木果楝的化学成分研究

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摘 要：目的 对木果楝 *Xylocarpus granatum* 果实的化学成分进行研究。**方法** 利用柱色谱、制备液相色谱、葡聚糖凝胶色谱等多种色谱方法分离纯化，用 HR-ESI-MS、^{1}H-NMR、^{13}C-NMR、H-H COSY、HMQC 和 HMBC 鉴定化合物结构。**结果** 从木果楝果实 95%乙醇提取物中分离得到 17 个化合物，分别鉴定为 grantumin C（**1**）、cipadesin A（**2**）、xylocarpin G（**3**）、febrifugin（**4**）、tigloylseneganolide A（**5**）、khaysin T（**6**）、cipadesin（**7**）、granatumin B（**8**）、xyloccensin S（**9**）、granaxylocarpin C（**10**）、xylogranatin E_2（**11**）、xyloccensin Q（**12**）、xyloccensin P（**13**）、cedrodorin（**14**）、xylorumphiins D（**15**）、hydroxydammarenone-II（**16**）、邻苯二甲酸双 (2-乙基己基) 酯（**17**）。**结论** 化合物 **2**、**7**、**14**、**16**、**17** 为首次从该属植物中分离得到；化合物 **15** 为首次从该植物中分离得到。

关键词：木果楝；柠檬苦素；cipadesin A；cedrodorin；邻苯二甲酸双 (2-乙基己基) 酯

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Chemical constituents of *Xylocarpus granatum*

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Abstract: Objective To study the chemical constituents of the fruits of *Xylocarpus granatum*. **Methods** The isolation and purification were carried out by silica gel column chromatography, Sephadex, preparative TLC, and preparative HPLC. The structures of the isolated compounds were identified with the help of HR-ESI-MS, ^{1}H-NMR, ^{13}C-NMR, H-H COSY, HMQC and HMBC techniques. **Results** Seventeen compounds were isolated and elucidated from the fruits of *X. granatum*, their structures were identified as: grantumin C (**1**), cipadesin A (**2**), xylocarpin G (**3**), febrifugin (**4**), tigloylseneganolide A (**5**), khaysin T (**6**), cipadesin (**7**), granatumin B (**8**), xyloccensin S (**9**), granaxylocarpin C (**10**), xylogranatin E_2 (**11**), xyloccensin Q (**12**), xyloccensin P (**13**), cedrodorin (**14**), xylorumphiins D (**15**), hydroxydammarenone-II (**16**), and bis (2-ethylhexyl) phthalate (**17**). **Conclusion** Compounds **2**, **7**, **14**, **16** and **17** are isolated from the plants of *Xylocarpus* Koenig for the first time. Compound **15** is obtained from *X. granatum* for the first time.

Key words: *Xylocarpus granatum* Koenig; limonoid; cipadesin A; cedrodorin; bis (2-ethylhexyl) phthalate

木果楝 *Xylocarpus granatum* Koenig 为楝科木果楝属植物，广泛分布于东南亚、澳洲、东非和印度沿海地带，我国只有海南省有生长[1-2]。已报道木果楝含有各种化学成分约 110 个，包括萜类、生物碱类、多酚类、黄酮类、甾体类等，且具有细胞毒、抗虫、抗菌以及止泻等活性[3-8]。本实验对木果楝果实的化学成分进行研究，从其 95%乙醇提取物中分离得到了 17 个化合物，分别鉴定为 grantumin C（**1**）、

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cipadesin A（**2**）、xylocarpin G（**3**）、febrifugin（**4**）、tigloylseneganolide A（**5**）、khaysin T（**6**）、cipadesin（**7**）、granatumin B（**8**）、xyloccensin S（**9**）、granaxylocarpin C（**10**）、xylogranatin E_2（**11**）、xyloccensin Q（**12**）、xyloccensin P（**13**）、cedrodorin（**14**）、xylorumphiins D（**15**）、hydroxydammarenone-II（**16**）、邻苯二甲酸双(2-乙基己基)酯 [bis (2-ethylhexyl) phthalate，**17**]。其中，化合物 **2**、**7**、**14**、**16**、**17** 为首次从该属植物中分离得到；化合物 **15** 为首次从该植物中分离得到。

1 仪器与材料

Bruker APEX II 质谱仪（美国布鲁克公司）；Bruker AV-500 核磁仪（美国布鲁克公司）；Agilent 1100 制备型高效液相色谱仪（美国 Agilent 公司）；色谱柱：Whatman partisil 10 ODS-3（250 mm×20 mm，5 μm），Whatman partisil 10 ODS-2（250 mm×9.4 mm，5 μm）；Laborota 4000 旋转蒸发仪（德国 Heidolph 公司）。薄层色谱硅胶 G 和 GF_{254}、柱色谱用硅胶（100～200、300～400 目），青岛海洋化工厂；甲醇、乙腈为色谱纯试剂（北京迪马科技有限公司），其他试剂均为分析纯。

木果楝果实 2013 年 3 月采自海南省，室温干燥，由厦门大学王文清教授鉴定为木果楝 *Xylocarpus granatum* Koenig 果实。标本（HEBNMC-2013-1）保存在河北医科大学药学院植物标本室。

2 提取与分离

取粉碎后的药材约 21 kg，用 95%乙醇冷浸提取 3 次。提取液滤过，减压浓缩至膏状，得浸膏约 1 000 g。分别用石油醚、二氯甲烷和醋酸乙酯萃取，得到石油醚部分浸膏（60 g）、二氯甲烷部分浸膏（485 g）和醋酸乙酯部分浸膏（25 g）。二氯甲烷浸膏采用硅胶柱色谱分离，石油醚-醋酸乙酯梯度洗脱（30∶1→1∶10），得到 9 个流分 Fr. 1～9。

