

为主,同时含 α-糖苷键的吡喃型多糖化合物。

<sup>13</sup>C-NMR 光谱分析:LCP-1 的 C<sub>1</sub> 化学位移值为 99.7, α 型连接, C<sub>2</sub>, C<sub>3</sub>, C<sub>4</sub> 和 C<sub>5</sub> 化学位移在 70~76 之间,而 C<sub>6</sub> 化学位移值为 60.5,进一步确定 LCP-1 为 α-糖苷键的多糖化合物。

#### 4 结论

LCP-1 为一由阿拉伯糖、葡萄糖和甘露糖组成的 α-糖苷键的吡喃型多糖化合物,相对分子质量为 2.83 × 10<sup>4</sup>,离散度为 1.60;LCP-2 为由阿拉伯糖、葡萄糖、半乳糖以及甘露糖组成,同时含有 α-和 β-糖苷键吡喃型多糖化合物,相对分子质量为 5.92 × 10<sup>4</sup>,离散度为 1.79;LCP-3 为由阿拉伯糖、葡萄糖、半乳糖以及甘露糖组成,同时含有 α-和 β-糖苷键并以 β-糖苷键为主的吡喃型多糖化合物,相对分子质量为 1.23 × 10<sup>4</sup>,离散度为 1.63;LCP-4 为由阿拉伯糖、葡萄糖和半乳糖组成,同时含有 α-和 β-糖苷键并以 β-糖苷键为主的吡喃型多糖化合物,相对分子质量为 6.31 × 10<sup>4</sup>,离散度为 1.88。

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## 蒙桑茎皮中 Diels-Alder 型加合物化学成分的研究

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**摘要:**目的 研究蒙桑 *Morus mongolica* 茎皮中的 Diels-Alder 型加合物化学成分。方法 利用硅胶柱色谱、RP-18 及 Sephadex LH-20 进行分离纯化,根据各种光谱技术进行结构鉴定。结果 分离得到 6 个 Diels-Alder 型加合物,分别为阿尔本 F(albanin F, I)、桑酮 L(kuwanon L, II)、双桑辛素(dimoracin, III)、桑酮 J(kuwanon J, IV)、桑呋喃 J(mulberrofuran J, V)、桑呋喃 Q(mulberrofuran Q, VI)。结论 化合物 II、III、V 为首次从该种植物分得。抗氧化筛选结果表明,化合物 IV~VI 具有抗氧化活性。

**关键词:**蒙桑;Diels-Alder 型加合物;抗氧化活性

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### Diels-Alder type adducts in stem bark of *Morus mongolica*

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**Abstract: Objective** To study the Diels-Alder type adducts in the stem bark of *Morus mongolica*. **Methods** The compounds were isolated and purified by silic gel column chromatography, RP-18 and Sephadex LH-20. The structures were identified by various spectral evidence. **Results** Six Diels-Alder type adducts were obtained and they were albanin F (I), kuwanon L (II), dimoracin (III), kuwanon J (IV), mulberrofuran J (V), mulberrofuran Q (VI). **Conclusion** Compounds II, III, and V are isolated from the plant for the first time and compounds IV—VI show the anti-oxidation activities.

**Key words:** *Morus mongolica* Schneid; Diels-Alder type adducts; anti-oxidation activity

蒙桑 *Morus mongolica* Schneid. 为桑科桑属植物,其同属植物桑 *M. alba* L. 的根皮(桑白皮)为常用中药,且研究较多,主要化学成分为 Diels-Alder 型加合物和黄酮类化合物;药理活性主要为利尿降压、镇咳抗炎等。桑属植物全世界共有 16 种,中国有 11 种,除桑白皮外,其余几种的研究均不多。本实验对蒙桑茎皮 95%乙醇提取物的醋酸乙酯部分进行了研究,共分离鉴定了 6 个 Diels-Alder 型加合物,其中 3 个化合物具有抗氧化活性。

### 1 仪器与材料

$^1\text{H-NMR}$ 和 $^{13}\text{C-NMR}$ 用 INOVA-500 型核磁共振仪测定。FAB-MS 用 VG Autospec-300 型质谱仪测定,ESI-MS 用 Agilent 1100 LC/MSD Trap SL 型质谱仪测定。红外光谱用 IMPACT 400 型红外光谱仪测定。旋光用 Perkin-Elmer-241 型旋光仪测定。Sephadex LH-20 购自 Pharmacia 公司,RP-18(40~60  $\mu\text{m}$ )购自 Merck 公司。薄层色谱硅胶 GF<sub>254</sub>和柱色谱硅胶(160~200 目)为青岛海洋化工厂产品。溶剂均为分析纯,由北京化学试剂厂生产。

