

· 化学与分析 ·

## 连翘叶化学成分

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**[摘要]** 目的:研究连翘叶的化学成分。方法:采用 D101 大孔树脂法、硅胶柱色谱法和 HPLC 制备等多种方法分离纯化,通过 MS,NMR 等方法鉴定化合物的结构。结果:从连翘叶水提取物中分离到 10 个化合物,分别为芦丁(1),连翘苷(2), (+)松脂素- $\beta$ -D-葡萄糖苷(3),表松脂素- $\beta$ -D-葡萄糖苷(4),表松脂素-4'-O- $\beta$ -D-葡萄糖苷(5),forsythenside F(6), (+)-1-羟基松脂素-4''-O- $\beta$ -D-葡萄糖苷(7),连翘酯苷 I(8),连翘酯苷 H(9),连翘酯苷 A(10)。结论:化合物 4~7,9 为首次从连翘叶中分离得到。

**[关键词]** 连翘叶; 化学成分; 结构鉴定

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**Chemical Constituents of Leaves of *Forsythia suspense*** FAN Yi, CHEN Ling, ZHU Jie, ZHANG Hai-yan, DONG Jian-jun, ZHAO Tian-zeng\* (Biotchnology Developing Center of Henan Academy of Sciences, Henan Plant Natural Products Development Engineering Technology Center, Zhengzhou 450002, China)

**[Abstract]** **Objective:** To study the chemical constituents of the leaves of *Forsythia suspense*. **Method:** The compounds were isolated and purified by D101 macroporous absorptive resin, column chromatography and preparative HPLC. Their structures were identified by MS, NMR and other methods. **Result:** Ten compounds were obtained and identified as rutin (1), phillyrin (2), (+) pinoresinol- $\beta$ -D-glucoside (3), epipinoresinol- $\beta$ -D-glucoside (4), epipinoresinol-4'-O- $\beta$ -D-glucoside (5), forsythenside F (6), (+)-1-hydroxypinoresinol-4''-O- $\beta$ -D-glucopyranoside (7), forsythoside I (8), forsythoside H (9), forsythoside A (10). **Conclusion:** Compounds 4-7, 9 were isolated from the leaves of *Forsythia suspense* for the first time.

**[Key words]** leaves of *Forsythia suspense*; chemical constituents; structural identification

连翘主产于山西、河南、陕西、山东等地,具有清热解毒、消肿散结之功效。长期以来,连翘叶作为连翘的废弃物而得不到充分利用,造成资源的极大浪费。近年来的研究表明连翘叶中连翘苷和连翘酯苷的含量高于连翘果实<sup>[1]</sup>,连翘叶具有抑菌、抗氧化、抗疲劳、降脂等活性<sup>[2-5]</sup>。因此为了充分利用连翘叶资源,阐明连翘叶的药效物质基础,有必要对连翘叶的化学成分做进一步的研究,本文从连翘叶中分离到 10 个化合物,其中化合物 4,5,6,7,9 为首次从连翘叶中分离得到。

### 1 材料

Bruker DPX400 型核磁共振仪测定(四甲基硅

烷为内标,瑞士 Bruker 公司),LCQ fleet 型质谱仪(美国 Thermo Fisher 公司),LC20AT 型高效液相色谱仪(日本岛津公司),600 型半制备高效液相色谱仪(美国 Waters 公司),Sunfire C<sub>18</sub> 色谱柱(4.6 mm × 150 mm,10 mm × 150 mm,美国 Waters)。薄层色谱硅胶、柱色谱用硅胶(青岛海洋化工厂),D101 型大孔树脂(天津市光复精细化工研究所)。连翘叶购买于河南省栾川县,由河南农业大学朱长山教授鉴定为木犀科植物连翘 *Forsythia suspense* 的叶。

