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PHYTOCHEMICAL STUDY OF STEM BARK OF *AFZELIA AFRICANA*

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*Afzelia africana* Sm. (Fabaceae) is a large deciduous tree, widespread in West Africa in dry forest areas, and is found mainly in the northern hemisphere, from Senegal to Cameroon and eastern Uganda [1]. The plant is used in folk medicine to treat various diseases. In Nigeria, *A. africana* leaves and bark are employed in unstable pregnant and miscarriage, seeds are used in sore throat [2]. Otherwise, leaves, roots, bark and mistletoes are used to treat neuropsychiatric disorders, in the Hauts Bassins region, in the western zone of Burkina Faso [3].

*A. africana* exhibits several biological activities including antimicrobial [4,5] antidiabetic and hypolipidemic effects [6]. On the other hand, a methanolic stem bark extract of *A. africana* exhibited, *in vitro*, a high activity against *Trypanosoma brucei brucei*. [7] while a 50% aqueous EtOH stem bark extract showed a low *in vitro* antiprotozoan activity against *Trypanosoma*, *Leishmania* and *Plasmodium* parasites [8]. In addition, an EtOAc extract prepared from stem bark of *A. africana* showed *in vitro* cytotoxic activity on prostate cancer and breast carcinoma cells [9].

However, very little research has been conducted on this plant. In order to explore the phytochemistry and potential bioactive molecules of *A. africana*, the stem bark have been investigated.

In the present study, we report the isolation of 15 compounds from this plant: five lignans (**4**, **9-11**, **14**) and 10 flavonoids and derivatives (**1-3**, **5-8**, **12**, **13**, **15**).

All the purified compounds are isolated for the first time from this plant.

Plant raw material was collected in the Department of Ndé, in Ouest Cameroon, in November 2014 and identified by Dr. Tan Barthelemy (National Herbarium of Yaoundé). A voucher specimen was deposited in the National Herbarium of Yaoundé (Cameroun, n°49836/HNC).

The air-dried, powdered stem barks (500 g) were extracted with methanol by percolation at room temperature. The MeOH extract was evaporated to dryness under vacuum to give a brown extract. The crude extract (6.25 g) was suspended in H<sub>2</sub>O (1.2 L) and successively partitioned with ethyl acetate and *n*-butanol. The EtOAc extract (1.4g) was initially subjected to column chromatography using polyamide to obtain prepurified fractions. The prepurified fraction were further purified by Sephadex LH-20 with elution by MeOH and by preparative HPLC, with RP-C18 column eluting with MeOH/H<sub>2</sub>O, to yield 6 compounds which were identified as **1** (12.3 mg), **2** (12.2 mg), **3** (10.0 mg), **4** (0.5 mg), **5** (2.5 mg) and **6** (5.3 mg). The BuOH extract (1.5g) was flash chromatographed over a C18 column with MeOH/H<sub>2</sub>O, the subfractions of which were separated by preparative HPLC to produce **7** and **8** (5.0 mg), **9** (11.1 mg), **10** (2.3 mg), **11** (0.4 mg), **12** (4.8 mg), **13** (0.9 mg), **14** (2.4 mg). The purification of the aqueous residual phase by flash chromatography afford **15** (1.5 mg).

The structures of these compounds (**1-15**) were identified based on  $^1\text{H}$  NMR,  $^{13}\text{C}$  NMR, 2D NMR and ESI-MS spectra and comparison with literature data and authentic samples.

