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姜状三七根茎的皂苷类化学成分研究

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摘要：为了解姜状三七(*Panax zingiberensis*)根茎的皂苷类化学成分，采用正相硅胶、反相硅胶和凝胶等色谱方法从其根茎的乙醇提取物中得到 9 个皂苷类化合物，根据理化性质和波谱数据，其结构分别鉴定为人参皂苷 SL_1 (**1**)、人参皂苷 Rh_1 (**2**)、三七皂苷 R_8 (**3**)、竹节参皂苷 IVa (**4**)、越南人参皂苷 R_{10} (**5**)、人参皂苷 Rg_1 (**6**)、菠菜皂苷 A 28-*O*-*β*-D-葡萄糖苷 (**7**)、齐墩果酸 28-*O*-*β*-D-葡萄糖苷 (**8**)和姜状三七苷 R_1 (**9**)。化合物 **1**、**3**、**5**、**7** 和 **8** 为首次从该植物中分离得到, 其中化合物 **5** 为奥克梯隆醇型皂苷，此类皂苷在该植物中未见报道。

关键词：五加科；姜状三七；根茎；皂苷类

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Saponins from Rhizomes of *Panax zingiberensis*

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Abstract: To investigate the chemical constituents from rhizomes of *Panax zingiberensis*, nine compounds were isolated and purified by methods of column chromatography on silica gel and Sephadex LH-20. Based on physical and chemical properties and spectral data,their structures were identified as ginsenoside SL_1 (**1**), ginsenoside Rh_1 (2), notoginsenoside R_8 (**3**), chikusetsusaponin IVa (**4**), vina-ginsenoside 10 (**5**), ginsenoside Rg_1 (**6**), spinasaponin A 28-*O*-*β*-D-glucopyranoside (**7**), oleanolic acid 28-*O*-*β*-D-glucopyranoside (**8**) and zingbroside R_1 (**9**). Compounds **1**, **3**, **5**, **7**, and **8** were isolated from *P. zingiberensis* for the first time. Compound **5** belongs to the ocotillol-type saponin, which was not previously reported from this species.

Key words: Araliaceae; *Panax zingiberensis*; Rhizome; Saponin

姜状三七(*Panax zingiberensis*)为五加科(Araliaceae)人参属植物，根茎入药，主要用于治疗跌打损伤、劳虚咳嗽、外伤出血及贫血等症, 也作三七代用品。国外主要分布于尼泊尔中部、不丹高海拔地区、缅甸东枝、掸邦等地[1–4]，国内分布于云南东南部的马关、砚山等地。野生资源分布狭窄及过度采挖，导致该物种于1997年被国际自然保护联盟列为濒危种。由于材料的难以采集导致关于该植物的研究较少。目前只从该植物中分离鉴定了三萜皂苷类化合物不到10个，有人参二醇型、人参三醇型和齐墩果烷型，且其生物活性的研究也鲜见报道[5–7]。本课题组在前期研究[8]的基础上对其化学成分进行了系统研究，从中分离鉴定了 9 个皂苷类化合物，为寻找活性物质提供了化学基础，也为姜状三七植物资源的合理开发和利用奠定理论基础。

1 材料和方法

1.1 材料

材料购自云南省普洱市镇沅县九甲镇三台村

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林下经济种植示范基地，经云南农业大学张广辉教授鉴定为五加科人参属植物姜状三七(*Panax zingiberensis*)，标本存放于云南农业大学西南中药材种质创新与利用国家地方联合工程研究中心。