### 2 提取分离

取连翘叶 5 kg 加 8 倍量水,85 °C 提取 1 h,共提取 2 次,提取液过滤,减压浓缩到约 4 L,离心,上清

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液放置一段时间后有黄色物质析出,过滤,得化合物**1**(300 mg),滤液上处理好的D101型树脂柱(3 kg)柱,依次用水,40%乙醇,95%乙醇洗脱。40%乙醇洗脱液减压浓缩至干,取115 g,上硅胶柱,用三氯甲烷-甲醇(20:1~1:1)梯度洗脱,经TLC,HPLC分析,半制备高效液相色谱制备纯化后共得到9个化合物,10:1洗脱部位得到化合物**2**(2.0 g),化合物**3**(495 mg),化合物**4**(83 mg),化合物**5**(71 mg),化合物**6**(43 mg),化合物**7**(20 mg),5:1洗脱部位得到化合物**8**(36 mg),化合物**9**(9 mg),化合物**10**(35 mg)。

化合物**1** 黄色粉末。ESI-MS  $m/z$  609 [M - H]<sup>-</sup>,提示相对分子质量610,结合NMR数据,确定其分子式为C<sub>27</sub>H<sub>30</sub>O<sub>16</sub>。<sup>1</sup>H-NMR(400 MHz, DMSO)  $\delta_{\text{H}}$ :6.23(1H, d,  $J$  = 1.8 Hz, H-6), 6.40(1H, d,  $J$  = 2.2 Hz, H-8), 6.87(1H, d,  $J$  = 9 Hz, H-5'), 7.56(1H, d,  $J$  = 1.7 Hz, H-2'), 7.58(1H, dd,  $J$  = 2, 8 Hz, H-6'), 4.42(1H, d,  $J$  = 1.5 Hz, H-1Rha), 5.37(1H, d,  $J$  = 7.5 Hz, H-1Glc); <sup>13</sup>C-NMR(100 MHz, DMSO)  $\delta_{\text{C}}$ :156.8(C-2), 133.5(C-3), 177.5(C-4), 161.4(C-5), 98.8(C-6), 164.2(C-7), 93.7(C-8), 156.6(C-9), 104.1(C-10), 121.3(C-1'), 115.4(C-2'), 144.9(C-3'), 148.6(C-4'), 116.4(C-5'), 121.7(C-6'), 101.3(Glc-1), 74.2(Glc-2), 76.0(Glc-3), 70.5(Glc-4), 76.6(Glc-5), 67.2(Glc-6), 100.9(Rha-1), 70.2(Rha-2), 70.7(Rha-3), 72.0(Rha-4), 68.4(Rha-5), 17.9(Rha-6), 以上波谱数据与文献[6]芦丁数据一致,故鉴定化合物**1**为芦丁(rutin)。

化合物**2** 白色粉末。ESI-MS  $m/z$  535 [M + H]<sup>+</sup>,提示相对分子质量534。结合NMR数据,确定其分子式为C<sub>27</sub>H<sub>34</sub>O<sub>11</sub>。<sup>1</sup>H-NMR(400 MHz, DMSO)  $\delta_{\text{H}}$ :7.03(1H, br s, H-2), 7.12(H, d,  $J$  = 8.2 Hz, H-5), 6.93(1H, dd,  $J$  = 8.2, 1.4 Hz, H-6), 6.96(1H, d,  $J$  = 1.8 Hz, H-2'), 6.93(1H, m, H-5'), 6.90(1H, dd,  $J$  = 8.4, 1.8 Hz, H-6'), 4.16(1H, m, H-9a), 4.86(1H, d,  $J$  = 7.2 Hz, H-1Glc), 3.76(9H, s, 3 × OMe)。 <sup>13</sup>C-NMR(100 MHz, DMSO)  $\delta_{\text{C}}$ :135.4(C-1), 110.5(C-2), 149.1(C-3), 146.1(C-4), 115.3(C-5), 118.3(C-6), 86.9(C-7), 54.2(C-8), 70.5(C-9), 131.3(C-1'), 109.5(C-2'), 148.6(C-3'), 147.8(C-4'), 111.7(C-5'), 117.7(C-6'), 81.4(C-7'), 49.5(C-8'), 69.2(C-9'), 100.3(Glc-1), 73.4(Glc-2), 77.1(Glc-3), 69.8(Glc-4), 77.2(Glc-5), 60.9(Glc-6), 55.8(3 × OCH<sub>3</sub>)。以上波谱数据与文