**Dihydrokaempferol-7-O- $\beta$ -D-glucopyranoside (Sinensin) (1).**  $\text{C}_{21}\text{H}_{22}\text{O}_{11}$ , yellow powder. HR-ESI-MS  $m/z$  449.1097  $[\text{M}-\text{H}]^-$  (calcd. for  $\text{C}_{21}\text{H}_{21}\text{O}_{11}^-$ : 449.1089) and  $m/z$  451.1224  $[\text{M}+\text{H}]^+$  (calcd. for  $\text{C}_{21}\text{H}_{23}\text{O}_{11}^+$ : 451.1235).  $^1\text{H}$  NMR spectrum (600MHz,  $\text{CD}_3\text{OD}$ ,  $\delta$ , ppm,  $J/\text{Hz}$ ): 5.02 (1H, d,  $J = 11.8$ , H-2), 4.60 (1H, d,  $J = 11.8$ , H-3), 6.23 (1H, d,  $J = 2.1$ , H-6), 6.20 (1H, d,  $J = 2.1$ , H-8), 7.36 (2H, d,  $J = 8.3$ , H-2', 6'), 6.83 (2H, d,  $J = 8.3$ , H-3', 5'), 4.97 (1H, d,  $J = 7.1$ , H-1"), 3.45 (1H, m, H-2"), 3.45 (1H, m, H-3"), 3.39 (1H, m, H-4"), 3.45 (1H, m, H-5"), 3.87 (1H, m, H-6"a), 3.68 (1H, m, H-6"b).  $^{13}\text{C}$  NMR spectrum (150 MHz,  $\text{CD}_3\text{OD}$ ,  $\delta$ , ppm): 85.1 (C-2), 73.7 (C-3), 199.5 (C-4), 164.9 (C-5), 98.3 (C-6), 167.4 (C-7), 97.0 (C-8), 165.4 (C-9), 103.5 (C-10), 129.0 (C-1'), 130.4 (C-2', 6'), 116.1 (C-3', 5'), 159.0 (C-4'), 101.1 (C-1"), 74.7 (C-2"), 77.8 (C-3"), 71.1 (C-4"), 78.0 (C-5"), 62.3 (C-6") [10].

**Isosinensin (2).**  $\text{C}_{21}\text{H}_{22}\text{O}_{11}$ , yellow powder. HR-ESI-MS  $m/z$  449.1090  $[\text{M}-\text{H}]^-$  (calcd. for  $\text{C}_{21}\text{H}_{21}\text{O}_{11}^-$ : 449.1089) and  $m/z$  451.1225  $[\text{M}+\text{H}]^+$  (calcd. for  $\text{C}_{21}\text{H}_{23}\text{O}_{11}^+$ : 451.1235).  $^1\text{H}$  NMR spectrum (600MHz,  $\text{CD}_3\text{OD}$ ,  $\delta$ , ppm,  $J/\text{Hz}$ ): 5.39 (1H, d,  $J = 2.1$ , H-2), 4.22 (1H, d,  $J = 2.1$ , H-3), 6.27 (1H, d,  $J = 2.1$ , H-6), 6.21 (1H, d,  $J = 2.1$ , H-8), 7.35 (2H, d,  $J = 8.3$ , H-2', 6'), 6.79 (2H, d,  $J = 8.3$ , H-3', 5'), 5.00 (1H, d,  $J = 7.1$ , H-1"), 3.45 (1H, m, H-2"), 3.45 (1H, m, H-3"), 3.39 (1H, m, H-4"), 3.45 (1H, m, H-5"), 3.87 (1H, m, H-6"a), 3.68 (1H, m, H-6"b).  $^{13}\text{C}$  NMR spectrum (150 MHz,  $\text{CD}_3\text{OD}$ ,  $\delta$ , ppm): 83.1 (C-2), 73.1 (C-3), 197.2 (C-4), 164.9 (C-5), 97.0 (C-6), 167.3 (C-7), 98.1 (C-8), 164.1 (C-9), 103.3 (C-10), 128.3 (C-1'), 129.8 (C-2', 6'), 115.9 (C-3', 5'), 158.6 (C-4'), 101.1 (C-1"), 74.7 (C-2"), 77.8 (C-3"), 71.1 (C-4"), 78.0 (C-5"), 62.3 (C-6") [11].

