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# 厚梗染木树化学成分的研究

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**摘 要**:为从染木树属(*Saprosma*)中获取更多有效的化合物,对中国特有的热带药用植物厚梗染木树(*Saprosma crassipes*)中的化学成分进行分离纯化以及鉴定,增加染木树属化合物多样性。综合利用正相硅胶柱层析、Sephadex LH-20凝胶柱层析、半制备HPLC等色谱分离技术,对该植物的乙醇提取物进行系统的分离纯化;并运用NMR以及质谱鉴定技术鉴定化合物的结构。从厚梗染木树中分离鉴定了8个化合物(**1~8**),包括2个环烯醚萜苷:车叶草苷(**1**)、去乙酰基车叶草苷酸(**2**),1个蒽醌:1,3-二羟基-2-羟甲基蒽醌(**3**)以及5个甾体类化合物:卡基二醇(**4**)、9,11-脱氢麦角甾醇过氧化物(**5**)、过氧化麦角甾醇(**6**)、羟基烯酮(**7**)和(3$\beta$)-柱头-5,25-二烯-3-醇(**8**);所有化合物均为首次从厚梗染木树中分离得到,化合物**3~8**为首次从染木树属中分离得到。

**关键词**:染木树属;厚梗染木树;化学成分;环烯醚萜苷;甾体;蒽醌
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# Study on Chemical Constituents of *Saprosma crassipes*

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**Abstract:** In order to obtain more effective compounds from *Saprosma*, the chemical components of *Saprosma crassipes*, a tropical medicinal plant endemic to China, were isolated, purified and identified to increase the diversity of compounds in *Saprosma crassipes*. The ethanol extracts of the plant were systematically separated and purified by integrating normal-phase silica gel column chromatography, Sephadex LH-20 gel column chromatography, and semi-prepared HPLC. The NMR and mass spectrometry were used to identify the structure of compounds. Eight compounds (**1-8**) were isolated from the stems of *Saprosma crassipes*, including two cyclic ether terpene glycosides asperuloside (**1**) and deacetyl asperulosidic acid (**2**); one anthraquinone 1,3-dihydroxy-2-hydroxymethylanthraquinone(**3**), and five steroids kakidiol (**4**), 9,11-dehydro-ergosterol peroxide (**5**), ergosterol peroxide (**6**), hydroxyenone (**7**), and (3$\beta$)-stigma-5,25-dien-3-ol (**8**). All compounds were isolated for the first time from *Saprosma crassipes*, and the compounds **3-8** were isolated for the first time from the genus *Saprosma*.

**Keywords:** *Saprosma*; *Saprosma crassipes*; chemical composition; cyclic etherterpene glycosides; steroids; anthraquinone

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茜草科（Rubiaceae）染木树属植物约30种，多为小乔木或灌木，主要分布在亚洲热带地区，我国有5种，包括海南染木树、厚梗染木树、琼岛染木树、染木树和云南染木树，常见于云南和海南，已经被用作治疗发烧的传统药物[1-3]。相关研究表明染木树属中主要含有环烯醚萜苷、硫代环烯醚萜苷、蒽醌类、生物碱类以及三萜类化合物；这些成分具有抑菌[4]、抗肿瘤[5]作用。其中厚梗染木树（*Saprosma crassipes*）是茜草科染木树属的小乔木，是我国特有的热带药用植物，主要产自海南东方、三亚、琼中、乐东、陵水和云南西双版纳。到目前为止仅有一篇文献报道其含有蒽醌类化合物且具有抗菌活性作用[6]。为了合理开发利用热带药用植物厚梗染木树，丰富染木树属化学结构多样性，本文对其活性化学成分进行了进一步的研究，通过系统分离纯化与鉴定从中分离鉴定了8个化合物（**1~8**），包括2个环烯醚萜苷车叶草苷（**1**）、去乙酰基车叶草苷酸（**2**），1个蒽醌1,3-二羟基-2-羟甲基蒽醌（**3**）以及5个甾体类化合物卡基二醇（**4**）、9,11-脱氢麦角甾醇过氧化物（**5**）、过氧化麦角甾醇（**6**）、羟基烯酮（**7**）和（3$\beta$）-柱头-5,25-二烯-3-醇（**8**）；所有化合物均为首次从厚梗染木树中分离得到，化合物**3~8**为首次从染木树属中分离得到（图1）。