实验药材茎皮由重庆市药物种植研究所易思荣副研究员采自四川金佛山,并鉴定为桑属植物蒙桑 *M. mongolica* Schneid.。

### 2 提取与分离

蒙桑的干燥茎皮 6.9 kg,95%乙醇回流提取 3 次,浸膏(600 g)用硅胶拌样后分别用石油醚、氯仿、醋酸乙酯、丙酮、甲醇洗脱。醋酸乙酯浸膏(190 g)经硅胶柱色谱分离,用石油醚-丙酮梯度洗脱,得到 11 个部分。第 5 和第 7 部分分别经 Sephadex LH-20 柱色谱分离,甲醇洗脱,得到化合物 III(10 mg)、IV(35 mg)。第 8 部分先经硅胶柱色谱分离[氯仿-甲醇(9:1)洗脱],再经 RP-18(40~60  $\mu\text{m}$ )柱色谱分离[甲醇-水(7:3)洗脱],得到化合物 I(40 mg)、II(15 mg)、V(42 mg)、VI(45 mg)。

### 3 结构鉴定

化合物 I:棕色无定形粉末, $[\alpha]_{\text{D}}^{25} - 529^\circ$  (MeOH)。IR $\nu_{\text{max}}^{\text{KBr}}$ ( $\text{cm}^{-1}$ ):3 311,1 699,1 653,1 624,

1 367,1 136,974。ESI-MS  $m/z$ :693[M+1]<sup>+</sup>,715[M+Na]<sup>+</sup>。 $^1\text{H-NMR}$ (acetone- $\text{d}_6$ ) $\delta$ :7.29(1H,d, $J=8.4$  Hz,H-6'),7.25(1H,d, $J=8.8$  Hz,H-14''),6.78(1H,d, $J=8.4$  Hz,H-20''),6.66(1H,br s,H-3'),6.56(1H,dd, $J=8.4,2.0$  Hz,H-5'),6.20(1H,br s,H-17''),6.06(1H,d, $J=8.4$  Hz,H-19''),6.01(1H,br s,H-11''),5.98(1H,br s,H-6),5.92(1H,d, $J=8.8$  Hz,H-13''),5.21(1H,br s,H-2''),5.18(1H,t, $J=7.0$  Hz,H-10),4.62(1H,br s,H-4''),4.42(1H,br d, $J=9.6$  Hz,H-3''),3.18(1H,m,H-5''),3.09(2H,m,H-9),1.80~2.00(2H,overlapped,H-6''),1.60(3H,s,H-12),1.51(3H,s,H-7''),1.47(3H,s,H-13)。 $^{13}\text{C-NMR}$ (acetone- $\text{d}_6$ ) $\delta$ :209.3(C-8''),183.2(C-4),166.0(C-10''),164.9(C-12''),162.0(C-8a),161.5(C-4'),161.3(C-2,7),157.3(C-5),157.0(C-2',18''),156.2(C-16''),133.9(C-14''),133.6(C-1''),132.1(C-11,6',20''),124.3(C-2''),122.8(C-10,15''),121.3(C-3),115.6(C-9''),113.3(C-1'),108.1(C-5',19''),107.8(C-8,13''),105.5(C-4a),103.7(C-3',17''),102.9(C-11''),98.3(C-6),47.6(C-4''),38.6(C-3'',5'',6''),25.8(C-12),24.4(C-9),23.0(C-7''),17.7(C-13)。因其旋光值为负值,且 H-3'' 的偶合常数为 9.6 Hz,依文献中规律<sup>[1]</sup>,该化合物为全反型加合物,其绝对构型为 3''R,4''R,5''S。以上数据与文献报道基本一致<sup>[2,3]</sup>,确定为阿尔本 F(albanin F)。