1.2 仪器和试剂

API Qstar Pulsa LC/TOF 质谱仪，Bruker AM-400、DRX-500 和 AVANCE III-600 MHz 超导核磁共振仪(瑞士 Bruker 公司)，TMS 为内标，化学位移 δ 用 ppm 表示，耦合常数 J 用 Hz 表示；中药高速粉碎机(浙江永历制药机械有限公司)；旋转蒸发仪(东京理化仪器有限公司)；超声波清洗机(上海声源超声波仪器有限责任公司)；柱层析硅胶和硅胶 G 薄层板(青岛海洋化工厂)；AB-8 (大孔树脂天津波鸿树脂科技有限公司)；反相硅胶板和柱层析用 RP-18 (德国 Merk 公司)；Sephadex LH-20 (Amer- sham Pharmacia Biotech 公司)；显色剂(5%硫酸乙醇溶液)，甲醇、氯仿、丙酮和乙酸乙酯为工业级重蒸后使用，正丁醇、冰乙酸为分析纯，乙腈为色谱纯，水为哇哈哈纯净水。

1.3 提取和分离

姜状三七根茎粗粉 2.8 kg，加 8 倍量 70%甲醇溶液常温浸泡 3 d，超声提取 3 次，提取时间分别为2、1.5 和 1.5 h，合并提取液回收甲醇至无醇味，药液分次过已预处理好的 AB-8 大孔树脂柱(2 kg)，以蒸馏水洗脱至 Molish 反应为阴性，再以 80%甲醇(共 8 L)洗脱，收集甲醇洗脱液，回收甲醇，浓缩干燥得姜状三七总皂苷(440 g)。

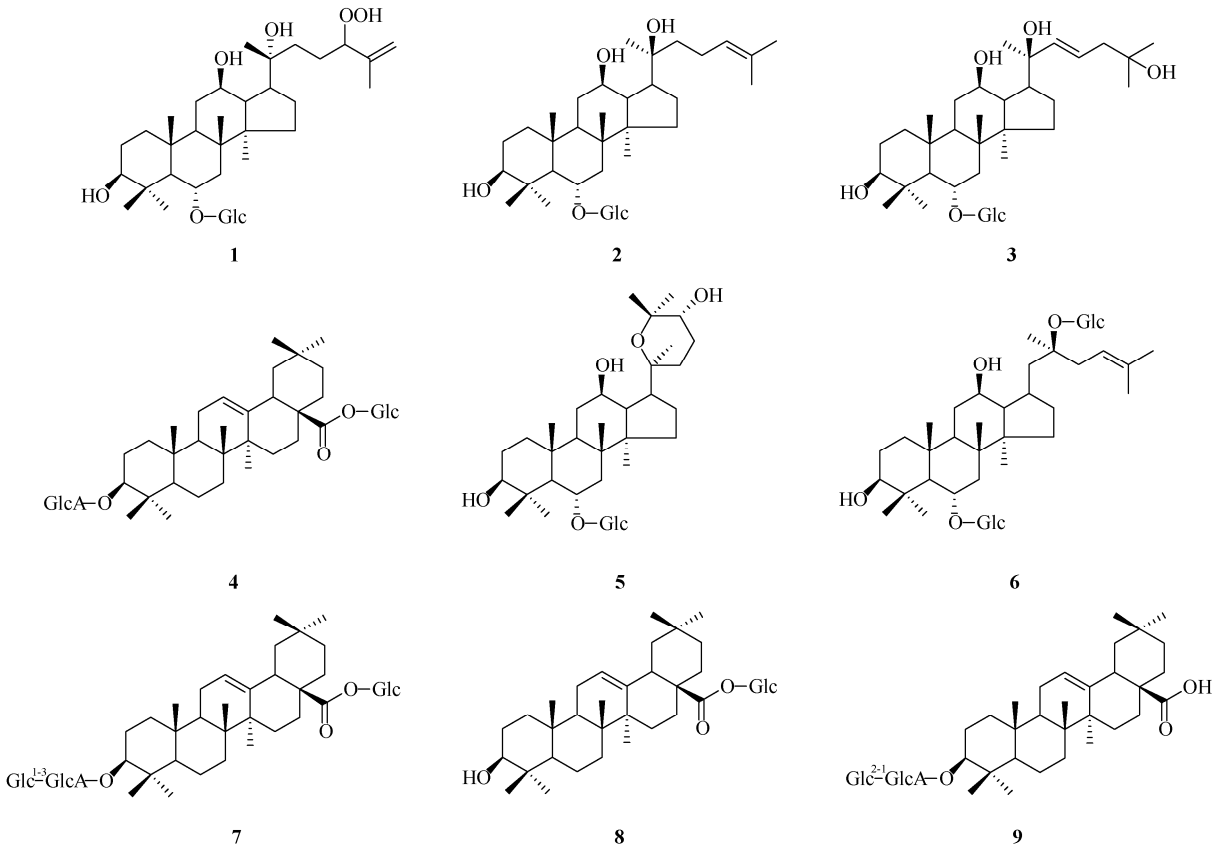
将总皂苷吸附于 1.0 kg 硅胶上进行柱层析，流动相以氯仿-甲醇-水(10∶1∶0~0∶1∶1)梯度洗脱，收集洗脱液，每份 0.5 L，共收集 292 份，薄层鉴别合并洗脱液得 Fr1~Fr8 组分。组分 Fr 4 (150 g)上硅胶柱色谱，用氯仿-甲醇-水溶剂系统进行梯度洗脱，得到 3 个馏分 Fr 4.1~Fr 4.3。Fr 4.1 (20 g)经硅胶柱色谱，以氯仿-甲醇-水(9∶1∶0.2~7∶3∶0.5)梯度洗脱，再以 Sephadex LH-20 (甲醇)纯化，得到化合物 **1** (2 mg)、**2** (8 mg)和 **3** (246 mg)。Fr 4.2 (50 g)经反复硅胶柱层析(氯仿-乙酸乙酯-甲醇-水；氯仿-甲醇-水)梯度洗脱，得到化合物 **4** (6 mg)、**5** (10 mg)、**6** (10 mg)和 **7** (2 g)。Fr 4.3 (30 g)过 Rp-18 柱色谱，以水-甲醇(9∶1~0∶1)梯度洗脱，再以 Sephadex LH- 20 (甲醇)纯化，得到化合物 **8** (6 mg)和 **9** (10 mg) (图 1)。

