

药西瓜化学成分

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[摘要] **目的:**研究药西瓜 *Citrullus colocynthis* 中的化学成分。**方法:**利用各种色谱方法进行分离纯化,根据理化性质和 NMR, MS 等波谱技术鉴定化合物的结构。**结果:**从药西瓜乙醇提取物中分离鉴定了 9 个化合物,包括 2 个生物碱类化合物,3 个三萜类化合物,4 个三萜皂苷类化合物。分别为烟酰胺(1),尿嘧啶(2),异葫芦素 B(3),葫芦素 E(4),双氢葫芦素 E(5),葫芦素 E-2-O-β-D-吡喃葡萄糖苷(6),khekadaengoside E(7),葫芦素 K-2-O-β-D-吡喃葡萄糖苷(8),deoxocucurbitoside B(9)。**结论:**化合物 1~3 均为首次从药西瓜中分离得到。

[关键词] 药西瓜; 化学成分; 三萜; 三萜皂苷

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Chemical Constituents from *Citrullus colocynthis*

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[Abstract] **Objective:** To study the chemical constituents from *Citrullus colocynthis*. **Method:** The compounds were separated and purified from *Citrullus colocynthis* by using a various chromatographic techniques including colum chromatography over silica gel, ODS and preparative HPLC. Their structures were elucidated by spectroscopic techniques including ¹H-NMR, ¹³C-NMR, 2D-NMR, ESI-MS and HRESI-MS. **Result:** Nine compounds were isolated from this plant and their structures were identified as nicotinamide (1), uracil (2), isocucurbitacin B (3), cucurbitacin E (4), dihydro-cucurbitacin E (5), cucurbitacin E-2-O-β-D-glucopyranoside (6), khekadaengoside E (7), cucurbitacinK-2-O-β-D-glucopyranoside (8), deoxocucurbitoside B (9). **Conclusion:** Compounds 1-3 were isolated from this plant for the first time.

[Key words] *Citrullus colocynthis*; chemical constituents; triterpene; cucurbitane glycosides

药西瓜为一种生活在沙漠地区的毛茎攀缘藤本植物,该植物生长于热带及亚热带地区,在非洲、印度和阿拉伯国家和地区有广泛分布。在宋朝,该植物由丝绸之路传入我国新疆地区,如今在新疆和田河叶城地区有广泛种植。药西瓜味苦,具有清泄火热^[1],抗氧化,导泻,保肝等作用。但目前国内外少有药西瓜降血糖功效^[2]研究的文献报道,为明确药

西瓜降血糖有效成分和物质基础,作者对其进行了深入系统的化学研究。此次报道从中分离并鉴定的 9 个化合物,其中化合物 1,2 为生物碱类化合物,化合物 3~5 为三萜化合物,化合物 6~9 为三萜皂苷化合物。化合物 1~3 为首次从药西瓜中分离到。

1 材料

1200 系列高效液相色谱仪 (Agilent), SPD-

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20AD 型高效液相色谱仪(岛津), Inova-500 型核磁共振波谱仪(Vairan), 1100 系列 LC/MSD Trap-SP 型质谱仪(Agilent)。反相中压(borosilicat3.3 code no 1780)、硅胶 H、200~300 目正相硅胶(青岛海洋化工有限公司), Rp C₁₈(日本 YMC 公司), 色谱级甲醇、色谱级乙腈(百灵威化学技术有限公司)。实验室所用其他试剂均为分析纯。

药西瓜于 2010 年 11 月购于安徽亳州, 由中国医学科学院 & 北京协和医学院药物研究所马林副研究员鉴定为葫芦科 Cucurbitaceae 西瓜属植物药西瓜 *Citrullus colocynthis* 的果实, 标本存放于中国医学科学院 & 北京协和医学院药物研究所标本室。

2 提取与分离

取干燥药西瓜果实 10.0 kg, 粉碎后, 95% 乙醇回流提取 3 次, 合并提取液, 减压回收溶剂后得 2.23 kg 浸膏。取 600 g 浸膏用 10 000 mL 蒸馏水混溶, 依次用石油醚, 乙酸乙酯, 正丁醇各萃取 4 次, 得石油醚部分浸膏 43 g, 乙酸乙酯部位浸膏 81 g, 正丁醇部位浸膏 121 g。取乙酸乙酯萃取部位浸膏 40 g, 经减压硅胶色谱, 硅胶色谱, 硅胶 H 色谱, 制备 HPLC 分离纯化, 得到化合物 **3**(28.0 mg), 化合物 **4**(22.0 mg), 化合物 **5**(18.0 mg)。取正丁醇萃取部位浸膏 80 g, 经减压硅胶色谱, 反相中压色谱, 硅胶 H 色谱, SephadexLH-20 凝胶柱色谱分离纯化, 得到化合物 **1**(12.0 mg), 化合物 **2**(11.0 mg), 化合物 **6**(112.0 mg), 化合物 **7**(87.0 mg), 化合物 **8**(51.0 mg), 化合物 **9**(28.0 mg)。