Fr. 3 采用柱色谱分离，石油醚-丙酮（15∶1）洗脱，进一步采用制备薄层色谱分离（石油醚-丙酮 5∶1），得到化合物 **16**（9.1mg）。

Fr. 5 采用柱色谱分离，石油醚-丙酮（3∶1）洗脱，得到化合物 **2**（2.0 g）；进一步制备液相色谱分离（250 mm×20 mm 制备柱），乙腈-水（53∶47）洗脱，得到化合物 **1**（2.0 mg）、**3**（2.2 mg）、**4**（2.5 mg）、**5**（3.9 mg）、**6**（4.3 mg）、**7**（5.5 mg）、**8**（4.7 mg）、**17**（2.0 mg）。

Fr. 8 采用柱色谱分离，石油醚-丙酮（2∶1、1∶1、1∶2、1∶3）洗脱，经制备液相色谱分离（250 mm×20 mm 制备柱），乙腈-水（38∶62）洗脱，得到化合物 **9**（2.4 mg）、**10**（3.5 mg）、**11**（11.6 mg）、**12**（9.4 mg）、**13**（8.1 mg）、**14**（5.4 mg）、**15**（7.2 mg）。

3 结构鉴定

化合物 **1**：白色粉末，^{1}H-NMR (500 MHz, $CDCl_3$) δ: 3.68 (1H, dd, J = 9.2, 2.2 Hz, H-2), 5.13 (1H, d, J = 9.2 Hz, H-3), 3.45 (1H, dd, J = 9.7, 1.6 Hz, H-5), 2.32 (2H, m, H-6), 1.98 (1H, m, H-9), 1.52 (1H, m, H-11a), 1.72 (1H, m, H-11b), 2.02 (1H, m, H-12a), 1.28 (1H, m, H-12b), 6.13 (1H, s, H-15), 5.23 (1H, s, H-17), 1.16 (3H, s, H-18), 1.09 (3H, s, H-19), 7.51 (1H, s, H-21), 6.51 (1H, d, J = 1.9 Hz, H-22), 7.46 (1H, d, J = 1.9 Hz, H-23), 0.83 (3H, s, H-28), 0.83 (3H, s, H-29), 3.95 (1H, d, J = 2.2 Hz, H-30), 3.72 (3H, s, 7-OMe), 7.08 (1H, qq, J = 7.2, 1.3 Hz, H-3′), 1.95 (3H, dq, J = 7.2, 1.3 Hz, H-4′), 1.96 (3H, q, J = 1.3 Hz, H-5′)；^{13}C-NMR (125 MHz, $CDCl_3$) δ: 214.0 (C-1), 48.1 (C-2), 76.9 (C-3), 39.8 (C-4), 41.3 (C-5), 32.7 (C-6), 173.4 (C-7), 60.2 (C-8), 55.5 (C-9), 48.6 (C-10), 20.5 (C-11), 32.6 (C-12), 39.0 (C-13), 160.7 (C-14), 118.5 (C-15), 163.6 (C-16), 79.0 (C-17), 20.8 (C-18), 15.5 (C-19), 119.6 (C-20), 141.5 (C-21), 110.0 (C-22), 142.9 (C-23), 20.0 (C-28), 20.5 (C-29), 61.7 (C-30), 52.0 (7-OMe), 166.4 (C-1′), 127.5 (C-2′), 139.7 (C-3′), 14.3 (C-4′), 11.9 (C-5′)。以上波谱数据与文献报道一致[9]，故鉴定化合物 **1** 为 grantumin C。

化合物 **2**：白色粉末，^{1}H-NMR (500 MHz, $CDCl_3$) δ: 3.58 (1H, dd, J = 9.4, 2.5 Hz, H-2), 5.10 (1H, d, J = 9.4 Hz, H-3), 3.24 (1H, dd, J = 8.6, 2.7 Hz, H-5), 2.34 (2H, m, H-6), 1.91 (1H, m, H-9), 2.19 (1H, m, H-11a), 1.57 (1H, m, H-11b), 1.75 (1H, m, H-12a), 1.52 (1H, m, H-12b), 1.55 (1H, m, H-14), 3.69 (1H, m, H-15a), 2.82 (1H, dd, J = 16.0, 4.6 Hz, H-15b), 5.17 (1H, s, H-17), 1.02 (3H, s, H-18), 1.08 (3H, s, H-19), 7.48 (1H, s, H-21), 6.46 (1H, d, J = 1.9 Hz, H-22), 7.44 (1H, d, J = 1.9 Hz, H-23), 0.82 (3H, s, H-28), 0.81 (3H, s, H-29), 3.33 (1H, d, J = 2.5 Hz, H-30), 3.74 (3H, s, 7-OMe), 2.58 (1H, m, H-2′), 1.76 (1H, m, H-3′a), 1.54 (1H, m, H-3′b), 0.99 (3H, t, J = 7.5 Hz, H-4′), 1.26 (3H, m, H-5′)；^{13}C-NMR (125 MHz, $CDCl_3$) δ: 213.9 (C-1), 48.7 (C-2), 76.7 (C-3), 39.2 (C-4), 42.2 (C-5), 32.9 (C-6), 173.9 (C-7), 60.4

(C-8), 55.8 (C-9), 48.0 (C-10), 20.5 (C-11), 33.0 (C-12), 36.2 (C-13), 45.6 (C-14), 33.9 (C-15), 172.0 (C-16), 78.6 (C-17), 26.4 (C-18), 15.6 (C-19), 120.0 (C-20), 140.9 (C-21), 110.1 (C-22), 143.1 (C-23), 20.8 (C-28), 22.3 (C-29), 63.4 (C-30), 52.1 (7-OMe), 175.7 (C-1′), 41.4 (C-2′), 26.8 (C-3′), 11.8 (C-4′), 17.1 (C-5′)。以上波谱数据与文献报道一致[10]，故鉴定化合物 **2** 为 cipadesin A。