献[7]连翘苷数据一致,故鉴定化合物**2**为连翘苷(phillyrin)。

化合物**3** 白色粉末。ESI-MS  $m/z$  521 [M + H]<sup>+</sup>,提示相对分子质量520。结合NMR数据,确定其分子式为C<sub>26</sub>H<sub>32</sub>O<sub>11</sub>。<sup>1</sup>H-NMR(400 MHz, DMSO)  $\delta_{\text{H}}$ :6.89(1H, d,  $J$  = 1.3 Hz, H-2), 6.71(1H, d,  $J$  = 8.1 Hz, H-5), 6.75(1H, dd,  $J$  = 8.1, 1.3 Hz, H-6), 6.95(1H, d,  $J$  = 1.8 Hz, H-2'), 7.04(1H, d,  $J$  = 8.5 Hz, H-5'), 6.85(1H, dd,  $J$  = 8.5, 1.8 Hz, H-6'), 4.60(1H, d,  $J$  = 4.0 Hz, H-7), 3.04(2H, m, H-8, 8'), 4.13(1H, m, H-9a), 3.74(1H, m, H-9b), 4.66(1H, d,  $J$  = 4.0 Hz, H-7'), 4.11(1H, m, H-9'a), 3.73(1H, m, H-9'b), 4.87(1H, d,  $J$  = 7.4 Hz, H-1Glc), 3.46~3.15  $\mu\text{m}$  sugar H), 3.75(6H, s, 2 × OMe)。 <sup>13</sup>C-NMR(100 MHz, DMSO)  $\delta_{\text{C}}$ :135.6(C-1), 110.9(C-2), 149.4(C-3), 146.4(C-4), 115.6(C-5), 118.6(C-6), 85.3(C-7), 54.2(C-8), 71.5(C-9), 132.6(C-1'), 110.8(C-2'), 148.0(C-3'), 146.3(C-4'), 115.5(C-5'), 119.1(C-6'), 85.7(C-7'), 54.0(C-8'), 71.4(C-9'), 100.5(Glc-1), 73.7(Glc-2), 77.3(Glc-3), 70.1(Glc-4), 77.5(Glc-5), 61.1(Glc-6), 56.1, 56.0(2 × OMe)。以上波谱数据与文献[8-9]报道的基本一致,故鉴定化合物**3**为(+)松脂素- $\beta$ -D-葡萄糖苷(pinoresinol- $\beta$ -D-glucoside)。

化合物**4** 白色粉末。ESI-MS  $m/z$  521 [M + H]<sup>+</sup>,提示相对分子质量520。结合NMR数据,确定其分子式为C<sub>26</sub>H<sub>32</sub>O<sub>11</sub>。<sup>1</sup>H-NMR(400 MHz, DMSO)  $\delta_{\text{H}}$ :8.91(1H, m, OH-4'), 6.96(1H, d,  $J$  = 1.6 Hz, H-2), 7.04(1H, d,  $J$  = 8.4 Hz, H-5), 6.85(1H, dd,  $J$  = 8.4, 1.6 Hz, H-6), 6.87(1H, d,  $J$  = 1.4 Hz, H-2'), 6.74(2H, br s, H-5', 6'), 4.35(1H, d,  $J$  = 6.8 Hz, H-7), 2.83(1H, m, H-8), 4.07(1H, d,  $J$  = 9.2 Hz, H-9a), 3.71(1H, m, H-9b), 4.75(1H, d,  $J$  = 5.8 Hz, H-7'), 3.34(1H, m, H-8'), 3.68(1H, m, H-9'a), 3.12(1H, m, H-9'b), 4.88(1H, d,  $J$  = 7.2 Hz, H-1Glc), 3.76(6H, s, 2 × OMe)。 <sup>13</sup>C-NMR(100 MHz, DMSO)  $\delta_{\text{C}}$ :135.7(C-1), 110.8(C-2), 149.3(C-3), 146.3(C-4), 115.6(C-5), 118.6(C-6), 87.1(C-7), 54.5(C-8), 70.7(C-9), 130.0(C-1'), 110.1(C-2'), 147.7(C-3'), 145.7(C-4'), 115.5(C-5'), 118.3(C-6'), 81.8(C-7'), 49.8(C-8'), 70.1(C-9'), 100.5(Glc-1), 73.7(Glc-2), 77.3(Glc-3), 69.4(Glc-4), 77.5(Glc-5), 61.1(Glc-6), 56.1, 56.0