化合物 II:棕色无定形粉末, $[\alpha]_{\text{D}}^{25} - 227^\circ$  (MeOH)。IR $\nu_{\text{max}}^{\text{KBr}}$ ( $\text{cm}^{-1}$ ):3 386,1 628,1 456,1 300,1 159,972。ESI-MS  $m/z$ :627(M<sup>+</sup>+1,95),153(98),115(100)。 $^1\text{H-NMR}$ (acetone- $\text{d}_6$ ) $\delta$ :7.46(1H,d, $J=8.8$  Hz,H-14''),7.36(1H,d, $J=8.8$  Hz,H-6'),6.93(1H,d, $J=8.8$  Hz,H-20''),6.20~5.88(7H,m,H-6,8,5',11'',13'',17'',19''),5.66(1H,m,H-2),5.50(1H,br s,H-2''),4.72(1H,br s,H-4''),4.50(1H,t, $J=10.0$  Hz,H-3''),3.52(1H,br s,H-5''),3.27~3.18(1H,m,H-3a),3.05~2.98(1H,

m, H-3b), 2.25~2.20 (2H, m, H-6''), 1.81 (3H, s, H-7'').  $^{13}\text{C-NMR}$  (acetone- $d_6$ )  $\delta$ : 208.2 (C-8''), 197.0 (C-4), 168.8 (C-7), 165.7 (C-5, 12''), 165.2 (C-8a), 164.6 (C-10''), 156.5 (C-18''), 156.1 (C-16''), 155.4 (C-2'), 154.1 (C-4'), 133.5 (C-1'', 20''), 127.0 (C-14'', 6'), 126.4 (C-2''), 123.8 (C-15''), 118.7 (C-1'), 108.1 (C-13'', 3'), 107.9 (C-19''), 107.3 (C-5'), 103.6 (C-11''), 102.7 (C-4a, 17''), 96.9 (C-6), 96.2 (C-8), 75.6 (C-2), 43.1 (C-3, 4''), 38.7 (C-3'', 5'', 6''), 23.5 (C-7''). 因其旋光值为负值, 且 H-3'' 的偶合常数为 10.0 Hz, 依照文献总结的规律<sup>[1]</sup>, 该化合物为全反型加合物, 其绝对构型为 3''R, 4''R, 5''S。以上数据与文献报道的基本一致<sup>[4]</sup>, 故确定为桑酮 L (kuwanon L)。

化合物 III: 棕色无定形粉末,  $[\alpha]_D^{25} - 5^\circ$  (EtOH)。IR  $\nu_{\text{max}}^{\text{KBr}}$  ( $\text{cm}^{-1}$ ): 3 346, 1 622, 1 365, 1 146, 1 061, 968。FAB-MS  $m/z$ : 616 ( $[\text{M}]^+$ , 40), 391 (30), 185 (50), 93 (100)。 $^1\text{H-NMR}$  (acetone- $d_6$ )  $\delta$ : 7.41 (1H, d,  $J = 8.5$  Hz, H-4), 7.37 (1H, d,  $J = 8.0$  Hz, H-4''), 7.04 (1H, s, H-3''), 6.97~7.00 (4H, m, H-3, 2'', 6'', 7''), 6.94 (1H, m, H-7), 6.82 (1H, dd,  $J = 8.0, 2.0$  Hz, H-5''), 6.80 (1H, dd,  $J = 8.5, 2.0$  Hz, H-5), 6.77 (2H, s, H-2', 6'), 5.62 (1H, d,  $J = 10.0$  Hz, H-17), 5.56 (1H, br s, H-9), 3.90 (1H, br s, H-8), 2.76 (1H, dd,  $J = 10.0, 4.5$  Hz, H-16), 2.32 (1H, d,  $J = 18.5$  Hz, H-12a), 1.68 (3H, s, H-11), 1.47 (1H, d,  $J = 18.5$  Hz, H-12b), 1.10 (3H, s, H-14), 0.74 (3H, s, H-15)。 $^{13}\text{C-NMR}$  (acetone- $d_6$ )  $\delta$ : 157.2 (C-2), 156.8 (C-6, 2''), 156.6 (C-7a, 6''), 156.5 (C-3'', 5''), 155.6 (C-3', 5'), 155.0 (C-7''a), 132.5 (C-10), 132.2 (C-1''), 130.3 (C-1'), 123.3 (C-9), 122.6 (C-3''a), 122.5 (C-3a), 122.0 (C-4''), 121.8 (C-4), 116.4 (C-4''), 114.6 (C-4'), 113.3 (C-5''), 113.1 (C-5), 104.6 (C-2', 6'), 103.3 (C-2'', 6''), 102.5 (C-3''), 101.7 (C-3), 98.4 (C-7, 7''), 71.7 (C-17), 42.4 (C-16), 41.2 (C-12), 33.2 (C-8), 33.0 (C-13), 30.0 (C-14, overlapped), 28.9 (C-15), 23.8 (C-11)。以上 IR、FAB-MS 和  $^1\text{H-NMR}$  的数据为文献报道的基本一致<sup>[5]</sup>, 其碳谱数据与首次报道, 故确定为双桑辛素 (dimoracin)。