化合物 **3**：白色粉末，^{1}H-NMR (500 MHz, $CDCl_3$) δ: 3.00 (1H, dd, J = 9.1, 4.5 Hz, H-2), 5.03 (1H, d, J = 9.1 Hz, H-3), 2.64 (1H, brd, J = 10.4 Hz, H-5), 2.37 (1H, m, H-6a), 2.19 (1H, m, H-6b), 2.16 (1H, m, H-9), 2.42 (1H, m, H-11a), 1.81 (1H, m, H-11b), 2.25 (1H, m, H-12a), 1.42 (1H, m, H-12b), 5.96 (1H, s, H-15), 4.96 (1H, s, H-17), 1.21 (3H, s, H-18), 1.07 (3H, s, H-19), 7.49 (1H, s, H-21), 6.43 (1H, d, J = 1.5 Hz, H-22), 7.42 (1H, d, J = 1.5 Hz, H-23), 1.25 (3H, s, H-28), 0.81 (3H, s, H-29), 5.67 (1H, d, J = 4.5 Hz, H-30), 3.70 (3H, s, 7-OMe), 6.86 (1H, qq, J = 7.2, 1.5 Hz, H-3′), 1.79 (3H, m, H-4′), 1.78 (3H, brs, H-5′), 3.74 (1H, brs, 1-OH), 1.92 (3H, s, 3-OAc)；^{13}C-NMR (125 MHz, $CDCl_3$) δ: 107.4 (C-1), 53.2 (C-2), 74.0 (C-3), 37.3 (C-4), 40.6 (C-5), 32.2 (C-6), 173.9 (C-7), 81.5 (C-8), 55.5 (C-9), 43.0 (C-10), 15.0 (C-11), 25.0 (C-12), 38.9 (C-13), 159.4 (C-14), 117.6 (C-15), 163.5 (C-16), 81.4 (C-17), 19.4 (C-18), 20.5 (C-19), 120.1 (C-20), 141.2 (C-21), 109.9 (C-22), 142.9 (C-23), 21.8 (C-28), 24.5 (C-29), 76.4 (C-30), 52.9 (7-OMe), 167.1 (C-1′), 127.6 (C-2′), 139.7 (C-3′), 14.6 (C-4′), 11.9 (C-5′), 170.0, 20.6 (3-OAc)。以上波谱数据与文献报道一致[11]，故鉴定定化合物 **3** 为 xylocarpin G。

化合物 **4**：白色粉末，^{1}H-NMR (500 MHz, $CDCl_3$) δ: 3.53 (1H, dd, J = 9.4, 1.9 Hz, H-2), 4.85 (1H, d, J = 9.4 Hz, H-3), 3.48 (1H, dd, J = 9.5, 2.4 Hz, H-5), 2.38 (2H, m, H-6), 2.22 (1H, m, H-9), 2.11 (1H, m, H-11a), 1.66 (1H, m, H-11b), 1.66 (1H, m, H-12a), 1.41 (1H, m, H-12b), 2.21 (1H, m, H-14), 2.85 (1H, dd, J = 18.9, 6.6 Hz, H-15a), 2.77 (1H, d, J = 18.9 Hz, H-15b), 5.64 (1H, s, H-17), 1.09 (3H, s, H-18), 1.16 (3H, s, H-19), 7.84 (1H, s, H-21), 6.48 (1H, d, J = 1.8 Hz, H-22), 7.43 (1H, d, J = 1.8 Hz, H-23), 0.85 (3H, s, H-28), 0.82 (3H, s, H-29), 5.35 (1H, d, J = 1.9 Hz, H-30), 3.73 (3H, s, 7-OMe), 6.94 (1H, qq, J = 7.2, 1.4 Hz, H-3′), 1.75 (3H, m, H-4′), 1.84 (3H, brs, H-5′)；^{13}C-NMR (125 MHz, $CDCl_3$) δ: 217.1 (C-1), 48.2 (C-2), 76.8 (C-3), 38.2 (C-4), 41.1 (C-5), 32.7 (C-6), 173.9 (C-7), 138.4 (C-8), 56.6 (C-9), 49.8 (C-10), 20.4 (C-11), 34.1 (C-12), 36.6 (C-13), 44.8 (C-14), 29.5 (C-15), 168.6 (C-16), 76.4 (C-17), 21.4 (C-18), 15.5 (C-19), 120.6 (C-20), 141.6 (C-21), 109.5 (C-22), 142.8 (C-23), 20.0 (C-28), 22.3 (C-29), 122.7 (C-30), 52.0 (7-OMe), 166.9 (C-1′), 127.4 (C-2′), 139.4 (C-3′), 14.2 (C-4′), 11.5 (C-5′)。以上波谱数据与文献报道一致[12]，故鉴定化合物 **4** 为 febrifugin。