1.4 结构鉴定

化合物 1　白色结晶，ESI-MS *m/z*: 693 $[M + Na]^+$; ^{1}H NMR (400 MHz, CD_3OD): δ 4.34 (1H, d, J = 7.8 Hz, H′-6), 1.76, 1.32, 1.12, 1.09, 1.00, 0.99, 0.94 (each 3H, s, CH_3×7); ^{13}C NMR (125 MHz, CD_3OD): δ 40.5 (C-1), 27.5 (C-2), 79.1 (C-3), 40.4 (C-4), 61.8 (C-5), 79.9 (C-6), 45.3 (C-7), 41.8 (C-8), 50.9 (C-9), 40.2 (C-10), 32.0 (C-11), 71.7 (C-12), 49.5 (C-13), 52.5 (C-14), 31.9 (C-15), 27.4 (C-16), 52.5 (C-17), 17.9 (C-18), 17.5 (C-19), 74.2 (C-20), 22.6 (C-21), 40.4 (C-22), 26.4 (C-23), 91.2 (C-24), 146.2 (C-25), 113.9 (C-26), 17.8 (C-27), 31.4 (C-28), 16.8 (C-29), 17.3 (C-30), 105.5 (C-1′), 75.5 (C-2′), 80.9 (C-3′), 72.0 (C-4′), 77.7 (C-5′), 62.9 (C-6′)。以上数据与文献[9]报道基本一致，故鉴定为人参皂苷 SL_1。