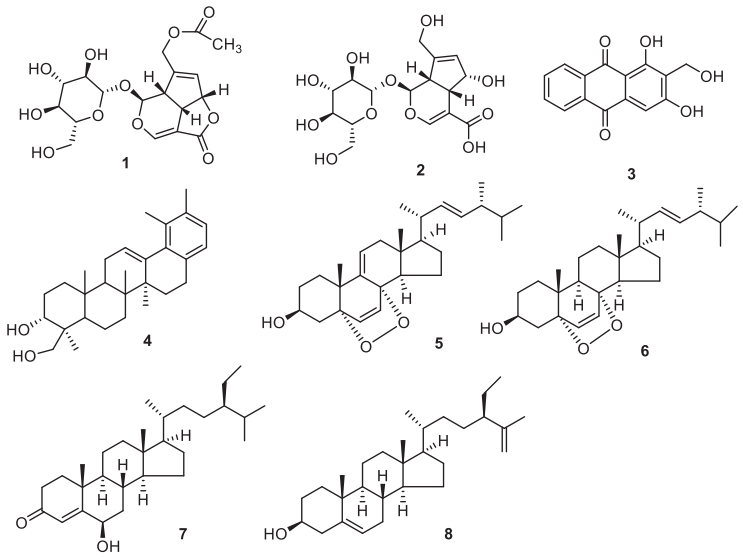

**图1　化合物 1~8的化学结构**

**Figure 1**　Structures of compounds 1-8

## 1 材料与方法

### 1.1 仪器与试剂

Bruker AV-400 型超导核磁共振仪，德国 Bruker 公司；GAA076 LC 质谱仪，瑞士 Bruker 公司；Agilent 1100分析型高效液相色谱仪，美国安捷伦公司；薄层硅胶GF254以及柱色谱硅胶，200~300目，青岛海洋化工厂；Sephadex LH-20凝胶，Amersham Blosclences 公司；恒温水浴锅 B-220，上海亚荣生化仪器厂；EYELAN-100旋转蒸发仪，日本东京理化有限公司；实验中所使用的化学试剂均来自广州西陇化工公司。

### 1.2 厚梗染木树的采集

厚梗染木树的茎于2018年6月采自中国海南省昌江县霸王岭国家级自然保护区（北纬18° 582539′，东经108° 876 352′），经海南师范大学化学化工学院周学明讲师鉴定。该植物是在当地林业局的指导下采集的，通过与文献中对该植物的描述进行比较，将其鉴定为厚梗染木树的茎。凭证标本（编号RMS20180622）保存在海南师范大学热带药用资源化学教育部重点实验室。

### 1.3 提取与分离

在室温下，将厚梗染木树风干和粉末化的粗茎用15 L的90%乙醇放置室内浸泡7 d，重复该操作3遍，合并得到的提取液，减压浓缩回收剂，获得粘稠状的浸膏。减压浓缩后，把浸膏分装在1 000 mL的蒸馏水中，

