-6''b), 3.47 (1H, m, H-2''), 3.41 (2H, m, H-3'', 4''), 3.18 (1H, m, H-5''), 3.13 (2H, m, H-8, 8'); ¹³C-NMR (CD₃OD, 100 MHz) δ : 154.4 (C-3, 5), 149.4 (C-3', 5'), 139.6 (C-1), 136.2 (C-4'), 135.6 (C-4), 133.1 (C-1'), 105.3 (C-1''), 104.8 (C-2, 6), 104.5 (C-2', 6'), 87.6 (C-7'), 87.2 (C-7), 78.4 (C-5''), 77.8 (C-3''), 75.7 (C-2''), 72.9 (C-9, 9'), 71.3 (C-4''), 62.6 (C-6''), 57.1 (3', 5'-OCH₃), 56.8 (3, 5-OCH₃), 55.7 (C-8'), 55.5 (C-8)。与文献 [12] 报道一致, 故鉴定为丁香脂素-4-*O*- β -D-吡喃葡萄糖苷。

化合物 **20**: 白色无定形粉末 (甲醇), Molish 反应呈阳性, 分子式 C₂₄H₂₄O₁₀; HR-ESI-MS m/z 471.128 6 [M-H]⁻。¹H-NMR (CD₃OD, 400 MHz) δ : 7.71 (1H, d, $J=15.9$ Hz, H-7'), 7.61 (1H, d, $J=15.9$ Hz, H-7''), 7.44 (4H, $J=8.6, 4.4$ Hz, H-2', 2'', 6', 6''), 6.79 (4H, dd, $J=8.7, 2.2$ Hz, H-3', 3'', 5', 5''), 6.37 (1H, d, $J=15.9$ Hz, H-8'), 6.33 (1H, d, $J=15.9$ Hz, H-8''), 5.60 (1H, d, $J=7.6$ Hz, H-1), 4.50 (1H, dd, $J=12.1, 2.0$ Hz, H-6a), 4.32 (1H, dd, $J=12.1, 5.6$ Hz, H-6b), 3.42~3.70 (4H, m, H-2, 3, 4, 5); ¹³C-NMR (CD₃OD, 100 MHz) δ : 169.2 (C-9''), 167.6 (C-9'), 161.6 (C-4'), 161.3 (C-4''), 148.1 (C-7'), 146.9 (C-7''), 131.4 (C-2', 6'), 131.3 (C-2'', 6''), 127.2 (C-1'), 127.1 (C-1''), 116.9 (C-3', 5'), 116.8 (C-3'', 5''), 114.9 (C-8''), 114.4 (C-8'), 95.8 (C-1), 77.9 (C-3), 76.3 (C-5), 74.0 (C-2), 71.4 (C-4), 64.4 (C-6)。与文献 [13] 报道一致, 故鉴定为 1, 6-di-*O*-coumaroyl glucopyranoside。

化合物 **21**: 白色无定形粉末 (甲醇), 分子式 C₁₁H₁₀O₄; HR-ESI-MS m/z 229.047 1 [M+Na]⁺。¹H-NMR (CD₃OD, 600 MHz) δ : 7.88 (1H, d, $J=9.5$ Hz, H-4), 7.13 (1H, s, H-5), 6.97 (1H, s, H-8), 6.26 (1H, d, $J=9.4$ Hz, H-3), 3.92 (3H, s, 6-OCH₃), 3.88 (3H, s, 7-OCH₃); ¹³C-NMR (CD₃OD, 150 MHz) δ : 163.8 (C-2), 154.8 (C-7), 151.3 (C-9), 148.1 (C-6), 145.9 (C-4), 113.5 (C-3), 113.0 (C-10), 110.0 (C-5), 101.0 (C-8), 56.8 (6, 7-OCH₃)。与文献 [14] 报道一致, 故鉴定为滨蒿内酯。

化合物 **22**: 白色无定形粉末 (甲醇), 分子式 C₁₀H₈O₄; HR-ESI-MS m/z 215.031 1 [M+Na]⁺。¹H-NMR (CD₃OD, 600 MHz) δ : 7.87 (1H, d, $J=9.4$ Hz, H-4), 7.12 (1H, s, H-5), 6.78 (1H, s, H-8), 6.21 (1H, d, $J=9.4$ Hz, H-3), 3.92 (3H, s, 6-OCH₃); ¹³C-NMR (CD₃OD, 150 MHz) δ : 164.1 (C-2), 153.0 (C-7), 151.5 (C-9), 147.1 (C-4), 146.2 (C-6), 112.7 (C-5, 10), 110.0 (C-3), 104.0 (C-8), 56.9 (6-OCH₃)。与文献 [15] 报道一致, 故鉴定为东莨菪内酯。