化合物 2　白色粉末, ESI-MS *m/z*: 661 $[M + Na]^+$; ^{1}H NMR (400 MHz, CD_3OD): δ 4.38 (1H, d, J = 7.8 Hz, H-1′), 1.70, 1.64, 1.34, 1.17, 1.11, 1.02, 1.01, 0.96 (each 3H, s, CH_3×8); ^{13}C NMR (100 MHz, CD_3OD): δ 40.2 (C-1), 27.6 (C-2), 79.9 (C-3), 40.5 (C-4), 61.8 (C-5), 77.6 (C-6), 45.3 (C-7), 41.8 (C-8), 50.9 (C-9), 40.4 (C-10), 31.9 (C-11), 71.7 (C-12), 48.5 (C-13), 52.5 (C-14), 31.9 (C-15), 27.4 (C-16), 55.1 (C-17), 17.8 (C-18), 17.9 (C-19), 74.5 (C-20), 26.6 (C-21), 36.3 (C-22), 23.3 (C-23), 126.2 (C-24), 132.1 (C-25), 26.0 (C-26), 17.6 (C-27), 31.4 (C-28), 16.2 (C-29), 17.1 (C-30), 105.5 (C-1′), 75.5 (C-2′), 81.8 (C-3′), 71.7 (C-4′), 79.0 (C-5′), 62.9 (C-6′)。以上数据与文献[10]报道基本一致，故鉴定为人参皂苷 Rh_1。

化合物 3　白色粉末, ESI-MS *m/z*: 653 $[M - H]^-$; ^{1}H NMR (400 MHz, C_5D_5N): δ 5.07 (1H, d, J = 7.8 Hz, H-1′), 2.06, 1.63, 1.56, 1.55, 1.39, 1.26, 1.06, 0.80 (each 3H, s, CH_3×8); ^{13}C NMR (100 MHz, C_5D_5N): δ 39.7 (C-1), 28.0 (C-2), 78.6 (C-3), 40.1 (C-4), 61.5 (C-5), 80.1 (C-6), 45.2 (C-7), 41.1 (C-8), 50.2 (C-9), 39.4 (C-10), 32.2 (C-11), 71.1 (C-12), 48.5 (C-13), 51.7 (C-14), 31.2 (C-15), 26.8 (C-16), 54.2 (C-17), 17.4 (C-18), 17.8 (C-19), 73.3 (C-20), 27.7 (C-21), 137.6 (C-22), 127.4 (C-23), 40.4 (C-24), 81.3 (C-25), 25.2 (C-26), 25.3 (C-27), 31.8 (C-28), 16.4 (C-29), 16.8 (C-30), 106.0 (C-1′), 75.5 (C-2′), 79.7 (C-3′), 71.8 (C-4′), 78.3 (C-5′), 63.1 (C-6′)。以上数据与文献[11]报道基本一致，故鉴定为三七皂苷 R_8。



Glc: *β*-D-glucopyranosy GlcA: *β*-D-glucuronopyranosyl

图 1 化合物 **1**~**9** 的结构

Fig. 1 Structures of compounds **1**–**9**

化合物 4 白色粉末, ESI-MS *m/z*: 793 [M – H]$^-$; ^{1}H NMR (400 MHz, CD_3OD): *δ* 6.53 (1H, d, *J* = 7.6 Hz, H-1′), 4.82 (1H, d, *J* = 7.2 Hz, H-1″), 1.15, 1.04, 0.94, 0.93, 0.90, 0.83, 0.796, 0.80 (each 3H, s, CH_3×7); ^{13}C NMR (100 MHz, CD_3OD): *δ* 40.2 (C-1), 26.4 (C-2), 90.8 (C-3), 40.0 (C-4), 57.0 (C-5), 19.5 (C-6), 33.5 (C-7), 40.8 (C-8), 49.0 (C-9), 37.8 (C-10), 24.7 (C-11), 123.8 (C-12), 144.8 (C-13), 43.0 (C-14), 28.7 (C-15), 24.1 (C-16), 48.0 (C-17), 42.9 (C-18), 47.4 (C-19), 31.6 (C-20), 35.0 (C-21), 34.1 (C-22), 28.6 (C-23), 17.0 (C-24), 16.1 (C-25), 17.8 (C-26), 26.4 (C-27), 178.0 (C-28), 33.5 (C-29), 24.0 (C-30), 106.7 (3-Glc, C-1′), 75.5 (C-2′), 78.0 (C-3′), 73.7 (C-4′), 75.5 (C-5′), 176.6 (C-6′), 95.7 (28-Glc, C-1″), 73.9 (C-2″), 78.6 (C-3″), 71.1 (C-4″), 78.7 (C-5″), 62.5 (C-6″)。以上数据与文献[12]报道基本一致，故鉴定为竹节参皂苷 IVa。