依次用等体积的石油醚和乙酸乙酯萃取。厚梗染木树乙醇浸膏经硅胶(48~75 μm)柱层析进行分离,以石油醚-乙酸乙酯混合液作为洗脱剂按体积比1∶0~0∶1进行梯度洗脱,并按500 mL收集流分。经TLC检测后合并相似流分,后分成8个流分(Frs. 1~8)。将组分Fr.2继续采用硅胶(48~75 μm)柱层析进行分离,以石油醚-乙酸乙酯混合液作为洗脱剂按5∶1(体积比)进行等度洗脱,按照250 mL收集流分,并经TLC检测后合并为4个小组分;其中小组分1采用Sephadex LH-20柱色层析进行纯化,用氯仿-甲醇(1∶1, *v/v*)进行洗脱除去色素后,用半制备HPLC进一步纯化(RP $C_{18}$柱,流速为2.5 mL/min,$CH_3CN-H_2O$=70∶30, *v/v*)得到化合物**5**(6.2 mg)与化合物**6**(7.8 mg);小组分2采用Sephadex LH-20柱色层析进行纯化,用氯仿-甲醇(1∶1, *v/v*)进行洗脱除去色素后,用半制备HPLC进一步纯化(RP $C_{18}$柱,流速为2.5 mL/min,$CH_3CN-H_2O$=65∶35, *v/v*)得到化合物**7**(4.6 mg)与化合物**8**(5.6 mg);小组分3采用Sephadex LH-20柱色层析进行纯化,用氯仿-甲醇(1∶1, *v/v*)进行洗脱除去色素后,用半制备HPLC进一步纯化(RP $C_{18}$柱,流速为2.5 mL/min,$CH_3CN-H_2O$=60∶40 *v/v*)得到化合物**4**(3.8 mg);小组分4经ODS柱层析进行纯化,用水与甲醇(50∶50, *v/v*) 进行洗脱得到化合物**3**(11.5 mg)。组分Fr. 5采用继续采用硅胶(48~75 μm)柱层析进行分离,以氯仿-甲醇混合液作为洗脱剂按20∶1(体积比) 进行等度洗脱,按照250 mL收集流分,并经TLC检测后合并为3个小组分;其中小组分2经ODS柱层析进行纯化处理后用半制备HPLC进一步纯化(RP $C_{18}$柱,流速为2.5 mL/min,$CH_3CN-H_2O$=20∶80, *v/v*)得到化合物**1**(4.2 mg)和**2**(4.8 mg)。

## 2 结构鉴定

化合物**1**:无色膏状;根据低分辨质谱ESI-MS给出的准分子离子峰为414.0,结合波谱数据,推断其相对分子式为$C_{18}H_{22}O_{11}$;$^1$H NMR (DMSO-$d_6$, 400 MHz):$\delta$ 7.40 (1H, d, *J*=2.0, H-3), 5.82 (1H, s, H-1), 5.71 (1H, s, H-7), 5.55 (1H, d, *J*=6.4 Hz, H-6), 4.66 (2H, d, *J*=6 Hz, H-10), 4.49 (1H, d, *J*=8.0 Hz, H-1′), 3.70 (1H, dd, *J*=10.4, 10 Hz, H-6′), 3.55 (1H, m, H-5), 3.45 (1H, m, H-6′), 3.19 (1H, m, H-9), 3.17-2.95 (4H, m, H-2′/3′/4′/5′), 2.05 (1H, s, Me);$^{13}$C NMR (DMSO-$d_6$, 100 MHz):$\delta$ 91.1 (CH-1), 148.7 (CH-3), 104.6 (C-4), 35.7 (CH-5), 84.0 (CH-6), 127.0 (CH-7), 142.6 (C-8), 43.5 (CH-9), 60.4 ($CH_2$-10), 169.5 (C-11), 20.5 ($CH_3$-13), 170.0 (C-12), 98.4 (CH-1′), 73.0 (CH-2′), 76.5 (CH-3′), 70.1 (CH-4′), 77.3 (CH-5′), 61.2 ($CH_2$-6′)。以上$^1$H NMR和$^{13}$C NMR数据与文献[7]报道相同,推断化合物**1**为asperuloside。

化合物**2**:无色膏状;根据低分辨质谱ESI-MS给出的准分子离子峰为390.0,结合波谱数据,推断其分子式为$C_{16}H_{22}O_{11}$;$^1$H NMR (DMSO-$d_6$, 400 MHz):$\delta$ 7.67 (1H, s, H-3), 6.04 (1H, s, H-7), 5.07 (1H, d, *J*=8.0 Hz, H-1), 4.74 (1H, d, *J*=5.6 Hz, H-7), 4.47 (1H, d, *J*=10.8 Hz, H-10), 4.23 (1H, d, *J*=10.4, H-10), 3.02 (1H, m, H-5), 2.58 (1H, m, H-9); $^{13}$C NMR (DMSO-$d_6$, 100 MHz):$\delta$ 101.5 (CH-1), 155.3 (CH-3), 108.6 (C-4), 42.8 (CH-5), 75.4 (CH-6), 129.9 (CH-7), 151.5 (C-8), 45.9 (CH-9), 61.7 ($CH_2$-10), 170.9 (C-11), 100.4 (CH-1′), 75.0 (CH-2′), 78.5 (CH-3′), 71.6 (CH-4′), 77.8 (CH-5′), 62.8 ($CH_2$-6′)。以上$^1$H NMR和$^{13}$C NMR数据与文献[8]报道相同,推断化合物**2**为deacetyl asperulosidic acid。