**Kaempferol-7-O- $\beta$ -D-glucopyranoside (3).**  $\text{C}_{21}\text{H}_{20}\text{O}_{11}$ , yellow powder. HR-ESI-MS  $m/z$  447.0939  $[\text{M}-\text{H}]^-$  (calcd. for  $\text{C}_{21}\text{H}_{19}\text{O}_{11}^-$ : 447.0933) and  $m/z$  449.1069  $[\text{M}+\text{H}]^+$  (calcd. for  $\text{C}_{21}\text{H}_{21}\text{O}_{11}^+$ : 449.1078).  $^1\text{H}$  NMR spectrum (600MHz,  $\text{CD}_3\text{OD}$ ,  $\delta$ , ppm,  $J/\text{Hz}$ ): 6.47 (1H, d,  $J = 2.0$ , H-6), 6.79 (1H, d,  $J = 2.0$ , H-8), 8.14 (2H, d,  $J = 8.7$ , H-2', 6'), 6.92 (2H, d,  $J = 8.7$ , H-3', 5'), 5.06 (1H, d,  $J = 7.2$ , H-1"), 3.50 (1H, m, H-2"), 3.50 (1H, m, H-3"), 3.40 (1H, brt,  $J = 9.1$ , H-4"), 3.58 (1H, ddd,  $J = 9.1, 6.0, 2.0$ , H-5"), 3.93 (1H, dd,  $J = 12.0, 2.0$ , H-6"a), 3.72 (1H, dd,  $J = 12.0, 6.0$ , H-6"b).  $^{13}\text{C}$  NMR spectrum (150 MHz,  $\text{CD}_3\text{OD}$ ,  $\delta$ , ppm): 148.7 (C-2), 137.5 (C-3), 179.8 (C-4), 162.0 (C-5), 100.2 (C-6), 167.4 (C-7), 95.5 (C-8), 157.7 (C-9), 106.3 (C-10), 123.6 (C-1'), 130.9 (C-2', 6'), 116.4 (C-3', 5'), 160.6 (C-4'), 101.7 (C-1"), 74.8 (C-2"), 77.9 (C-3"), 71.3 (C-4"), 78.4 (C-5"), 62.5 (C-6") [12].

**2,3-trans-dihydrodehydrodiconiferyl alcohol (4).**  $\text{C}_{20}\text{H}_{24}\text{O}_6$ , colorless oil. HR-ESI-MS  $m/z$  359.1489  $[\text{M}-\text{H}]^-$  (calcd. for  $\text{C}_{20}\text{H}_{23}\text{O}_6^-$ : 359.1500) and  $m/z$  361.1633  $[\text{M}+\text{H}]^+$  (calcd. for  $\text{C}_{20}\text{H}_{25}\text{O}_6^+$ : 361.1646).  $^1\text{H}$  NMR spectrum (600MHz,  $\text{CD}_3\text{OD}$ ,  $\delta$ , ppm,  $J/\text{Hz}$ ): 6.95 (1H, d,  $J = 1.8$ , H-2), 6.76 (1H, d,  $J = 8.1$ , H-5), 6.83 (1H, dd,  $J = 8.1, 1.8$ , H-6), 5.49 (1H, d,  $J = 6.2$ , H-7), 3.46 (1H, brq,  $J =$

6.5, H-8), 3.83 (1H, dd,  $J = 11.0, 5.0$ , H-9a), 3.75 (1H, dd,  $J = 11.0, 7.0$ , H-9b), 3.82 (3H, s, H-10), 6.73 (1H, brs, H-2'), 6.73 (1H, brs, H-6'), 2.63 (2H, t,  $J = 7.7$ , H-7'), 1.82 (2H, m, H-8'), 3.57 (2H, t,  $J = 6.5$ , H-9'), 3.86 (3H, s, H-10').  $^{13}\text{C}$  NMR spectrum (150 MHz,  $\text{CD}_3\text{OD}$ ,  $\delta$ , ppm): 134.4 (C-1), 110.2 (C-2), 149.1 (C-3), 147.0 (C-4), 115.8 (C-5), 119.3 (C-6), 88.6 (C-7), 55.0 (C-8), 64.7 (C-9), 56.1 (C-10), 136.6 (C-1'), 113.8 (C-2'), 144.9 (C-3'), 147.2 (C-4'), 134.5 (C-5'), 117.7 (C-6'), 32.6 (C-7'), 35.5 (C-8'), 62.0 (C-9'), 56.4 (C10') [13].