化合物 5 白色粉末, ESI-MS *m/z*: 653 [M – H]$^-$; ^{1}H NMR (400 MHz, C_5D_5N): *δ* 5.03 (1H, d, *J* = 7.8 Hz, H-1′), 2.06, 1.68, 1.60, 1.52, 1.31, 1.19, 1.03, 0.80 (each 3H, s, CH_3×7); ^{13}C NMR (100 MHz, C_5D_5N): *δ* 39.5 (C-1), 28.1 (C-2), 78.7 (C-3), 40.5 (C-4), 61.6 (C-5), 78.3 (C-6), 45.1 (C-7), 41.2 (C-8), 50.2 (C-9), 39.7 (C-10), 32.2 (C-11), 70.7 (C-12), 48.9 (C-13), 52.4 (C-14), 31.8 (C-15), 28.0 (C-16), 52.1 (C-17), 17.6 (C-18), 17.9 (C-19), 78.1 (C-20), 26.4 (C-21), 27.8 (C-22), 25.9 (C-23), 74.5 (C-24), 78.7 (C-25), 23.3 (C-26), 30.1 (C-27), 31.8 (C-28), 16.4 (C-29), 17.3 (C-30), 106.1 (3-Glc, C-1′), 75.7 (C-2′), 80.3 (C-3′), 71.9 (C-4′), 79.7 (C-5′), 63.1 (C-6′)。以上数据与文献[13]报道基本一致，故鉴定为越南人参皂苷 R_{10}。

化合物6 白色粉末, ESI-MS *m/z*: 799 [M – H]$^-$; ^{1}H NMR (500 MHz, CD_3OD): *δ* 5.15 (1H, d, *J* = 7.8 Hz, H-1″), 4.98 (1H, d, *J* = 7.8 Hz, H-1′), 2.03, 1.58, 1.56, 1.56, 1.55, 1.11, 0.98, 0.75 (each 3H, s, CH_3×8); ^{13}C NMR (125 MHz, CD_3OD): *δ* 39.5 (C-1), 28.0 (C-2), 78.7 (C-3), 40.5 (C-4), 61.5 (C-5), 78.3 (C-6),

45.2 (C-7), 41.2 (C-8), 50.1 (C-9), 39.7 (C-10), 31.0 (C-11), 70.4 (C-12), 49.2 (C-13), 51.5 (C-14), 30.8 (C-15), 26.7 (C-16), 51.7 (C-17), 17.7 (C-18), 17.7 (C-19), 83.4 (C-20), 22.5 (C-21), 36.1 (C-22), 23.4 (C-23), 126.0 (C-24), 131.1 (C-25), 17.9 (C-26), 25.9 (C-27), 31.9 (C-28), 17.2 (C-29), 16.2 (C-30), 106.1 (3-Glc, C-1′), 75.5 (C-2′), 80.3 (C-3′), 71.8 (C-4′), 80.0 (C-5′), 63.1 (C-6′), 98.3 (20-Glc, C-1″), 75.2 (C-2″), 79.3 (C-3″), 71.6 (C-4″), 78.4 (C-5″), 62.8 (C-6″)。以上数据与文献[14]报道基本一致，鉴定为人参皂苷Rg_1。