化合物**3**:黄色粉末;根据低分辨质谱ESI-MS给出的准分子离子峰为270.0,结合波谱数据,推断其分子式为$C_{15}H_{10}O_5$;$^1$H NMR (DMSO-$d_6$, 400 MHz):$\delta$ 13.12 (1H, s, 1-OH), 8.11 (1H, d, *J* = 8 Hz, H-8), 8.06 (1H, d, *J* =8 Hz, H-5), 7.88-7.81 (2H, m, H-6,7), 7.16 (1H, s, H-4), 4.47 (2H, s, 2-$CH_2$OH);$^{13}$C NMR (DMSO-$d_6$, 100 MHz):$\delta$ 186.0 (C-9), 181.7 (C-10), 163.8 (C-3), 163.1 (C-1), 134.6 (CH-7), 134.4 (CH-6), 133.2 (C-4a), 133.0 (C-8a), 132.8 (C-10a), 126.7 (CH-5), 126.3 (CH-8), 120.2 (C-2), 108.9 (C-9a), 51.2 (2-$CH_2$OH)。以上$^1$H NMR和$^{13}$C NMR数据与文献[9]报道相同,推断化合物**3**为1,3-dihydroxy-2-hydroxymethyl-anthraquinone。

化合物**4**:无色针晶;根据低分辨质谱ESI-MS给出的准分子离子峰为422.0,结合波谱数据,推断其分子式为$C_{29}H_{42}O_2$;$^1$H NMR ($CDCl_3$, 600 MHz):$\delta$ 6.93 (1H, d, *J* = 7.2 Hz, H-22), 6.79 (1H, d, *J* = 7.8 Hz, H-21), 5.50 (1H, dd, *J* = 3.0, 3.0 Hz, H-12), 4.12 (1H, m, H-3), 3.76/3.58 (1H, d, *J* = 10.8 Hz, H-24), 2.27 (3H, d, J= 1.8, H-29), 1.25 (3H, s, H-23), 1.12 (3H, s, H-25), 1.00 (3H, s, H-26), 0.95 (3H, d, J = 12.6, H-27), 2.05

(3H, s, H-30)；$^{13}$C NMR ($CDCl_3$, 150 MHz):$\delta$ 34.3 ($CH_2$-1), 27.6 ($CH_2$-2), 70.8 (CH-3), 43.0 (C-4), 44.2 (CH-5), 18.7 ($CH_2$-6), 34.3 ($CH_2$-7), 40.2 (C-8), 47.9 (CH-9), 37.0 (C-10), 23.6 ($CH_2$-11), 125.4 (CH-12), 139.1 (C-13), 50.0 (C-14), 29.9 ($CH_2$-15), 31.1 ($CH_2$-16), 138.6 (C-17), 138.6 (C-18), 133.9 (C-19), 127.4 (C-20), 125.4 (CH-21), 123.0 (CH-22), 23.6 ($CH_3$-23), 66.8 ($CH_2$-24), 21.0 ($CH_3$-25), 16.6 ($CH_3$-26), 27.6 ($CH_3$-27), 17.1 ($CH_3$-29), 21.8 ($CH_3$-30)。以上$^1$H NMR和$^{13}$C NMR数据与文献[10]报道相同，推断化合物**4**为kakidiol。