化合物 **5**：白色粉末，^{1}H-NMR (500 MHz, $CDCl_3$) δ: 3.74 (1H, dd, J = 9.3, 2.8 Hz, H-2), 4.92 (1H, d, J = 9.3 Hz, H-3), 3.35 (1H, dd, J = 10.0, 1.7 Hz, H-5), 2.38 (2H, m, H-6), 2.26 (1H, m, H-9), 1.78 (1H, m, H-11a), 1.50 (1H, m, H-11b), 1.94 (2H, m, H-12), 6.17 (1H, s, H-15), 5.16 (1H, s, H-17), 1.04 (3H, s, H-18), 1.21 (3H, s, H-19), 7.51 (1H, s, H-21), 6.49 (1H, d, J = 1.9 Hz, H-22), 7.44 (1H, d, J = 1.9 Hz, H-23), 0.84 (3H, s, H-28), 0.80 (3H, s, H-29), 6.26 (1H, d, J = 2.8 Hz, H-30), 3.70 (3H, s, 7-OMe), 7.01 (1H, qq, J = 7.0, 1.0 Hz, H-3′), 1.91 (3H, m, H-4′), 1.93 (3H, brs, H-5′)；^{13}C-NMR (125 MHz, $CDCl_3$) δ: 214.2 (C-1), 48.9 (C-2), 78.0 (C-3), 38.9 (C-4), 40.1 (C-5), 32.5 (C-6), 173.4 (C-7), 135.8 (C-8), 53.9 (C-9), 51.8 (C-10), 21.9 (C-11), 32.5 (C-12), 37.2 (C-13), 160.5 (C-14), 112.3 (C-15), 164.6 (C-16), 79.3 (C-17), 22.0 (C-18), 15.4 (C-19), 119.8 (C-20), 141.0 (C-21), 110.1 (C-22), 143.1 (C-23), 20.8 (C-28), 22.3 (C-29), 129.1 (C-30), 51.8 (7-OMe), 166.7 (C-1′), 127.6 (C-2′), 139.2 (C-3′), 14.6 (C-4′), 11.9 (C-5′)。以上波谱数据与文献报道一致[10]，故鉴定化合物 **5** 为 tigloylseneganolide A。

化合物 **6**：白色粉末，^{1}H-NMR (500 MHz, $CDCl_3$) δ: 3.25 (1H, m, H-2), 4.86 (1H, d, J = 9.6 Hz, H-3), 3.38 (1H, brd, J = 9.8 Hz, H-5), 2.39 (2H, m, H-6), 2.03 (1H, m, H-9), 1.83 (1H, m, H-11a), 1.72 (1H, m, H-11b), 1.81 (1H, m, H-12a), 1.10 (1H, m, H-12b), 3.62 (1H, d, J = 21.3 Hz, H-15a), 3.26 (1H, d, J = 21.3 Hz, H-15b), 5.56 (1H, s, H-17), 1.03 (3H, s, H-18), 1.17 (3H, s, H-19), 7.58 (1H, s, H-21), 6.49 (1H, d, J = 1.9 Hz, H-22), 7.42 (1H, d, J = 1.9 Hz,

H-23), 0.83 (3H, s, H-28), 0.78 (3H, s, H-29), 2.67 (1H, d, *J* = 15.1 Hz, H-30a), 2.09 (1H, d, *J* = 15.1 Hz, H-30b), 3.74 (3H, s, 7-OMe), 6.97 (1H, brq, *J* = 7.0 Hz, H-3′), 1.84 (3H, brd, *J* = 7.0 Hz, H-4′), 1.90 (3H, brs, H-5′); ^{13}C-NMR (125 MHz, $CDCl_3$) *δ*: 218.3 (C-1), 48.3 (C-2), 79.3 (C-3), 38.7 (C-4), 40.5 (C-5), 32.9 (C-6), 174.3 (C-7), 127.8 (C-8), 52.1 (C-9), 53.2 (C-10), 18.8 (C-11), 28.8 (C-12), 38.1 (C-13), 131.9 (C-14), 32.9 (C-15), 169.7 (C-16), 80.9 (C-17), 17.3 (C-18), 16.7 (C-19), 120.8 (C-20), 141.7 (C-21), 110.0 (C-22), 142.8 (C-23), 20.3 (C-28), 23.8 (C-29), 33.4 (C-30), 52.2 (7-OMe), 167.4 (C-1′), 128.9 (C-2′), 139.4 (C-3′), 14.6 (C-4′), 12.4 (C-5′)。以上波谱数据与文献报道一致[13]，故鉴定化合物 **6** 为 khaysin。

化合物 **7**：白色粉末，^{1}H-NMR (500 MHz, $CDCl_3$) *δ*: 3.50 (1H, dd, *J* = 9.4, 1.9 Hz, H-2), 4.83 (1H, d, *J* = 9.4 Hz, H-3), 3.43 (1H, dd, *J* = 8.3, 3.2 Hz, H-5), 2.38 (2H, m, H-6), 2.23 (1H, m, H-9), 2.10 (1H, m, H-11a), 1.66 (1H, m, H-11b), 1.66 (1H, m, H-12a), 1.41 (1H, m, H-12b), 2.25 (1H, m, H-14), 2.90 (1H, dd, *J* = 18.5, 6.2 Hz, H-15a), 2.84 (1H, d, *J* = 18.4 Hz, H-15b), 5.69 (1H, s, H-17), 1.10 (3H, s, H-18), 1.15 (3H, s, H-19), 7.79 (1H, s, H-21), 6.47 (1H, d, *J* = 1.5 Hz, H-22), 7.42 (1H, d, *J* = 1.5 Hz, H-23), 0.83 (3H, s, H-28), 0.79 (3H, s, H-29), 5.39 (1H, d, *J* = 1.9 Hz, H-30), 3.72 (3H, s, 7-OMe), 2.46 (1H, m, H-2′), 1.71 (1H, m, H-3′a), 1.47 (1H, m, H-3′b), 0.93 (3H, t, H-4′), 1.15 (3H, d, *J* = 7.2 Hz, H-5′); ^{13}C-NMR (125 MHz, $CDCl_3$) *δ*: 216.9 (C-1), 48.6 (C-2), 76.6 (C-3), 38.5 (C-4), 41.1 (C-5), 32.6 (C-6), 173.8 (C-7), 137.9 (C-8), 56.5 (C-9), 49.8 (C-10), 20.3 (C-11), 34.2 (C-12), 36.6 (C-13), 44.9 (C-14), 29.4 (C-15), 169.0 (C-16), 76.7 (C-17), 21.6 (C-18), 15.5 (C-19), 120.4 (C-20), 141.7 (C-21), 109.4 (C-22), 142.6 (C-23), 20.4 (C-28), 22.1 (C-29), 122.7 (C-30), 51.8 (7-OMe), 175.8 (C-1′), 40.5 (C-2′), 26.8 (C-3′), 11.1 (C-4′), 15.7 (C-5′)。以上波谱数据与文献报道一致[14]，故鉴定化合物 **7** 为 cipadesin。