3 结构鉴定

化合物 **1** 白色粉末。ESI-MS m/z 123 [M + H]⁺。¹H-NMR (400 MHz, DMSO-*d*₆) δ : 9.01 (1H, s, H-2), 8.67 (1H, dd, $J = 1.5, 1.0$ Hz, H-6), 8.20~8.18 (1H, dt, $J = 1.5, 1.5$ Hz, H-5), 8.13 (1H, br s, N-H), 7.57 (1H, br s, N-H), 7.49 (1H, dd, $J = 4.5, 5.0$ Hz, H-4)。¹³C-NMR (100 MHz, DMSO-*d*₆) δ : 166.4 (C-7), 152.0 (C-2), 148.7 (C-6), 135.2 (C-4), 129.8 (C-3), 123.6 (C-5)。以上数据与文献[3]报道一致, 故鉴定为烟酰胺(nicotinamide)。

化合物 **2** 白色粉末。ESI-MS m/z 113 [M + H]⁺。¹H-NMR (400 MHz, DMSO-*d*₆) 2 个氮氢 δ : 11.02, 10.83, 2 个双键氢 δ : 7.36 (1H, $J = 8.0$), 5.41 (1H, dd, $J = 1.5, J = 1.0$)。¹³C-NMR (100 MHz, DMSO-*d*₆) 2 个羰基碳 δ : 164.2, 151.4, 1 对双键碳 δ : 142.2, 100.1。以上数据与文献[4]报道一

致, 故鉴定为尿嘧啶(uracil)。

化合物 **3** 白色方晶。ESI-MS m/z 581 [M + Na]⁺。¹H-NMR (400 MHz, CDCl₃) δ : 0.80 (3H, s, 28-CH₃) 0.96 (3H, s, 18-CH₃), 1.18 (3H, s, 19-CH₃), 1.25 (3H, s, 29-CH₃), 1.32 (3H, s, 30-CH₃), 1.41 (3H, s, 21-CH₃), 1.53 (3H, s, 26-CH₃), 1.54 (3H, s, 27-CH₃), 1.99 (3H, s, COCH₃), 1.44 (1H, m, H-15 β), 1.89 (1H, m, H-15a), 1.96 (1H, d, $J = 12$ Hz, H-8), 2.03 (1H, m, H-7) 2.22 (1H, m, H-1 β), 2.63 (1H, d, $J = 14.8$ Hz, H-12 β), 3.10 (1H, d, $J = 14.8$ Hz, H-12a), 3.89 (1H, s, H-3), 4.34 (1H, m, H-15), 5.93 (1H, s, H-6), 6.43 (1H, s, H-23), 7.02 (1H, s, H-24)。¹³C-NMR (100 MHz, CDCl₃) δ : 210.5 (C-2), 41.2 (C-4), 138.1 (C-9), 211.7 (C-11), 50.6 (C-13), 48.3 (C-14), 78.1 (C-20), 202.3 (C-22), 79.2 (C-25), 28.0 (COCH₃), 79.2 (C-3), 121.8 (C-6), 42.7 (C-8), 32.2 (C-10), 71.2 (C-16), 58.1 (C-17), 120.2 (C-23), 152.0 (C-24), 36.2 (C-1), 23.9 (C-7), 48.5 (C-12), 45.3 (C-15), 19.8 (C-18), 20.0 (C-19), 23.9 (C-21), 25.9 (C-26), 26.3 (C-27), 24.0 (C-28), 27.7 (C-29), 18.8 (C-30)。以上数据与文献[5]报道一致, 故鉴定为异葫芦素 B(isocucurbitacin B)。