化合物**5**：无色针晶；根据低分辨质谱ESI-MS给出的准分子离子峰为426.0，结合波谱数据，推断其分子式为$C_{28}H_{42}O_3$；$^1$H NMR ($CDCl_3$, 600 MHz):$\delta$ 6.60 (1H, *J* = 9.0 Hz, H-7), 6.29 (1H, *J* = 9.0 Hz, H-19), 5.43 (1H, dd, *J* = 6.0, 1.8 Hz, H-11), 5.24 (1H, dd, *J* = 15.0, 7.2 Hz, H-23), 5.16 (1H, dd, *J* = 15.0, 6.4 Hz, H-22), 4.00 (1H, m, H-3), 1.09 (3H, s, H-19), 1.00 (3H, d, *J* = 6.6 Hz, H-21), 0.91 (3H, d, *J* = 6.6 Hz, H-28), 0.84 (3H, d, *J* = 6.6 Hz, H-26), 0.82 (3H, d, *J* = 6.6 Hz, H-27), 0.74 (3H, s, H-18); $^{13}$C NMR ($CDCl_3$, 150 MHz):$\delta$ 32.7 ($CH_2$-1), 30.7 ($CH_2$-2), 66.5 (CH-3), 36.2 ($CH_2$-4), 82.8 (C-5), 135.6 (CH-6), 130.9 (CH-7), 78.5 (C-8), 142.5 (C-9), 38.1 (C-10), 119.9 (CH-11), 41.3 ($CH_2$-12), 43.7 (C-13), 48.3 (CH-14), 21.0 ($CH_2$-15), 28.8 ($CH_2$-16), 55.9 (CH-17), 13.1 ($CH_3$-18), 25.7 ($CH_3$-19), 40.1 (CH-20), 21.0 ($CH_3$-21), 135.2 (CH-22), 132.5 (CH-23), 42.9 (CH-24), 33.2 (CH-25), 19.8 ($CH_3$-26), 20.1 ($CH_3$-27), 17.7 ($CH_3$-28)。以上$^1$H NMR和$^{13}$C NMR数据与文献[11]报道相同，推断化合物**5**为9,11-dehydro ergosterol peroxide。

化合物**6**：无色针晶；根据低分辨质谱ESI-MS给出的准分子离子峰为428.0，结合波谱数据，推断其分子式为$C_{28}H_{44}O_3$；$^1$H NMR ($CDCl_3$, 600 MHz):$\delta$ 6.50 (1H, d, *J* =12 Hz, H-7), 6.24 (1H, d, *J* = 12 Hz, H-6), 5.22 (1H, dd, *J* = 12, 18 Hz, H-23), 5.14 (1H, dd, *J* =12, 18 Hz, H-22), 3.97 (1H, m, H-3), 0.99 (3H, d, *J* = 6 Hz, H-21), 0.90 (3H, d, *J* = 6 Hz, H-28), 0.88 (3H, s, H-19), 0.83 (3H, d, *J* = 6 Hz, H-26), 0.82 (3H, d, *J* = 6 Hz, H-27), 0.81 (3H, s, H-18)；$^{13}$C NMR ($CDCl_3$, 150 MHz):$\delta$ 34.8 ($CH_2$-1), 30.3 ($CH_2$-2), 66.6 (CH-3), 37.1 ($CH_2$-4), 82.3 (C-5), 135.6 (CH-6), 130.9 (CH-7), 79.6 (C-8), 51.2 (CH-9), 37.1 (C-10), 20.8 ($CH_2$-11), 39.5 ($CH_2$-12), 44.7 (C-13), 51.8 (CH-14), 23.5 ($CH_2$-15), 28.8 ($CH_2$-16), 56.3 (CH-17), 13.0 ($CH_3$-18), 18.3 ($CH_3$-19), 39.9 (CH-20), 21.0 ($CH_3$-21), 135.3 (CH-22), 132.4 (CH-23), 42.9 (CH-24), 33.2 (CH-25), 19.8 ($CH_3$-26), 20.1 ($CH_3$-27), 17.7 ($CH_3$-28)。以上$^1$H NMR和$^{13}$C NMR数据与文献[12]报道相同，推断化合物**6**为ergosterol peroxide。