**2R,3R-trans-aromadendrin-7-O- $\beta$ -D-glucopyranoside-6''-(4'''-hydroxy-2'''-methylenebutanoate) (5).**  $\text{C}_{26}\text{H}_{28}\text{O}_{13}$ , yellow powder. HR-ESI-MS  $m/z$  547.1458  $[\text{M}-\text{H}]^-$  (calcd. for  $\text{C}_{26}\text{H}_{27}\text{O}_{13}^-$ : 547.1457) and  $m/z$  549.1588  $[\text{M}+\text{H}]^+$  (calcd. for  $\text{C}_{26}\text{H}_{29}\text{O}_{13}^+$ : 549.1603).  $^1\text{H}$  NMR spectrum (600MHz,  $\text{CD}_3\text{OD}$ ,  $\delta$ , ppm,  $J/\text{Hz}$ ): 5.02 (1H, d,  $J = 11.8$ , H-2), 4.60 (1H, d,  $J = 11.8$ , H-3), 6.20 (1H, d,  $J = 2.2$ , H-6), 6.20 (1H, d,  $J = 2.2$ , H-8), 7.38 (2H, d,  $J = 8.6$ , H-2', 6'), 6.86 (2H, d,  $J = 8.6$ , H-3', 5'), 4.96 (1H, d,  $J = 7.5$ , H-1''), 3.45 (1H, m, H-2''), 3.47 (1H, m, H-3''), 3.34 (1H, dd,  $J = 9.8, 8.7$ , H-4''), 3.74 (1H, ddd,  $J = 9.8, 7.8, 2.0$ , H-5''), 4.56 (1H, dd,  $J = 12.0, 2.0$ , H-6''a), 4.15 (1H, dd,  $J = 12.0, 7.8$ , H-6''b), 2.44 (1H, brdt,  $J = 14.1, 6.7$ , H-3'''a), 2.40 (1H, brdt,  $J = 14.1, 6.7$ , H-3'''b), 3.56 (2H, t,  $J = 6.7$ , H-4'''), 6.19 (1H, brq,  $J = 1.2$ , H-5'''a), 5.39 (1H, brq,  $J = 1.2$ , H-5'''b).  $^{13}\text{C}$  NMR spectrum (150 MHz,  $\text{CD}_3\text{OD}$ ,  $\delta$ , ppm): 84.9 (C-2), 73.6 (C-3), 199.1 (C-4), 164.1 (C-5), 98.1 (C-6), 166.9 (C-7), 96.5 (C-8), 164.1 (C-9), 103.3 (C-10), 129.0 (C-1'), 130.2 (C-2'), 115.9 (C-3'), 159.1 (C-4'), 115.9 (C-5'), 130.2 (C-6'), 100.9 (C-1''), 74.3 (C2''), 77.4 (C3''), 71.4 (C-4''), 75.4 (C-5''), 65.0 (C-6''), 168.0 (C-1'''), 137.8 (C-2'''), 36.0 (C-3'''), 61.2 (C-4'''), 128.2 (C-5''') [14].

**Dihydrokaempferol (6).**  $\text{C}_{15}\text{H}_{12}\text{O}_6$ , yellow powder. HR-ESI-MS  $m/z$  287.0563  $[\text{M}-\text{H}]^-$  (calcd. for  $\text{C}_{15}\text{H}_{11}\text{O}_6^-$ : 287.0561) and  $m/z$  289.0696  $[\text{M}+\text{H}]^+$  (calcd. for  $\text{C}_{15}\text{H}_{13}\text{O}_6^+$ : 289.0707).  $^1\text{H}$  NMR spectrum (600MHz,  $\text{CD}_3\text{OD}$ ,  $\delta$ , ppm,  $J/\text{Hz}$ ): 4.95 (1H, d,  $J = 11.5$ , H-2), 4.51 (1H, d,  $J = 11.5$ , H-3), 5.88 (1H, brs, H-6), 5.84 (1H, brs, H-8), 7.35 (2H, d,  $J = 8.5$ , H-2', 6'), 6.83 (2H, d,  $J = 8.5$ , H-3', 5').  $^{13}\text{C}$  NMR spectrum (150 MHz,  $\text{CD}_3\text{OD}$ ,  $\delta$ , ppm): 85.9 (C-2), 73.6 (C-3), 197.8 (C-4), 163.4 (C-5), 97.9 (C-6), 171.1 (C-7), 96.9 (C-8), 154.4 (C-9), 101.3 (C-10), 129.5 (C-1'), 130.3 (C-2', 6'), 116.1 (C-3', 5'), 159.2 (C-4') [15].