化合物 7　白色粉末, ESI-MS *m/z*: 955 [M − H]$^-$; ^{1}H NMR (400 MHz, CD_3OD): δ 5.37 (1H, d, J = 8.0 Hz, H-12), 1.15, 1.04, 0.94, 0.93, 0.90, 0.83, 0.79 (each 3H, s, $CH_3\times7$); ^{13}C NMR (100 MHz, CD_3OD): δ 39.8 (C-1), 27.0 (C-2), 90.8 (C-3), 40.3 (C-4), 57.0 (C-5), 19.0 (C-6), 34.0 (C-7), 40.7 (C-8), 48.4 (C-9), 37.5 (C-10), 23.9 (C-11), 123.8 (C-12), 144.8 (C-13), 41.9 (C-14), 28.9 (C-15), 23.5 (C-16), 47.5 (C-17), 42.2 (C-18), 46.4 (C-19), 32.0 (C-20), 33.5 (C-21), 33.5 (C-22), 28.5 (C-23), 17.7 (C-24), 16.0 (C-25), 17.7 (C-26), 26.3 (C-27), 178.1 (C-28), 33.5 (C-29), 24.0 (C-30), 109.4 (3-Glc A, C-1′), 73.9 (C-2′), 86.4 (C-3′), 71.1 (C-4′), 76.7 (C-5′), 176.2 (C-6′), 106.8 (20-Glc, C-1″), 75.4 (C-2″), 76.6 (C-3″), 76.5 (C-4″), 78.7 (C-5″), 63.2 (C-6″), 95.7 (28-Glc, C-1‴), 73.9 (C-2‴), 82.9 (C-3‴), 71.1 (C-4‴), 78.7 (C-5‴), 62.4 (C-6‴)。以上数据与文献[12,15]报道基本一致, 鉴定为菠棱皂苷 A 28-*O*-葡萄糖苷。

化合物 8　白色粉末, ESI-MS *m/z*: 617 [M − H]$^-$; ^{1}H NMR (400 MHz, C_5D_5N): δ 6.32 (1H, d, J = 6.5 Hz, H-1′), 1.23, 1.22, 1.12, 1.02, 0.90, 0.89, 0.85 (each 3H, s, $CH_3\times7$); ^{13}C NMR (100 MHz, C_5D_5N): δ 39.1 (C-1), 28.4 (C-2), 78.3 (C-3), 40.4 (C-4), 55.9 (C-5), 18.9 (C-6), 33.3 (C-7), 39.5 (C-8), 48.2 (C-9), 39.7 (C-10), 23.9 (C-11), 123.0 (C-12), 144.3 (C-13), 41.9 (C-14), 28.9 (C-15), 23.5 (C-16), 47.1 (C-17), 42.2 (C-18), 46.3 (C-19), 30.9 (C-20), 34.1 (C-21), 32.6 (C-22), 30.9 (C-23), 15.7 (C-24), 16.7 (C-25), 17.6 (C-26), 26.2 (C-27), 176.7 (C-28), 23.8 (C-29), 33.2 (C-30), 95.9 (28-Glc, C-1′), 79.5 (C-2′), 79.0 (C-3′), 71.2 (C-4′), 74.2 (C-5′), 62.3 (C-6′)。以上数据与文献[16]报道数据一致, 故鉴定为齐墩果酸 28-*O*-*β*-D-葡萄糖苷。

化合物 9　白色粉末, ESI-MS *m/z*: 793 [M − H]$^-$; ^{1}H NMR (500 MHz, C_5D_5N): δ 5.33 (1H, d, J = 7.7 Hz, H-1″), 4.91 (1H, d, J = 7.3 Hz, H-1′), 1.24, 1.22, 1.03, 0.95, 0.92, 0.91, 0.66 (each 3H, s, $CH_3\times7$); ^{13}C NMR (125 MHz, C_5D_5N): δ 38.6 (C-1), 26.6 (C-2), 89.3 (C-3), 39.7 (C-4), 55.8 (C-5), 18.5 (C-6), 33.4 (C-7), 39.6 (C-8), 48.0 (C-9), 36.9 (C-10), 23.7 (C-11), 122.6 (C-12), 144.9 (C-13), 42.0 (C-14), 28.3(C-15), 23.8 (C-16), 46.7 (C-17), 42.1 (C-18), 46.5 (C-19), 31.0 (C-20), 34.2 (C-21), 33.2 (C-22), 28.2 (C-23), 15.5 (C-24), 16.8 (C-25), 17.4 (C-26), 26.2 (C-27), 180.3 (C-28), 23.8 (C-29), 33.2 (C-30), 106.0 (3-GluUA C-1′), 82.8 (C-2′), 78.5 (C-3′), 73.7 (C-4′), 77.6 (C-5′), 174.9 (C-6′), 105.3 (C-1″), 77.9 (C-2″), 78.5 (C-3″), 71.6 (C-4″), 77.2 (C-5″), 62.6 (C-6″)。以上数据与文献[5]报道一致，故鉴定为姜状三七苷 R_1。