化合物 **4** 白色粉末。ESI-MS m/z 579 [M + Na]⁺。¹H-NMR (400 MHz, CDCl₃) δ : 0.99 (3H, s, 18-CH₃), 1.03 (3H, s, 19-CH₃), 1.25 (3H, s, 29-CH₃), 1.36 (3H, s, 28-CH₃), 1.38 (3H, s, 30-CH₃), 1.43 (3H, s, 21-CH₃), 1.54 (3H, s, 26-CH₃), 1.57 (3H, s, 27-CH₃), 2.01 (3H, s, COCH₃), 3.49 (1H, br s, $J = 5.6$ Hz, 10-CH₃), 5.76 (1H, t, $J = 2.0$ Hz, H-6), 5.95 (2H, d, $J = 2.4$ Hz, H-2), 6.46 (1H, d, $J = 15.6$ Hz, H-23), 7.06 (1H, d, $J = 15.6$ Hz, H-24)。¹³C-NMR (100 MHz, CDCl₃) δ : 18.4 (C-19), 19.9 (C-18), 20.2 (-COCH₃), 20.3 (C-30), 22.0 (C-28), 23.6 (C-7), 24.0 (C-21), 26.0 (C-26), 26.5 (C-27), 28.0 (C-29), 34.8 (C-10), 41.7 (C-8), 45.6 (C-15), 47.6 (C-4), 48.2 (C-13, 14), 48.9 (C-12), 50.8 (C-9), 58.3 (C-17), 71.4 (C-16), 78.2 (C-25), 79.4 (C-20), 114.8 (C-1), 120.4 (C-6), 120.8 (C-23), 136.8 (C-5), 144.6 (C-2), 152.0 (C-24), 170.3 (-COCH₃), 198.7 (C-3), 202.5 (C-22), 212.8 (C-11)。以上数据与文献[5]报道一致, 故鉴定为葫芦素 E(cucurbitacin E)。

化合物 5 白色方晶。ESI-MS m/z 581 [M + Na]⁺。¹H-NMR (400 MHz, CDCl₃) δ : 0.97 (3H, s, 18-CH₃), 1.01 (3H, s, 19-CH₃), 1.25 (3H, s, 29-CH₃), 1.34 (3H, s, 28-CH₃), 1.40 (3H, s, 30-CH₃), 1.42 (3H, s, 21-CH₃), 1.44 (3H, s, 26-CH₃), 1.46 (3H, s, 27-CH₃), 1.94 (3H, s, COCH₃), 1.43 (1H, m, H-15a), 1.84 (1H, m, H-15b), 2.05 (1H, m, H-8), 2.04 (1H, m, H-7a), 2.38 (1H, m, H-7b), 2.51 (1H, m, H-16), 2.72 (1H, d, $J = 14.5$ Hz, H-12b), 2.52 (1H, m, H-23a), 2.80 (1H, m, H-23b), 3.22 (1H, d, $J = 14.5$ Hz, H-12a), 4.34 (1H, m, H-16), 5.75 (1H, br s, H-6)。¹³C-NMR (100 MHz, CDCl₃) δ : 18.3 (C-30), 19.9 (C-19), 20.2 (C-18), 20.3 (C-29), 22.5 (-COCH₃), 23.7 (C-7), 24.6 (C-21), 26.0 (C-26), 26.2 (C-27), 28.0 (C-28), 30.8 (C-23), 34.8 (C-10, 24), 41.7 (C-8), 45.8 (C-15), 47.6 (C-4), 48.5 (C-13), 48.9 (C-9), 49.0 (C-12), 50.8 (C-14), 57.9 (C-17), 71.1 (C-16), 79.0 (C-20), 81.4 (C-25), 114.9 (C-1), 120.8 (C-6), 136.9 (C-5), 144.7 (C-2), 170.5 (-COCH₃), 198.7 (C-3), 212.9 (C-11), 214.0 (C-22)。以上数据与文献 [6] 报道一致, 故鉴定为双氢葫芦素 E (dihydrocucurbitacin E)。