化合物 **23**: 白色无定形粉末 (甲醇), Molish 反应呈阳性, 分子式 C₃₆H₅₈O₁₀; HR-ESI-MS m/z 673.392 0 [M+Na]⁺。¹H-NMR (CD₃OD, 600 MHz) δ : 5.32 (1H, d, $J=8.1$ Hz, H-1'), 5.31 (1H, t, $J=2.5$ Hz, H-12), 3.80 (1H, dd, $J=12.0, 2.0$ Hz, H-6'a), 3.69 (1H, dd, $J=12.0, 4.7$ Hz, H-6'b), 3.63 (1H, m, H-2), 3.39 (1H, d, $J=8.8$ Hz, H-3), 1.33 (3H, s, H-27), 1.20 (3H, s, H-29), 1.01 (6H, s, H-23, 26), 0.94 (3H, d, $J=6.7$ Hz, H-30), 0.80 (3H, s, H-24), 0.78 (3H, s, H-25); ¹³C-NMR (CD₃OD, 150 MHz) δ : 178.5 (C-28), 139.7 (C-13), 129.5 (C-12), 95.8 (C-1'), 84.5 (C-3), 78.6 (C-5'), 78.3 (C-3'), 73.9 (C-2'), 73.6 (C-19), 71.1 (C-4'), 69.5 (C-2), 62.4 (C-6'), 56.7 (C-5), 55.0 (C-18), 48.7 (C-17), 48.5 (C-9), 48.2 (C-1), 42.9 (C-14), 42.7 (C-20), 41.3 (C-8), 40.5 (C-10), 39.2 (C-4), 38.3 (C-22), 34.1 (C-7), 29.6 (C-15), 29.3 (C-23), 27.2 (C-21), 27.0 (C-29), 26.5 (C-16), 24.8 (C-11), 24.6 (C-27), 19.7 (C-6), 17.6 (C-26), 17.4 (C-24), 17.1 (C-30), 16.6 (C-25)。与文献 [16] 报道一致, 故鉴定为野蔷薇亭。

化合物 **24**: 白色无定形粉末 (甲醇), 分子式

$C_{11}H_{16}O_3$; HR-ESI-MS m/z 219.099 7 $[M+Na]^+$ 。¹H-NMR (CD_3OD , 600 MHz) δ : 5.75 (1H, s, H-7), 4.22 (1H, m, H-3), 2.42 (1H, dt, $J=13.9$, 2.8 Hz, H-4b), 1.99 (1H, dt, $J=14.4$, 2.8 Hz, H-2b), 1.76 (3H, s, H-11), 1.74 (1H, d, $J=4.0$ Hz, H-4a), 1.53 (1H, dd, $J=14.4$, 3.7 Hz, H-2a), 1.47 (3H, s, H-9), 1.28 (3H, s, H-10); ¹³C-NMR (CD_3OD , 150 MHz) δ : 185.7 (C-6), 174.4 (C-8), 113.3 (C-7), 88.9 (C-5), 67.2 (C-3), 48.0 (C-2), 46.4 (C-4), 37.2 (C-1), 31.0 (C-10), 27.4 (C-11), 27.0 (C-9)。与文献 [17] 报道一致, 故鉴定为 (-)-loliolide。

化合物 **25**: 白色无定形粉末 (甲醇), 分子式 $C_{11}H_{16}O_3$; HR-ESI-MS m/z 219.099 7 $[M+Na]^+$ 。¹H-NMR (CD_3OD , 600 MHz) δ : 5.78 (1H, s, H-7), 4.10 (1H, m, H-3), 2.47 (1H, ddd, $J=11.7$, 4.0, 2.2 Hz, H-4b), 2.00 (1H, ddd, $J=12.7$, 4.0, 2.2 Hz, H-2b), 1.59 (3H, s, H-11), 1.42 (1H, m, H-4a), 1.32 (3H, s, H-10), 1.29 (3H, s, H-9), 1.29 (1H, overlap, H-2a); ¹³C-NMR (CD_3OD , 150 MHz) δ : 183.9 (C-6), 174.0 (C-8), 113.7 (C-7), 88.5 (C-5), 65.2 (C-3), 50.7 (C-2), 48.6 (C-4), 36.1 (C-1), 30.3 (C-10), 25.8 (C-11), 25.3 (C-9)。与文献 [17] 报道一致, 故鉴定为 (+)-isololiolide。