2 结果和讨论

对姜状三七根茎中的化学成分进行了研究, 从 70%甲醇提取物中分离鉴定了 9 个皂苷类化合物, 分别是人参皂苷 SL_1 (**1**)、人参皂苷 Rh_1 (**2**)、三七皂苷 R_8 (**3**)、竹节参皂苷 IVa (**4**)、越南人参皂苷 R_{10} (**5**)、人参皂苷 Rg_1 (**6**)、菠棱皂苷 A 28-*O*-葡萄糖苷 (**7**)、齐墩果酸 28-*O*-*β*-D-葡萄糖苷 (**8**)和姜状三七苷 R_1 (**9**)。化合物 **1**、**3**、**5**、**7** 和 **8** 为首次从姜状三七中分离得到。化合物 **1**~**3** 和 **6** 为原人参三醇型皂苷, 化合物 **4**、**7**~**9** 为齐墩果烷型皂苷，化合物 **5** 为奥克梯隆醇型人参皂苷，此类型皂苷为首次从姜状三七中得到。

人参皂苷 Rg_1 (**6**)是中药人参、三七等人参属药材的主要活性成分之一，可通过抗氧化作用维持细胞内 Ca^{2+}的稳态达到保护心肌细胞的作用[17]; 对多种动物中枢神经系统疾病模型具有治疗作用，主要是通过 NMDA 受体、MAPK、MEK- ERK1/2-PI3K、Ca^{2+}-CaM-CaMK II 等信号通路进行调控，作用于皮层、海马、纹状体、下丘脑, 影响神经递质的合成与释放，促进神经细胞生长、神经突触的发生，保护神经元，促进神经干细胞分化等[18]; 还可明显抑制人白血病 TF-1 细胞的增殖并促进其凋亡，辅助 T 细胞免疫活性增加，对小鼠免疫性肝损伤也有一定

的保护作用[19]。此外, 人参皂苷 Rg_1 还有抗肿瘤, 降血糖、降血脂，抗骨质疏松和抗炎等作用[20]。

竹节参苷IVa (**4**)也具有多种药理活性，尤其是其卓越的心脑血管保护作用，降糖、降脂作用，抗炎、抗凝血作用以及特异性的肿瘤细胞增殖抑制作用等，相关作用机制及靶点主要包括激活 SIRT1/ERK1/2/Homer1a 途径，增强机体清除氧自由基的能力、降低心肌细胞膜脂质过氧化的程度，上调 PTEN、下调 NF-κB 的表达，调节 Ca^{2+}、K^+、Na^+等离子的跨膜转运，促进 GSK-3β 的磷酸化，抑制 PKC 的磷酸化，抑制 Wnt/β-catenin 信号通路，抑制炎症因子表达，抑制血小板聚集，诱导肿瘤细胞周期停滞及凋亡，导致病毒直接失活或抑制子代病毒释放[21]。越南人参皂苷 R_{10} (**5**)为奥克梯隆醇型皂苷, 是一类侧链具有呋喃环的四环三萜类皂苷，主要存在于人参属植物西洋参(*P. quinquefolius*)、竹节参(*P. japonicus*)、狭叶竹节参(*P. bipinnatifidus* var. *angustifolius*)、珠子参(*P. japonicus* var. *major*)、喜马拉雅假人参(*P. pseudo-ginseng* subsp. *himalaicus*)中，葫芦科(Cucurbitaceae)和桦木属(*Betula*)植物中也发现了多种奥克梯隆型皂苷。奥克梯隆型皂苷在这些植物中虽然含量甚微，但具有较强的生物活性，包括保护心肌损伤、增强神经元活性、抗肿瘤等[22–23]。

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