化合物 **8**：白色粉末，^{1}H-NMR (500 MHz, $CDCl_3$) *δ*: 3.68 (1H, dd, *J* = 9.3, 2.8 Hz, H-2), 4.90 (1H, d, *J* = 9.3 Hz, H-3), 3.32 (1H, dd, *J* = 9.6, 2.1 Hz, H-5), 2.39 (1H, dd, *J* = 16.8, 9.6 Hz, H-6a), 2.34 (1H, dd, *J* = 16.8, 2.1 Hz, H-6b), 2.30 (1H, m, H-9), 1.76 (1H, m, H-11a), 1.54 (1H, m, H-11b), 1.91 (1H, m, H-12a), 1.29 (1H, m, H-12b), 6.25 (1H, s, H-15), 5.16 (1H, s, H-17), 1.06 (3H, s, H-18), 1.20 (3H, s, H-19), 7.52 (1H, s, H-21), 6.49 (1H, d, *J* = 1.7 Hz, H-22), 7.44 (1H, d, *J* = 1.7 Hz, H-23), 0.83 (3H, s, H-28), 0.79 (3H, s, H-29), 6.31 (1H, d, *J* = 2.8 Hz, H-30), 3.70 (3H, s, 7-OMe), 2.23 (1H, m, H-2′), 1.79 (1H, m, H-3′a), 1.56 (1H, m, H-3′b), 1.02 (3H, t, H-4′), 1.24 (3H, brd, *J* = 7.2 Hz, H-5′); ^{13}C-NMR (125 MHz, $CDCl_3$) *δ*: 214.1 (C-1), 48.7 (C-2), 77.6 (C-3), 38.9 (C-4), 40.2 (C-5), 32.6 (C-6), 173.5 (C-7), 135.7 (C-8), 53.2 (C-9), 51.9 (C-10), 21.0 (C-11), 32.1 (C-12), 37.4 (C-13), 160.3 (C-14), 112.0 (C-15), 164.6 (C-16), 79.2 (C-17), 21.4 (C-18), 15.3 (C-19), 119.9 (C-20), 141.1 (C-21), 109.8 (C-22), 142.8 (C-23), 20.9 (C-28), 21.9 (C-29), 128.7 (C-30), 51.6 (7-OMe), 175.3 (C-1′), 40.9 (C-2′), 26.6 (C-3′), 11.5 (C-4′), 16.7 (C-5′)。以上波谱数据与文献报道一致[9]，故鉴定化合物 **8** 为 granatumin B。

化合物 **9**：白色粉末，^{1}H-NMR (500 MHz, $CDCl_3$) *δ*: 5.18 (1H, s, H-3), 2.29 (1H, brs, H-5), 5.09 (1H, brs, H-6), 2.35 (1H, dd, *J* = 14.1, 4.2 Hz, H-11a), 1.97 (1H, m, H-11b), 4.82 (1H, dd, *J* = 13.3, 4.2 Hz, H-12), 6.61 (1H, s, H-15), 5.84 (1H, s, H-17), 1.59 (3H, s, H-18), 1.55 (3H, s, H-19), 7.40 (1H, s, H-21), 6.55 (1H, d, *J* = 1.7 Hz, H-22), 7.41 (1H, d, *J* = 1.7 Hz, H-23), 2.40 (1H, d, *J* = 11.0 Hz, H-28a), 1.84 (1H, d, *J* = 11.0 Hz, H-28b), 0.91 (3H, s, H-29), 5.34 (1H, s, H-30), 1.71 (3H, s, H-32), 3.82 (3H, s, 7-OMe), 3.38 (1H, brs, 1-OH), 2.55 (1H, brs, 6-OH), 2.18 (3H, s, 2-OAc), 2.07 (3H, s, 3-OAc), 1.53 (3H, s, 12-OAc); ^{13}C-NMR (125 MHz, $CDCl_3$) *δ*: 84.1 (C-1), 85.7 (C-2), 85.4 (C-3), 44.2 (C-4), 44.9 (C-5), 70.6 (C-6), 174.3 (C-7), 83.5 (C-8), 86.1 (C-9), 48.4 (C-10), 32.3 (C-11), 68.8 (C-12), 42.9 (C-13), 152.4 (C-14), 123.7 (C-15), 163.2 (C-16), 78.5 (C-17), 14.1 (C-18), 16.8 (C-19), 120.7 (C-20), 141.5 (C-21), 109.9 (C-22), 142.9 (C-23), 40.5 (C-28), 14.9 (C-29), 73.8 (C-30), 119.2 (C-31), 16.2 (C-32), 52.8 (7-OMe), 170.2/21.7 (2-OAc), 168.8/21.5 (3-OAc), 170.1/19.7 (12-OAc)。以上波谱数据与文献报道一致[15]，故鉴定鉴定化合物 **9** 为 xyloccensin S。