化合物**7**：无色针晶；根据低分辨质谱ESI-MS给出的准分子离子峰为428.0，结合波谱数据，推断其分子式为$C_{29}H_{48}O_2$；$^1$H NMR ($CDCl_3$, 600 MHz):$\delta$ 5.81 (1H, s, H-4), 4.34 (1H, t, *J* = 3.0 Hz, H-6), 1.37 (3H, s, H-19), 0.92(3H, d, *J* = 6.6 Hz, H-21), 0.84 (3H, t, *J* = 7.8 Hz, H-29), 0.83 (3H, d, *J* =2 Hz, H-26), 0.80 (3H, d, *J* = 7.2 Hz, H-27), 0.73 (3H, s, H-18)；$^{13}$C NMR ($CDCl_3$, 150 MHz):$\delta$ 37.2 ($CH_2$-1), 34.4 ($CH_2$-2), 200.7 (C-3), 126.4 (CH-4), 168.8 (C-5), 73.4 (CH-6), 38.6 ($CH_2$-7), 29.8 (CH-8), 53.7 (CH-9), 38.1 (C-10), 21.1 ($CH_2$-11), 39.7 ($CH_2$-12), 42.6 (C-13), 56.1 (CH-14), 24.3 ($CH_2$-15), 28.3 ($CH_2$-16), 56.0 (CH-17), 12.1 ($CH_3$-18), 19.6 ($CH_3$-19), 36.2 (CH-20), 18.9 ($CH_3$-21), 34.0 ($CH_2$-22), 26.1 ($CH_2$-23), 45.9 (CH-24), 29.2 ($CH_2$-25), 20.0 ($CH_3$-26), 19.1 ($CH_3$-27), 23.2 (CH-28), 12.1($CH_3$-29)。以上$^1$H NMR和$^{13}$C NMR数据与文献[13]报道相同，推断化合物**7**为hydroxyenone。

化合物**8**：无色针晶；根据低分辨质谱ESI-MS给出的准分子离子峰为412.0，结合波谱数据，推断其分子式为$C_{29}H_{48}O$；$^1$H NMR ($CDCl_3$, 600 MHz):$\delta$ 5.35 (1H, t, *J* = 3.0 Hz, H-6), 4.72 (1H, m, H-26a), 4.64 (1H, m, H-26b), 3.52 (1H, m, H-3), 1.56 (3H, s, H-27), 1.01 (3H, s, H-19), 0.90 (3H, d, *J* = 6.6 Hz, H-21), 0.80 (3H, t, *J* = 7.2 Hz, H-29), 0.67 (3H, s, H-18)；$^{13}$C NMR ($CDCl_3$, 150 MHz):$\delta$ 37.4 ($CH_2$-1), 31.8 ($CH_2$-2), 72.0 (CH-3), 42.4 ($CH_2$-4), 140.9 (C-5), 121.9 (CH-6), 32.0 ($CH_2$-7), 32.1 (CH-8), 50.3 (CH-9), 36.6 (C-10), 21.2 ($CH_2$-11), 39.9 ($CH_2$-12), 42.5 (C-13), 56.9 (CH-14), 24.4 ($CH_2$-15), 28.3 ($CH_2$-16), 56.2 (CH-17), 12.0 ($CH_3$-18), 19.5 ($CH_3$-19), 35.7 (CH-20), 18.8 ($CH_3$-21), 33.8 ($CH_2$-22), 29.5 ($CH_2$-23), 49.7 (CH-24), 147.7 (C-25), 17.9 ($CH_3$-26), 111.5 ($CH_2$-27), 26.7 ($CH_2$-28), 12.2($CH_3$-29)。以上$^1$H NMR和$^{13}$C NMR数据

与文献[14]报道相同,推断化合物**8**为(3$\beta$)-Stigmasta-5,25-dien-3-ol。

## 3 结论

本研究选择热带药用植物厚梗染木树粗茎进行化学成分研究,从中分离并鉴定出8个化合物,所有化合物均为首次从厚梗染木树中分离得到,化合物**3~8**为首次从染木树属中分离得到。研究发现厚梗染木树主要化学成分是甾体类化合物,其具有极强的消炎及免疫抑制作用;其次是蒽醌类化合物,其具有止血、抗菌的作用;还有环烯醚萜苷类化合物,其具有抗肿瘤、保肝护肝、抗炎等作用。本研究丰富了对染木树属化学成分的认识,为进一步阐明其药效物质基础提供了数据支持。

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