**Taxifolin-7-O- $\beta$ -D-glucopyranoside (7).**  $\text{C}_{21}\text{H}_{22}\text{O}_{12}$ , brown amorphous powder. HR-ESI-MS  $m/z$  465.1031  $[\text{M}-\text{H}]^-$  (calcd. for  $\text{C}_{21}\text{H}_{21}\text{O}_{12}^-$ : 465.1039) and  $m/z$  467.1171  $[\text{M}+\text{H}]^+$  (calcd. for  $\text{C}_{21}\text{H}_{23}\text{O}_{12}^+$ : 467.1184).  $^1\text{H}$  NMR spectrum (600MHz,  $\text{CD}_3\text{OD}$ , d, ppm,  $J/\text{Hz}$ ): 4.96 (1H, d,  $J = 11.1$ , H-2), 4.57 (1H, d,  $J = 11.1$ , H-3), 6.23 (1H, d,  $J = 2.0$ , H-6), 6.20 (1H, d,  $J = 2.0$ , H-8), 6.97 (2H, brs, H-2'), 6.80 (1H, d,  $J = 8.1$ , H-5'), 6.85 (1H, dd,  $J = 8.2, 1.6$ , H-6'), 4.97 (1H, d,  $J = 7.7$ , H-1''), 3.45 (3H, m, H-2'', 3'', 5''), 3.39 (1H, brt,  $J = 8.7$ , H-4''), 3.87 (1H, dd,  $J = 12.1, 1.8$ , H-6''a), 3.68 (1H, dd,  $J = 12.1, 6.0$ , H-6''b).  $^{13}\text{C}$  NMR spectrum (150 MHz,  $\text{CD}_3\text{OD}$ , d, ppm): 85.3 (C-2), 73.8 (C-3), 199.2 (C-4), 164.9 (C-5), 98.3 (C-6), 167.4 (C-7), 97.0 (C-8), 164.3 (C-9), 103.5 (C-10), 129.6 (C-1'), 116.0

(C-2'), 146.7 (C-3'), 147.1 (C-4'), 116.1 (C-5'), 121.0 (C-6'), 101.2 (C-1''), 74.6 (C-2''), 77.8 (C-3''), 71.2 (C-4''), 78.1 (C-5''), 62.2 (C-6'') [16].

**Catechin (8).** C<sub>15</sub>H<sub>14</sub>O<sub>6</sub>, white powder, HR-ESI-MS *m/z* 289.0715 [M-H]<sup>-</sup> (calcd. for C<sub>15</sub>H<sub>13</sub>O<sub>6</sub><sup>-</sup>: 289.0718) and *m/z* 291.0856 [M+H]<sup>+</sup> (calcd. for C<sub>15</sub>H<sub>15</sub>O<sub>6</sub><sup>+</sup>: 291.0863). The compound is identical to the authentic sample [17].

**(+)-Lyoniside (9).** C<sub>27</sub>H<sub>36</sub>O<sub>12</sub>, white crystals. [*a*]<sub>D</sub><sup>25</sup> = +34° (*c* 2.24; MeOH). HR-ESI-MS *m/z* 551.2127 [M-H]<sup>-</sup> (calcd. for C<sub>27</sub>H<sub>35</sub>O<sub>12</sub><sup>-</sup>: 551.2134) and *m/z* 553.2269 [M+H]<sup>+</sup> (calcd. for C<sub>27</sub>H<sub>37</sub>O<sub>12</sub><sup>+</sup>: 553.2280). <sup>1</sup>H NMR spectrum (600MHz, CD<sub>3</sub>OD, δ, ppm, *J*/Hz): 4.38 (1H, d, *J* = 6.6, H-1), 2.05 (1H, m, H-2), 3.84 (1H, dd, *J* = 9.8, 5.3, H-2aa), 3.42 (1H, dd, *J* = 9.8, 4.0, H-2ab), 1.70 (1H, m, H-3), 3.65 (1H, dd, *J* = 11.0, 4.3, H-3aa), 3.55 (1H, dd, *J* = 11.0, 6.5, H-3ab), 2.71 (1H, dd, *J* = 15.3, 4.7, H-4a), 2.63 (1H, dd, *J* = 15.3, 11.6, H-4b), 6.57 (1H, s, H-5), 6.43 (2H, s, H-2', 6'), 4.21 (1H, d, *J* = 7.6, H-1''), 3.22 (1H, dd, *J* = 9.0, 7.6, H-2''), 3.31 (1H, m, H-3''), 3.47 (1H, ddd, *J* = 10.4, 8.8, 5.3, H-4''), 3.83 (1H, dd, *J* = 11.5, 5.3, H-5''a), 3.17 (1H, dd, *J* = 11.5, 10.4, H-5''b), 3.33 (3H, s, 3-OCH<sub>3</sub>), 3.85 (3H, s, 6-OCH<sub>3</sub>), 3.75 (6H, s, 3', 5'-OCH<sub>3</sub>). <sup>13</sup>C NMR spectrum (150 MHz, CD<sub>3</sub>OD, δ, ppm): 43.0 (C-1), 46.7 (C-2), 70.9 (C-2a), 40.4 (C-3), 66.0 (C-3a), 33.9 (C-4), 130.1 (C-4a), 107.8 (C-5), 148.6 (C-6), 138.9 (C-7), 147.6 (C-8), 126.1 (C-8a), 139.4 (C-1'), 106.9 (C-2', 6'), 149.0 (C-3', 5'), 134.4 (C-4'), 105.5 (C-1''), 75.0 (C-2''), 78.0 (C-3''), 71.2 (C-4''), 67.0 (C-5''), 56.5 (6-OCH<sub>3</sub>), 60.0 (8-OCH<sub>3</sub>), 56.8 (3', 5'-OCH<sub>3</sub>) [18].