化合物 **10**：白色粉末，^{1}H-NMR (500 MHz,

$CDCl_3$) δ: 3.07 (1H, dd, J = 10.2, 2.8 Hz, H-2), 5.17 (1H, d, J = 10.2 Hz, H-3), 2.90 (1H, d, J = 11.3 Hz, H-5), 2.43 (1H, dd, J = 17.0, 11.0 Hz, H-6a), 2.33 (1H, d, J = 17.0 Hz, H-6b), 2.27 (1H, m, H-9), 1.81 (2H, m, H-11), 1.81 (1H, m, H-12a), 1.75 (1H, m, H-12b), 3.02 (1H, d, J = 17.0 Hz, H-15a), 2.65 (1H, d, J = 17.0 Hz, H-15b), 5.30 (1H, s, H-17), 0.98 (3H, s, H-18), 1.08 (3H, s, H-19), 7.65 (1H, s, H-21), 6.50 (1H, d, J = 1.9 Hz, H-22), 7.43 (1H, d, J = 1.9 Hz, H-23), 3.91 (1H, d, J = 9.8 Hz, H-28a), 3.54 (1H, d, J = 9.8 Hz, H-28b), 0.67 (3H, s, H-29), 3.41 (1H, d, J = 2.8 Hz, H-30), 3.73 (3H, s, 7-OMe), 7.04 (1H, brq, J = 7.0 Hz, H-3′), 1.81 (3H, brd, J = 7.0 Hz, H-4′), 1.91 (3H, brs, H-5′), 3.86 (1H, brs, 1-OH), 2.57 (1H, brs, 14-OH)；^{13}C-NMR (125 MHz, $CDCl_3$) δ: 96.8 (C-1), 41.8 (C-2), 75.1 (C-3), 40.0 (C-4), 35.5 (C-5), 32.0 (C-6), 173.8 (C-7), 63.8 (C-8), 45.0 (C-9), 41.7 (C-10), 17.8 (C-11), 31.9 (C-12), 40.0 (C-13), 72.1 (C-14), 39.3 (C-15), 168.8 (C-16), 78.5 (C-17), 19.6 (C-18), 15.1 (C-19), 120.4 (C-20), 141.4 (C-21), 110.3 (C-22), 142.9 (C-23), 67.4 (C-28), 15.1 (C-29), 60.9 (C-30), 52.2 (7-OMe), 167.6 (C-1′), 127.4 (C-2′), 140.6 (C-3′), 14.7 (C-4′), 12.2 (C-5′)。以上波谱数据与文献报道一致[16]，故鉴定化合物 **10** 为 granaxylocarpin C。

化合物 **11**：白色粉末，^{1}H-NMR (500 MHz, $CDCl_3$) δ: 2.75 (1H, m, H-2), 4.71 (1H, d, J = 10.6 Hz, H-3), 2.72 (1H, d, J = 11.9 Hz, H-5), 2.35 (1H, m, H-6), 1.75 (1H, m, H-9), 1.74 (2H, m, H-11), 1.60 (2H, m, H-12), 6.16 (1H, s, H-15), 5.19 (1H, s, H-17), 1.30 (3H, s, H-18), 1.06 (3H, s, H-19), 7.51 (1H, s, H-21), 6.49 (1H, d, J = 1.9 Hz, H-22), 7.44 (1H, d, J = 1.9 Hz, H-23), 2.04 (1H, d, J = 11.3 Hz, H-28a), 1.41 (1H, d, J = 11.3 Hz, H-28b), 0.88 (3H, s, H-29), 2.14 (1H, dd, J = 15.7, 10.2 Hz, H-30a), 1.89 (1H, m, H-30b), 3.68 (3H, s, 7-OMe), 7.05 (1H, brq, J = 7.0 Hz, H-3′), 1.85 (3H, brd, J = 7.0 Hz, H-4′), 1.90 (3H, brs, H-5′)；^{13}C-NMR (125 MHz, $CDCl_3$) δ: 79.8 (C-1), 40.1 (C-2), 79.9 (C-3), 45.0 (C-4), 37.3 (C-5), 34.1 (C-6), 173.5 (C-7), 73.7 (C-8), 47.4 (C-9), 45.8 (C-10), 32.1 (C-11), 33.0 (C-12), 38.2 (C-13), 164.7 (C-14), 115.9 (C-15), 168.8 (C-16), 79.9 (C-17), 23.0 (C-18), 20.3 (C-19), 120.9 (C-20), 141.4 (C-21), 110.0 (C-22), 142.8 (C-23), 42.7 (C-28), 14.7 (C-29), 26.4 (C-30), 51.6 (7-OMe), 167.8 (C-1′), 128.2 (C-2′), 138.5 (C-3′), 14.3 (C-4′), 11.9 (C-5′)。以上波谱数据与文献报道一致[17]，故鉴定化合物 **11** 为 xylogranatin E_2。