(2 × OMe)。以上波谱数据与文献[8,10]报道的基本一致,故鉴定化合物4为表松脂素-β-D-葡萄糖苷(epipinoresinol-β-D-glucoside)。

化合物5 白色粉末。ESI-MS  $m/z$  521 [M + H]<sup>+</sup>,提示相对分子质量520。结合NMR数据,确定其分子式为C<sub>26</sub>H<sub>32</sub>O<sub>11</sub>。<sup>1</sup>H-NMR(400 MHz, DMSO) δ<sub>H</sub>: 8.94(1H, m, OH-4), 6.95(1H, d,  $J$  = 1.3 Hz, H-2), 7.04(1H,  $J$  = 8.4 Hz, H-5), 6.83(1H, dd,  $J$  = 8.4, 1.3 Hz, H-6), 6.89(1H, d,  $J$  = 1.4 Hz, H-2'), 6.71(1H, br s, H-5'), 6.75(1H, dd,  $J$  = 8.2, 1.4 Hz, H-6'), 4.30(1H, d,  $J$  = 7.1 Hz, H-7), 2.82(1H, m, H-8), 4.05(1H, d,  $J$  = 9.3 Hz, H-9a), 3.73(1H, m, H-9b), 4.79(1H, d,  $J$  = 5.8 Hz, H-7'), 3.15(1H, m, H-9'b), 4.88(1H, d,  $J$  = 7.2 Hz, H-1Glc), 3.76, 3.77(6H, s, 2 × OMe)。<sup>13</sup>C-NMR(100 MHz, DMSO) δ<sub>C</sub>: 132.8(C-1), 110.7(C-2), 148.0(C-3), 146.4(C-4), 115.2(C-5), 119.1(C-6), 87.4(C-7), 54.4(C-8), 70.1(C-9), 132.9(C-1'), 110.4(C-2'), 149.0(C-3'), 145.9(C-4'), 115.6(C-5'), 118.0(C-6'), 81.6(C-7'), 49.8(C-8'), 69.3(C-9'), 100.5(Glc-1), 73.7(Glc-2), 77.3(Glc-3), 70.8(Glc-4), 77.5(Glc-5), 61.1(Glc-6), 56.0, 56.1(2 × OMe)。以上波谱数据与文献[7]报道的基本一致,故鉴定化合物5为表松脂素-4'-O-β-D-葡萄糖苷(epipinoresinol-4'-O-β-D-glucoside)。

化合物6 白色粉末。ESI-MS  $m/z$  451 [M + H]<sup>+</sup>,提示相对分子质量450。结合NMR数据,确定其分子式为C<sub>22</sub>H<sub>26</sub>O<sub>10</sub>。<sup>1</sup>H-NMR(400 MHz, DMSO) δ<sub>H</sub>: 6.03(2H, d,  $J$  = 10.0 Hz, H-2, 6), 6.93(2H, dd,  $J$  = 10.0, 2.6 Hz, H-3, 5), 1.92(2H, m, H-4), 4.10(1H, d,  $J$  = 7.8 Hz, H-1'), 7.04(2H, d,  $J$  = 8.4 Hz, H-2'', 6''), 6.69(2H, d,  $J$  = 8.4 Hz, H-3'', 5''), 3.52(2H, s, H-7'')。 <sup>13</sup>C-NMR(100 MHz, DMSO) δ<sub>C</sub>: 185.3(C-1), 126.3(C-2, 6), 153.2(C-3, 5), 67.3(C-4), 39.6(C-7), 67.3(C-8), 102.7(C-1'), 73.2(C-2'), 73.5(C-3'), 70.0(C-4'), 76.3(C-5'), 63.9(C-6'), 124.3(C-1''), 130.2(C-2'', 6''), 115.0(C-3'', 5''), 156.2(C-4''), 39.6(C-7''), 171.5(C-8'')。以上波谱数据与文献[11]报道的基本一致,故鉴定化合物6为forsythenside F。