化合物 IV: 棕色无定形粉末,  $[\alpha]_D^{25} + 85^\circ$  (MeOH)。IR  $\nu_{\text{max}}^{\text{KBr}}$  ( $\text{cm}^{-1}$ ): 3 340, 1 612, 1 271, 1 093, 976。FAB-MS  $m/z$ : 679 ( $[\text{M} + 1]^+$ , 20), 447 (98),

205 (50), 149 (100)。 $^1\text{H-NMR}$  (acetone- $d_6$ )  $\delta$ : 8.37 (1H, d,  $J = 9.0$  Hz, H-14''), 8.15 (1H, d,  $J = 15.5$  Hz, H- $\beta$ ), 7.83 (1H, d,  $J = 8.5$  Hz, H-6'), 7.70 (1H, d,  $J = 15.5$  Hz, H- $\alpha$ ), 7.64 (1H, d,  $J = 8.5$  Hz, H-6), 6.96 (1H, d,  $J = 8.0$  Hz, H-20''), 6.52 (1H, d,  $J = 2.0$  Hz, H-17''), 6.47 (1H, d,  $J = 2.0$  Hz, H-3), 6.42 (1H, d,  $J = 9.0$  Hz, H-13''), 6.41 (1H, dd,  $J = 8.5, 2.0$  Hz, H-5), 6.35 (1H, d,  $J = 8.5$  Hz, H-5'), 6.30 (1H, dd,  $J = 8.0, 2.0$  Hz, H-19''), 5.67 (1H, br s, H-2''), 5.16 (1H, t,  $J = 7.5$  Hz, H-22''), 4.66 (1H, t,  $J = 5.0$  Hz, H-4''), 4.13 (1H, br s, H-3''), 3.80 (1H, br s, H-5''), 3.25 (2H, d,  $J = 7.5$  Hz, H-21''), 2.49 (1H, d,  $J = 18.5$  Hz, H-6''a), 2.21 (1H, d,  $J = 18.5$  Hz, H-6''b), 1.93 (3H, s, H-7''), 1.73 (3H, s, H-25''), 1.58 (3H, s, H-25'')。  $^{13}\text{C-NMR}$  (acetone- $d_6$ )  $\delta$ : 208.8 (C-8''), 192.7 (C=O), 165.1 (C-4'), 164.0 (C-10''), 162.8 (C-2'), 162.7 (C-12''), 161.7 (C-4), 159.3 (C-2), 157.3 (C-18''), 155.8 (C-16''), 140.3 (C- $\beta$ ), 134.1 (C-1''), 131.5 (C-14''), 131.1 (C-6), 130.8 (C-6'), 130.0 (C-23''), 128.1 (C-20''), 122.8 (C-2''), 122.5 (C-22''), 121.1 (C-15''), 116.7 (C- $\alpha$ ), 115.6 (C-3'), 115.2 (C-1), 114.6 (C-1'), 113.3 (C-9''), 112.7 (C-11''), 109.4 (C-5'), 108.5 (C-5), 107.6 (C-13''), 106.9 (C-19''), 102.9 (C-3, 17''), 46.8 (C-4''), 35.8 (C-5''), 31.9 (C-6''), 31.7 (C-3''), 25.2 (C-25''), 23.2 (C-7''), 21.5 (C-21''), 17.2 (C-24'')。因其旋光值为正值, 且 H-4'' 的偶合常数为 5.0 Hz, 依照文献总结的规律<sup>[1]</sup>, 该化合物为顺-反型加合物, 其绝对构型为 3''S, 4''R, 5''S。以上数据与文献报道的基本一致<sup>[6]</sup>, 故确定为桑酮 J (kuwanon J)。