化合物 **12**：白色粉末，^{1}H-NMR (500 MHz, $CDCl_3$) δ: 5.15 (1H, s, H-3), 2.51 (1H, brs, H-5), 6.34 (1H, brs, H-6), 2.34 (1H, dd, J = 14.0, 4.2 Hz, H-11a), 2.03 (1H, m, H-11b), 4.92 (1H, dd, J = 13.4, 4.2 Hz, H-12), 6.44 (1H, s, H-15), 5.95 (1H, s, H-17), 1.60 (3H, s, H-18), 1.26 (3H, s, H-19), 7.45 (1H, s, H-21), 6.56 (1H, d, J = 1.7 Hz, H-22), 7.41 (1H, d, J = 1.7 Hz, H-23), 2.13 (1H, d, J = 10.8 Hz, H-28a), 1.84 (1H, d, J = 10.8 Hz, H-28b), 0.95 (3H, s, H-29), 4.55 (1H, s, H-30), 1.71 (3H, s, H-32), 3.76 (3H, s, 7-OMe), 3.46 (1H, brs, 1-OH), 3.55 (1H, brs, 2-OH), 2.05 (3H, s, 3-OAc), 2.22 (3H, s, 6-OAc), 1.53 (3H, s, 12-OAc)；^{13}C-NMR (125 MHz, $CDCl_3$) δ: 84.0 (C-1), 75.0 (C-2), 85.4 (C-3), 44.2 (C-4), 45.2 (C-5), 70.9 (C-6), 171.7 (C-7), 83.4 (C-8), 86.9 (C-9), 47.7 (C-10), 31.9 (C-11), 67.8 (C-12), 42.7 (C-13), 153.0 (C-14), 123.5 (C-15), 163.2 (C-16), 78.4 (C-17), 14.0 (C-18), 16.1 (C-19), 120.9 (C-20), 141.7 (C-21), 109.9 (C-22), 142.5 (C-23), 39.9 (C-28), 15.1 (C-29), 77.6 (C-30), 119.2 (C-31), 16.0 (C-32), 52.8 (7-OMe), 169.4/21.2 (3-OAc), 169.0/20.6 (6-OAc), 170.1/19.5 (12-OAc)。以上波谱数据与文献报道一致[15]，故鉴定化合物 **12** 为 xyloccensin Q。

化合物 **13**：白色粉末，^{1}H-NMR (500 MHz, $CDCl_3$) δ: 5.22 (1H, s, H-3), 2.48 (1H, brs, H-5), 6.33 (1H, brs, H-6), 2.36 (1H, dd, J = 14.2, 4.3 Hz, H-11a), 2.01 (1H, m, H-11b), 4.95 (1H, dd, J = 13.3, 4.2 Hz, H-12), 6.61 (1H, s, H-15), 5.91 (1H, s, H-17), 1.60 (3H, s, H-18), 1.33 (3H, s, H-19), 7.46 (1H, s, H-21), 6.57 (1H, d, J = 1.7 Hz, H-22), 7.41 (1H, d, J = 1.7 Hz, H-23), 2.13 (1H, d, J = 10.8 Hz, H-28a), 1.84 (1H, d, J = 10.8 Hz, H-28b), 0.93 (3H, s, H-29), 5.36 (1H, s, H-30), 1.71 (3H, s, H-32), 3.75 (3H, s, 7-OMe), 3.46 (1H, brs, 1-OH), 2.19 (3H, s, 2-OAc), 2.08 (3H, s, 3-OAc), 2.22 (3H, s, 6-OAc), 1.57 (3H, s, 12-OAc)；^{13}C-NMR (125 MHz, $CDCl_3$) δ: 84.1 (C-1), 44.4 (C-2), 85.1 (C-3), 40.4 (C-4), 44.5 (C-5), 70.8 (C-6), 171.4 (C-7), 83.4 (C-8), 83.4 (C-9), 48.4 (C-10), 32.2 (C-11), 68.1 (C-12), 42.6 (C-13), 152.3 (C-14), 123.9 (C-15), 163.0 (C-16), 78.5 (C-17), 14.0 (C-18), 16.0

(C-19), 120.6 (C-20), 141.6 (C-21), 109.9 (C-22), 142.8 (C-23), 39.9 (C-28), 14.9 (C-29), 73.8 (C-30), 119.0 (C-31), 16.0 (C-32), 52.9 (7-OMe), 169.4/21.6 (3-OAc), 168.8/21.5 (3-OAc), 168.8/20.6 (6-OAc), 170.1/19.5 (12-OAc)。以上波谱数据与文献报道一致[15]，故鉴定化合物 **13** 为 xyloccensin P。

化合物 **14**：白色粉末，^{1}H-NMR (500 MHz, $CDCl_3$) δ: 3.05 (1H, m, H-2), 4.00 (1H, d, J = 5.9 Hz, H-3), 2.84 (1H, d, J = 2.5 Hz, H-5), 4.37 (1H, dd, J = 2.5, 4.5 Hz, H-6), 2.06 (1H, m, H-9), 2.17 (1H, m, H-11a), 1.52 (1H, m, H-11b), 1.69 (2H, m, H-12), 3.32 (1H, d, J = 17.0 Hz, H-15a), 2.55 (1H, d, J = 17.0 Hz, H-15b), 6.23 (1H, s, H-17), 1.01 (3H, s, H-18), 1.34 (3H, s, H-19), 7.50 (1H, s, H-21), 6.47 (1H, d, J = 1.9 Hz, H-22), 7.48 (1H, d, J = 1.9 Hz, H-23), 1.05 (3H, s, H-28), 0.95 (3H, s, H-29), 2.54 (2H, m, H-30), 3.84 (3H, s, 7-OMe), 2.60 (1H, brd, J = 4.5 Hz, 6-OH), 2.57 (1H, brs, 14-OH)；^{13}C-NMR (125 MHz, $CDCl_3$) δ: 213.4 (C-1), 48.8 (C-2), 92.8 (C-3), 37.7 (C-4), 48.3 (C-5), 70.9 (C-6), 174.8 (C-7), 85.3 (C-8), 52.5 (C-9), 50.9 (C-10), 11.7 (C-11), 28.6 (C-12), 40.1 (C-13), 74.6 (C-14), 37.3 (C-15), 169.3 (C-16), 75.9 (C-17), 15.9 (C-18), 18.2 (C-19), 121.4 (C-20), 142.0 (C-21), 109.7 (C-22), 143.3 (C-23), 29.6 (C-28), 21.3 (C-29), 48.4 (C-30), 52.6 (7-OMe)。以上波谱数据与文献报道一致[18]，故鉴定化合物 **14** 为 cedrodorin。