化合物7 白色粉末。ESI-MS  $m/z$  537 [M + H]<sup>+</sup>,提示相对分子质量536。结合NMR数据,确定其分子式为C<sub>26</sub>H<sub>32</sub>O<sub>12</sub>。<sup>1</sup>H-NMR(400 MHz, DMSO) δ<sub>H</sub>: 6.95(1H, d,  $J$  = 1.4 Hz, H-2), 6.72(1H,

br s, H-5), 6.76(1H, dd,  $J$  = 8.2, 1.4 Hz, H-6), 4.52(1H, s, H-7), 7.02(1H, d,  $J$  = 1.8 Hz, H-2'), 7.06(1H, d,  $J$  = 8.4 Hz, H-5'), 6.90(1H, dd,  $J$  = 8.4, 1.8 Hz, H-6'), 4.80(1H, d,  $J$  = 5.0 Hz, H-7'), 4.89(1H, d,  $J$  = 7.3 Hz, H-1Glc), 3.75, 3.77(6H, s, 2 × OMe)。 <sup>13</sup>C-NMR(100 MHz, DMSO) δ<sub>C</sub>: 135.2(C-1), 110.8(C-2), 148.8(C-3), 145.8(C-4), 115.1(C-5), 118.3(C-6), 85.0(C-7), 60.8(C-8), 70.3(C-9), 127.9(C-1'), 112.2(C-2'), 146.9(C-3'), 145.9(C-4'), 114.5(C-5'), 120.2(C-6'), 87.2(C-7'), 91.0(C-8'), 74.7(C-9'), 100.0(Glc-1), 73.2(Glc-2), 76.8(Glc-3), 69.6(Glc-4), 77.0(Glc-5), 60.6(Glc-6), 55.5, 55.6(2 × OMe)。以上波谱数据与文献[8,12]报道的基本一致,故鉴定化合物7为(+)-1-羟基松脂素-4''-O-β-D-葡萄糖苷[(+)-1-hydroxypinoresinol-4''-O-β-D-glucopyranoside]。

化合物8 紫红色粉末。ESI-MS  $m/z$  625 [M + H]<sup>+</sup>,提示相对分子质量624。结合NMR数据,确定其分子式为C<sub>29</sub>H<sub>36</sub>O<sub>15</sub>。<sup>1</sup>H-NMR(400 MHz, DMSO) δ<sub>H</sub>: 6.59(1H, d,  $J$  = 1.8 Hz, H-2), 6.62(1H, d,  $J$  = 8.0 Hz, H-5), 6.48(1H, dd,  $J$  = 8.0, 1.8 Hz, H-6), 2.65(1H, m, H-7), 4.32(1H, d,  $J$  = 7.2 Hz, H-1'), 4.56(1H, br s, H-1''), 1.14(1H, d,  $J$  = 6.6 Hz, H-6''), 7.03(1H, d,  $J$  = 1.8 Hz, H-2'''), 6.75(1H, d,  $J$  = 8.2 Hz, H-5'''), 7.01(1H, dd,  $J$  = 8.4, 1.8 Hz, H-6'''), 7.45(1H, d,  $J$  = 15.2 Hz, H-7'''), 6.25(1H, d,  $J$  = 15.2 Hz, H-8''')。 <sup>13</sup>C-NMR(100 MHz, DMSO) δ<sub>C</sub>: 129.1(C-1), 115.5(C-2), 144.7(C-3), 143.5(C-4), 116.3(C-5), 119.5(C-6), 35.1(C-7), 70.2(C-8), 102.6(C-1'), 71.4(C-2'), 77.5(C-3'), 68.1(C-4'), 75.1(C-5'), 66.4(C-6'), 100.7(C-1''), 70.6(C-2''), 70.4(C-3''), 71.9(C-4''), 68.4(C-5''), 17.8(C-6'') 125.6(C-1'''), 114.7(C-2'''), 145.5(C-3'''), 148.2(C-4'''), 115.8(C-5'''), 121.2(C-6'''), 144.9(C-7'''), 114.6(C-8'''), 166.1(C-9''')。以上波谱数据与文献[13]报道的基本一致,故鉴定化合物8为连翘酯苷I(forsythoside I)。