化合物 6 白色方晶。ESI-MS m/z 741 [M + Na]⁺。¹H-NMR (400 MHz, CD₃OD) δ : 0.94 (3H, s, 18-CH₃), 0.96 (3H, s, 30-CH₃), 1.20 (3H, s, 28-CH₃), 1.26 (3H, s, 29-CH₃), 1.36 (3H, s, 21-CH₃), 1.40 (3H, s, 19-CH₃), 1.49 (1H, m, H-15b), 1.52 (3H, s, 27-CH₃), 1.54 (3H, s, 26-CH₃), 1.85 (1H, m, H-15a), 1.99 (3H, s, -COCH₃), 2.18 (1H, m, H-8), 2.18 (1H, m, H-7), 2.35 (1H, m, H-7a), 2.47 (1H, d, $J = 6.4$ Hz, H-12b), 3.43 (1H, d, $J = 6.4$ Hz, H-12a), 3.43 (1H, m, H-5'), 3.47 (1H, m, H-4'), 3.24 (1H, m, H-2'), 3.47 (1H, m, H-3'), 3.60 (1H, br s, 10-H), 4.09 (1H, dd, $J = 12$ Hz, H-6a'), 3.89 (1H, dd, $J = 12$ Hz, H-6b'), 4.66 (1H, t, $J = 15.2$ Hz, H-1'), 5.73 (1H, br s, H-6), 6.12 (1H, br s, H-1), 6.50 (1H, d, $J = 15.6$ Hz, H-23), 7.04 (1H, d, $J = 15.6$ Hz, H-24); ¹³C-NMR (100 MHz, CD₃OD) δ : 19.0 (C-19), 20.8 (C-18), 21.0 (C-28, 30), 22.1 (-COCH₃), 24.9 (C-7), 25.7 (C-21), 26.7 (C-26), 27.1 (C-27), 28.6 (C-29), 36.7 (C-10), 43.4 (C-8), 46.9 (C-15), 49.9 (C-12), 50.1 (C-

9), 50.3 (C-13), 51.9 (C-14), 60.4 (C-17), 62.2 (C-6'), 70.9 (C-4'), 72.1 (C-16), 74.6 (C-2'), 77.8 (C-5'), 78.4 (C-3'), 80.5 (C-20), 81.3 (C-25), 101.4 (C-1'), 122.6 (C-1), 122.9 (C-23), 123.8 (C-6), 137.8 (C-5), 147.5 (C-2), 151.8 (C-24), 172.1 (-COCH₃), 200.0 (C-3), 205.5 (C-22), 216.8 (C-11)。以上数据与文献 [7] 报道一致, 故鉴定为葫芦素 E-2-O- β -D-吡喃葡萄糖苷 (cucurbitacin E-2-O- β -D-glucopyranoside)。

化合物 7 白色方晶。ESI-MS m/z 679 [M + H]⁺。¹H-NMR (400 MHz, CD₃OD) δ : 0.87 (3H, s, 30-CH₃), 0.92 (3H, s, 28-CH₃), 1.10 (3H, s, 29-CH₃), 1.18 (3H, s, 27-CH₃), 1.20 (3H, s, 26-CH₃), 1.22 (3H, s, 18-CH₃), 1.30 (3H, s, 19-CH₃), 1.32 (3H, s, 21-CH₃), 1.96 (2H, d, 8.5 Hz, 15-CH₂), 2.00 (1H, m, H-8), 2.30 (2H, m, H-7), 2.49 (1H, d, 7.5 Hz, H-17), 3.28 (1H, m, 5-H), 3.30 (1H, m, 2-H), 3.33 (1H, m, 3-H), 3.41 (1H, d, 12-H), 3.59 (1H, br s, 10-H), 3.76 (1H, dd, $J = 12$ Hz, H-6a), 4.04 (1H, dd, $J = 12$ Hz, H-6b), 5.84 (1H, s, H-6), 6.12 (1H, d, $J = 2.6$ Hz, H-1)。¹³C-NMR (100 MHz, CD₃OD) δ : 123.9 (C-1), 147.5 (C-2), 200.1 (C-3), 50.6 (C-4), 137.7 (C-5), 122.6 (C-6), 24.9 (C-7), 43.4 (C-8), 50.5 (C-9), 38.4 (C-10), 216.7 (C-11), 50.1 (C-12), 50.4 (C-13), 50.2 (C-14), 47.1 (C-15), 71.7 (C-16), 59.9 (C-17), 20.9 (C-18), 20.8 (C-19), 81.0 (C-20), 25.7 (C-21), 205.3 (C-22), 121.5 (C-23), 155.6 (C-24), 71.9 (C-25), 29.5 (C-26), 29.1 (C-27), 28.5 (C-28), 20.9 (C-29), 19.0 (C-30), 101.4 (C-1'), 71.9 (C-2'), 78.4 (C-3'), 70.9 (C-4'), 80.2 (C-5'), 62.2 (C-6')。以上数据与文献 [8] 报道一致, 故鉴定为 khekadaengosid E。