化合物 V: 棕色无定形粉末,  $[\alpha]_D^{25} - 343^\circ$  (EtOH)。IR  $\nu_{\text{max}}^{\text{KBr}}$  ( $\text{cm}^{-1}$ ): 3 363, 1 697, 1 626, 1 442, 1 367, 1 142, 970。ESI-MS  $m/z$ : 603  $[\text{M} + \text{Na}]^+$ 。  $^1\text{H-NMR}$  (acetone- $d_6$ )  $\delta$ : 7.71 (1H, d,  $J = 8.8$  Hz, H-14''), 7.33 (1H, d,  $J = 8.4$  Hz, H-4), 6.90 (1H, d,  $J = 8.4$  Hz, H-20''), 6.90 (1H, br s, H-7), 6.83, 6.66 (2H, each br s, H-2', 6'), 6.82 (1H, s, H-3), 6.76 (1H, dd,  $J = 8.4, 2.0$  Hz, H-5), 6.22 (1H, br s, H-17''), 6.11 (1H, dd,  $J = 8.4, 2.0$  Hz, H-19''), 6.04 (1H, dd,  $J = 8.8, 2.4$  Hz, H-13''), 5.95 (1H, d,  $J = 2.4$  Hz, H-11''), 5.39 (1H, br s, H-2''), 4.90 (1H, br s, H-4''), 4.52 (1H, d,  $J = 10.0$  Hz, H-3''), 3.54 (1H, br s, H-5''), 2.51~2.21 (2H, m, H-6''), 1.75 (3H, s, H-7'')。  $^{13}\text{C-NMR}$  (acetone- $d_6$ )  $\delta$ : 209.7 (C-8''), 166.1

(C-10"), 164.8 (C-12"), 158.3 (C-7a), 157.2 (C-16", 18"), 156.7 (C-2), 156.5 (C-3', 5'), 155.3 (C-6), 134.2 (C-1"), 133.9 (C-20"), 130.6 (C-14", 1'), 125.1 (C-2"), 122.5 (C-15"), 121.8 (C-4, 3a), 117.3 (C-9"), 115.9 (C-4'), 113.1 (C-5), 107.7 (C-13"), 107.4 (C-19"), 104.7 (C-17"), 103.8 (C-2', 6'), 102.8 (C-3), 101.7 (C-11"), 98.3 (C-7), 46.7 (C-4"), 38.8 (C-3", 5", 6"), 23.4 (C-7"). 因其旋光值为负值,且 H-3"的偶合常数为 10.0 Hz,依照文献总结的规律<sup>[1]</sup>,该化合物为全反型加合物,其绝对构型为 3'R,4'R,5"S。以上数据与文献报道的基本一致<sup>[7]</sup>,故确定为桑味喃 J(mulberrofuran J)。

化合物 VI: 棕色无定形粉末,  $[\alpha]_D^{25} + 82.4^\circ$  (EtOH)。IR、<sup>1</sup>H-NMR、<sup>13</sup>C-NMR 光谱与文献报道的基本一致<sup>[8]</sup>,故确定为桑味喃 Q(mulberrofuran Q)。

#### 4 药理活性

对所得的 6 个化合物进行体外抗氧化活性筛选(Fe<sup>2+</sup>-半胱氨酸诱导的肝微粒体脂质过氧化模型),浓度为  $1 \times 10^5$  mol/L 时,化合物 IV ~ VI 有不同程度的抑

制脂质过氧化产物生成作用,抑制率分别为 43.3%、91.2%、85.6%。阳性对照 VE 抑制率为 66.7%。

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## 可疑翼手参中 4 个脑苷脂

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**摘要:**目的 研究可疑翼手参体内的活性成分。方法 对乙醇提取物的极性较小部分采用反复硅胶柱色谱方法分离纯化,并根据化合物的理化特性和光谱数据确定其结构。结果 分离并鉴定了 4 个脑苷脂类化合物:1-O-β-D-吡喃葡萄糖-(2S,3R,4E,8E)-2-正二十二烷酰氨基-13-甲基-4,8-十六烷二烯-1,3-二醇(I)、1-O-β-D-吡喃葡萄糖-(2S,3R,4E,8E)-2-[(2R)-2-羟基-二十二烷酰氨基]-13-甲基-4,8-十六烷二烯-1,3-二醇(II)、1-O-β-D-吡喃葡萄糖-(2S,3R,4E,8E)-2-[(2R)-2-羟基-二十三烷酰氨基]-13-甲基-4,8-十六烷二烯-1,3-二醇(III)、1-O-β-D-吡喃葡萄糖-(2S,3R,4E,8E)-2-[(2R)-2-羟基-二十四烷酰氨基]-13-甲基-4,8-十六烷二烯-1,3-二醇(IV)。结论 4 个脑苷脂化合物均为首次从此种海参体内分得。

**关键词:**可疑翼手参;海参;脑苷脂;甲醇分解;乙酰化

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## Four new cerebrosides from sea cucumber *Colochirus anceps*

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**Abstract: Objective** To study the active compounds of the sea cucumber *Colochirus anceps*. **Methods** The compounds were isolated and purified from the low-polarity part of EtOH extract of the sea cucumber,