化合物9 紫红色粉末。ESI-MS  $m/z$  625 [M + H]<sup>+</sup>,提示相对分子质量624。结合NMR数据,确定其分子式为C<sub>29</sub>H<sub>36</sub>O<sub>15</sub>。<sup>1</sup>H-NMR(400 MHz, DMSO) δ<sub>H</sub>: 6.53(1H, d,  $J$  = 1.8 Hz, H-2), 6.55(1H, d,  $J$  = 8.4 Hz, H-5), 6.40(1H, dd,  $J$  = 8.4, 1.8 Hz,

H-6), 2.55(1H, m, H-7), 4.48(1H, d,  $J = 7.8$  Hz, H-1'), 4.60(1H, br s, H-1''), 1.14(1H, d,  $J = 6.6$  Hz, H-6''), 7.05(1H, br s, H-2'''), 6.76(1H, d,  $J = 7.2$  Hz, H-5'''), 7.00(1H, br s, H-6'''), 7.49(1H, d,  $J = 16.0$  Hz, H-7'''), 6.25(1H, d,  $J = 16.0$  Hz, H-8''')。<sup>13</sup>C-NMR(100 MHz, DMSO)  $\delta_c$ : 129.1(C-1), 115.4(C-2), 144.9(C-3), 143.4(C-4), 116.2(C-5), 119.5(C-6), 35.0(C-7), 69.8(C-8), 100.2(C-1'), 73.4(C-2'), 74.1(C-3'), 70.6(C-4'), 75.5(C-5'), 66.6(C-6'), 100.7(C-1''), 70.4(C-2''), 70.3(C-3''), 71.9(C-4''), 68.4(C-5''), 17.9(C-6''), 125.5(C-1'''), 114.8(C-2'''), 145.6(C-3'''), 148.4(C-4'''), 115.8(C-5'''), 121.2(C-6'''), 145.1(C-7'''), 114.2(C-8'''), 165.6(C-9''')。以上波谱数据与文献[13]报道的基本一致,故鉴定化合物**9**为连翘酯苷H(forsythoside H)。

化合物**10** 紫红色粉末。ESI-MS  $m/z$  625 [M+H]<sup>+</sup>,提示相对分子质量624。结合NMR数据,确定其分子式为C<sub>29</sub>H<sub>36</sub>O<sub>15</sub>。<sup>1</sup>H-NMR(400 MHz, DMSO)  $\delta_H$ : 6.65(1H, d,  $J = 1.8$  Hz, H-2), 6.63(1H, d,  $J = 7.8$  Hz, H-5), 6.50(1H, dd,  $J = 7.8, 1.8$  Hz, H-6), 2.67(1H, m, H-7), 4.30(1H, d,  $J = 7.8$  Hz, H-1'), 4.50(1H, br s, H-1''), 1.04(1H, d,  $J = 6.1$  Hz, H-6''), 7.05(1H, br s, H-2'''), 6.76(1H, d,  $J = 8.2$  Hz, H-5'''), 7.00(1H, br s, H-6'''), 7.50(1H, d,  $J = 15.6$  Hz, H-7'''), 6.25(1H, d,  $J = 15.6$  Hz, H-8''')。<sup>13</sup>C-NMR(100 MHz, DMSO)  $\delta_c$ : 129.2(C-1), 115.5(C-2), 144.9(C-3), 143.5(C-4), 116.3(C-5), 119.5(C-6), 35.1(C-7), 70.3(C-8), 102.8(C-1'), 72.9(C-2'), 73.4(C-3'), 71.0(C-4'), 73.9(C-5'), 66.0(C-6'), 100.5(C-1''), 70.5(C-2''), 70.4(C-3''), 71.9(C-4''), 68.4(C-5''), 17.8(C-6''), 125.4(C-1'''), 114.8(C-2'''), 145.6(C-3'''), 148.5(C-4'''), 115.8(C-5'''), 121.4(C-6'''), 145.6(C-7'''), 113.7(C-8'''), 165.8(C-9''')。以上波谱数据与文献[13-14]报道的基本一致,故鉴定化合物**10**为连翘酯苷A(orsythoside A)。

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