化合物 **15**：白色粉末。^{1}H-NMR (500 MHz, $CDCl_3$) δ: 4.00 (1H, s, H-3), 3.12 (1H, dd, J = 11.0, 2.0 Hz, H-5), 2.26 (1H, m, H-6a), 2.16 (1H, m, H-6b), 2.00 (1H, m, H-9), 2.17 (1H, m, H-11a), 1.52 (1H, m, H-11b), 1.69 (2H, m, H-12), 3.32 (1H, d, J = 17.8 Hz, H-15a), 2.55 (1H, d, J = 17.9 Hz, H-15b), 6.19 (1H, s, H-17), 1.00 (3H, s, H-18), 1.08 (3H, s, H-19), 7.56 (1H, s, H-21), 6.49 (1H, d, J = 1.9 Hz, H-22), 7.46 (1H, d, J = 1.9 Hz, H-23), 0.63 (3H, s, H-28), 1.08 (3H, s, H-29), 2.72 (1H, d, J = 11.9 Hz, H-30a), 1.97 (1H, d, J = 11.9 Hz, H-30b), 3.72 (3H, s, 7-OMe), 3.49 (1H, s, 2-OH), 1.66 (1H, s, 14-OH)；^{13}C-NMR (125 MHz, $CDCl_3$) δ: 214.4 (C-1), 80.5 (C-2), 92.7 (C-3), 37.0 (C-4), 43.4 (C-5), 32.3 (C-6), 173.9 (C-7), 85.3 (C-8), 52.1 (C-9), 50.2 (C-10), 17.7 (C-11), 28.4 (C-12), 40.0 (C-13), 74.4 (C-14), 37.3 (C-15), 169.3 (C-16), 76.0 (C-17), 15.8 (C-18), 16.4 (C-19), 120.5 (C-20), 140.7 (C-21), 109.6 (C-22), 142.8 (C-23), 18.8 (C-28), 27.9 (C-29), 48.4 (C-30), 51.7 (7-OMe)。以上波谱数据与文献报道一致[18]，故鉴定化合物 **15** 为 xylorumphiins D。

化合物 **16**：白色粉末，^{1}H-NMR (500 MHz, $CDCl_3$) δ: 1.93 (1H, m, H-1a), 1.46 (1H, m, H-1b), 2.49 (1H, ddd, J = 15.7, 9.4, 7.6 Hz, H-2a), 2.43 (1H, ddd, J = 15.7, 7.6, 4.5 Hz, H-2b), 1.38 (1H, m, H-5), 1.55 (1H, m, H-6a), 1.45 (1H, m, H-6b), 1.57 (1H, m, H-7a), 1.31 (1H, m, H-7b), 1.43 (1H, dd, J = 12.3, 2.8 Hz, H-9), 1.50 (1H, m, H-11a), 1.31 (1H, m, H-11b), 1.92 (1H, m, H-12a), 1.26 (1H, m, H-12b), 1.74 (1H, m, H-13), 1.47 (1H, m, H-15a), 1.08 (1H, m, H-15b), 1.75 (1H, m, H-16a), 1.32 (1H, m, H-16b), 1.74 (1H, m, H-17), 1.01 (3H, s, H-18), 0.95 (3H, s, H-19), 1.14 (3H, s, H-21), 1.48 (1H, d, J = 1.9 Hz, H-22), 2.07 (1H, d, J = 1.9 Hz, H-23), 5.13 (1H, m, H-24), 1.63 (3H, s, H-26), 1.69 (3H, s, H-27), 1.05 (3H, s, H-28), 1.09 (3H, s, H-29), 0.90 (3H, s, H-30)；^{13}C-NMR (125 MHz, $CDCl_3$) δ: 40.3 (C-1), 34.1 (C-2), 218.1 (C-3), 47.4 (C-4), 55.3 (C-5), 19.6 (C-6), 34.6 (C-7), 49.9 (C-8), 49.9 (C-9), 36.8 (C-10), 21.9 (C-11), 27.5 (C-12), 42.3 (C-13), 39.8 (C-14), 31.2 (C-15), 25.4 (C-16), 49.5 (C-17), 15.2 (C-18), 16.0 (C-19), 75.7 (C-20), 23.6 (C-21), 41.8 (C-22), 22.3 (C-23), 124.6 (C-24), 131.7 (C-25), 17.7 (C-26), 25.7 (C-27), 26.7 (C-28), 21.0 (C-29), 16.2 (C-30)。以上波谱数据与文献报道一致[19]，故鉴定化合物 **16** 为 hydroxydammarenone-II。

化合物 **17**：无色油状物。^{1}H-NMR (500 MHz, $CDCl_3$) δ: 7.63 (2H, dd, J = 5.0, 1.0 Hz, H-3, 6), 7.49 (2H, m, H-4, 5), 4.21 (4H, d, J = 5.2 Hz, H-1′,1″), 1.65 (2H, m, H-2′, 2″), 1.22～1.31 (16H, m, 8×CH_2), 0.81 (6H, t, J = 5.0 Hz, H-6′, 6″), 0.84 (6H, t, J = 5.0 Hz, H-8′, 8″)。^{13}C-NMR (125 MHz, $CDCl_3$) δ: 167.0 (C = O), 132.7 (C-1, 2), 130.6 (C-4, 5), 128.5 (C-3, 6), 67.7 (C-1′, 1″), 38.5 (C-2′, 2″), 30.5 (C-3′, 3″), 28.4 (C-4′, 4″), 22.9 (C-7′, 7″), 22.7 (C-5′, 5″), 13.3 (C-8′, 8″), 10.4 (C-6′, 6″)。以上波谱数据与文献报道一致[20]，故鉴定化合物 **17** 为邻苯二甲酸双 (2-乙基己基) 酯。

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