化合物 **26**: 黄色油状物 (甲醇), 分子式 $C_{16}H_{22}O_4$; HR-ESI-MS m/z 301.140 7 $[M+Na]^+$ 。¹H-NMR (CD_3OD , 600 MHz) δ : 7.72 (2H, dd, $J=5.7$, 3.3 Hz, H-2, 5), 7.62 (2H, dd, $J=5.7$, 3.3 Hz, H-3, 4), 4.30 (4H, t, $J=6.6$ Hz, H-1', 1''),

1.73 (4H, m, H-2', 2''), 1.47 (4H, m, H-3', 3''), 1.00 (6H, t, $J=7.4$ Hz, H-4', 4''); ¹³C-NMR (CD_3OD , 150 MHz) δ : 169.3 (C-7, 7'), 133.6 (C-1, 6), 132.3 (C-3, 4), 129.9 (C-2, 5), 66.6 (C-1', 1''), 31.7 (C-2', 2''), 20.2 (C-3', 3''), 14.0 (C-4', 4'')。与文献 [18] 报道一致, 故鉴定为邻苯二甲酸二丁酯。

化合物 **27**: 白色无定形粉末 (甲醇), 分子式 $C_7H_6O_3$; HR-ESI-MS m/z 145.026 1 $[M+Na]^+$ 。¹H-NMR (CD_3OD , 400 MHz) δ : 9.75 (1H, s, -CHO), 7.76 (2H, d, $J=8.6$ Hz, H-2, 6), 6.90 (2H, d, $J=8.6$ Hz, H-3, 5); ¹³C-NMR (CD_3OD , 100 MHz) δ : 192.8 (-CHO), 165.4 (C-4), 133.4 (C-2, 6), 130.2 (C-1), 116.9 (C-3, 5)。与文献 [17] 报道一致, 故鉴定为对羟基苯甲醛。

化合物 **28**: 白色无定形粉末 (甲醇), 分子式 $C_9H_{10}O_5$; HR-ESI-MS m/z 221.041 8 $[M+Na]^+$ 。¹H-NMR (CD_3OD , 600 MHz) δ : 7.05 (2H, s, H-2, 6), 4.27 (2H, q, $J=7.1$ Hz, H-8), 1.35 (3H, t, $J=7.1$ Hz, H-9); ¹³C-NMR (CD_3OD , 150 MHz) δ : 168.6 (C-7), 146.5 (C-3, 5), 139.7 (C-4), 121.8 (C-1), 110.0 (C-2, 6), 61.6 (C-8), 14.6 (C-9)。与文献 [19] 报道一致, 故鉴定为没食子酸乙酯。

4 抗炎活性筛选

参照文献 [2], 利用脂多糖 (LPS) 刺激 RAW264.7 巨噬细胞炎症模型, 对各化合物抑制 NO 生成作用进行测定, 结果见表 1。各化合物均能对 NO 释放呈现不同程度的抑制作用, 其中化合物 **9**、**20** 作用较强。

表 1 各化合物对 LPS 刺激 RAW264.7 细胞中 NO 生成的影响 ($\mu\text{mol/L}$, $\bar{x} \pm s$, $n=3$)

Tab. 1 Effects of various compounds on NO production in LPS-stimulated RAW264.7 cells ($\mu\text{mol/L}$, $\bar{x} \pm s$, $n=3$)

化合物	IC ₅₀	化合物	IC ₅₀	化合物	IC ₅₀
N-甲基-L-精氨酸	14.77±1.50	10	>50	20	16.26±0.64
1	48.02±3.71	11	45.96±1.76	21	>50
2	>50	12	37.49±3.15	22	>50
3	>50	13	35.76±2.20	23	41.84±2.24
4	47.67±1.55	14	>50	24	>50
5	44.19±3.86	15	>50	25	41.56±2.24
6	>50	16	45.39±2.39	26	—
7	>50	17	>50	27	—
8	33.66±2.11	18	36.98±1.77	28	—
9	25.14±3.54	19	>50		

5 讨论

本研究采用各种分离鉴定手段, 最终从洋金花叶 70% 乙醇提取物中分离出 28 个化合物, 包括 14 个醉茄内酯类、1 个糖苷类、7 个苯丙素类、6 个其他类, 并且各化合物均能不同程度地抑制 NO 的释放, 其中化合物 9、20 作用更明显, 3、13、14 被发现具有潜在的抗增殖、抗免疫作用^[4], 可为该药材开发利用、化学成分研究及生物活性筛选提供理论依据和实验基础。

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