化合物 8 白色方晶。ESI-MS m/z 717 [M + Na]⁺。¹H-NMR (400 MHz, CD₃OD) δ : 0.95 (3H, s, 18-CH₃), 1.01 (3H, s, 19-CH₃), 1.16 (3H, s, 26-CH₃), 1.18 (3H, s, 27-CH₃), 1.26 (3H, s, 29-CH₃), 1.29 (3H, s, 28-CH₃), 1.39 (3H, s, 21-CH₃), 1.40 (3H, s, 30-CH₃), 1 s, 21-CH₃), 1.40 (3H, s, 30-CH₃), 1.90 (1H, m, H-15b), 2.08 (1H, m, H-8), 2.11 (1H, m, H-7b), 2.42 (1H, m, H-7b), 2.55 (1H, d, $J = 7.5$ Hz, H-17), 2.68 (1H, d, $J = 7.5$ Hz, H-12b), 2.92 (1H, m, H-23b), 2.96 (1H, m, H-23a), 3.29 (1H, m, 5'-H), 3.39 (1H, m, H-12a), 3.41 (1H,

m,2'-H), 3.43 (1H, m, 3'-H), 3.55 (1H, m, 4'-H), 3.73 (1H, br s, H-10), 3.92 (1H, m, 6b'-H), 3.99 (1H, m, H-24), 4.01 (1H, m, 6a'-H), 4.50 (1H, m, H-16), 4.69 (1H, d, $J = 7$ Hz, 1'-H), 5.88 (1H, br s, H-6), 6.15 (1H, br s, H-1)。¹³C-NMR (100 MHz, CD₃OD) δ : 123.5 (C-1), 146.7 (C-2), 199.4 (C-3), 50.6 (C-4), 137.1 (C-5), 122.6 (C-6), 24.9 (C-7), 42.8 (C-8), 50.2 (C-9), 36.0 (C-10), 216.7 (C-11), 50.0 (C-12), 51.4 (C-13), 49.2 (C-14), 46.1 (C-15), 71.7 (C-16), 58.9 (C-17), 20.9 (C-18), 20.8 (C-19), 81.0 (C-20), 24.9 (C-21), 215.3 (C-22), 40.3 (C-23), 74.7 (C-24), 73.9 (C-25), 24.6 (C-26), 25.7 (C-27), 28.5 (C-28), 20.9 (C-29), 18.4 (C-30), 100.9 (C-1'), 74.9 (C-2'), 77.4 (C-3'), 70.9 (C-4'), 77.9 (C-5'), 62.2 (C-6')。以上数据与文献[9]报道一致,故鉴定为葫芦素 K-2-O- β -D-吡喃葡萄糖苷 (cucurbitacinK-2-O- β -D-glucopyranoside)。

化合物 9 白色粉末。ESI-MS m/z 813 [M + Na]⁺。¹H-NMR (400 MHz, CD₃OD) δ : 0.92 (3H, s, 18-CH₃), 0.94 (3H, s, 19-CH₃), 1.11 (3H, s, 21-CH₃), 1.20 (3H, s, 30-CH₃), 1.24 (3H, s, 29-CH₃), 1.27 (3H, s, 28-CH₃), 1.61 (3H, s, 26-CH₃), 1.67 (3H, s, 27-CH₃), 2.33 (1H, d, $J = 8$ Hz, H-17), 2.43 (1H, d, $J = 15$ Hz, H-12b), 3.18 (1H, d, $J = 15$ Hz, H-12a), 5.07 (1H, d, $J = 8$ Hz, H-24), 4.45 (1H, m, H-16), 4.55 (1H, m, H-23), 5.76 (1H, br s, H-6), 5.96 (1H, m, H-1)。¹³C-NMR (100 MHz, CD₃OD) δ : 123.7 (C-1), 147.7 (C-2), 198.9 (C-3), 46.9 (C-4), 138.1 (C-5), 122.4 (C-6), 24.9 (C-7), 42.6 (C-8), 49.1 (C-9), 36.6 (C-10), 216.6 (C-11), 48.7 (C-12), 50.6 (C-13), 51.0 (C-14), 46.9 (C-15), 71.8 (C-16), 56.9 (C-17), 21.0 (C-18), 18.7 (C-19), 77.8 (C-20), 26.0 (C-21), 42.3 (C-22), 74.8 (C-23), 126.7 (C-24), 138.1 (C-25), 30.1 (C-26), 29.3 (C-27), 20.4 (C-28), 28.1 (C-29), 20.4 (C-30), 99.9 (C-1'), 79.2 (C-2'), 71.8 (C-3'), 71.2 (C-4'), 72.4 (C-5'), 62.3 (C-6') 102.5 (C-1''), 72.7 (C-2''), 72.4 (C-3''), 74.8 (C-

4''), 70.2 (C-5''), 18.1 (C-6'')。以上数据与文献[10]报道一致,故鉴定为 deoxocucurbitoside B。

[参考文献]

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