**(-)-Nudiposide (10).** C<sub>27</sub>H<sub>36</sub>O<sub>12</sub>, white crystals. [*a*]<sub>D</sub><sup>25</sup> = -52° (*c* 0.64; MeOH). HR-ESI-MS *m/z* 551.2130 [M-H]<sup>-</sup> (calcd. for C<sub>27</sub>H<sub>35</sub>O<sub>12</sub><sup>-</sup>: 551.2134) and *m/z* 553.2276 [M+H]<sup>+</sup> (calcd. for C<sub>27</sub>H<sub>37</sub>O<sub>12</sub><sup>+</sup>: 553.2280). <sup>1</sup>H NMR spectrum (600MHz, CD<sub>3</sub>OD, δ, ppm, *J*/Hz): 4.23 (1H, d, 7.0, H-1), 2.03 (1H, m, H-2), 3.81 (1H, dd, *J* = 10.1, 4.6, H-2aa), 3.58 (1H, dd, *J* = 10.1, 4.7, H-2ab), 1.71 (1H, m, H-3), 3.63 (1H, dd, *J* = 11.1, 4.4, H-3aa), 3.60 (1H, dd, *J* = 11.1, 6.1, H-3ab), 2.71 (1H, dd, *J* = 15.4, 5.5, H-4a), 2.67 (1H, dd, *J* = 15.4, 10.4, H-4b), 6.57 (1H, s, H-5), 6.41 (2H, s, H-2', 6'), 4.09 (1H, d, *J* = 7.6, H-1''), 3.19 (1H, dd, *J* = 9.0, 7.7, H-2''), 3.27 (1H, t, *J* = 9.0, H-3''), 3.49 (1H, ddd, *J* = 10.6, 9.0, 5.4, H-4''), 3.86 (1H, dd, *J* = 11.4, 5.4, H-5''a), 3.13 (1H, dd, *J* = 11.4, 10.6, H-5''b), 3.86 (3H, s, 6-OCH<sub>3</sub>), 3.30 (3H, s, 8-OCH<sub>3</sub>), 3.75 (6H, s, 3', 5'-OCH<sub>3</sub>). <sup>13</sup>C NMR spectrum (150 MHz, CD<sub>3</sub>OD, δ, ppm): 43.1 (C-1), 46.6 (C-2), 70.8 (C-2a), 40.3 (C-3), 65.8 (C-3a), 33.7 (C-4), 129.7 (C-4a), 107.5 (C-5), 148.4 (C-6), 138.6 (C-7), 147.3 (C-8), 126.1 (C-8a), 139.4 (C-1'), 106.7 (C-2', 6'), 148.7 (C-3', 5'), 134.1 (C-4'), 104.7 (C-1''), 74.7 (C-2''), 77.7 (C-3''), 70.9 (C-4''), 66.8 (C-5''), 59.7 (8-OCH<sub>3</sub>), 56.3 (6-OCH<sub>3</sub>), 56.4 (3', 5'-OCH<sub>3</sub>) [18].

**β-D-Xylopyranoside, [(2*R*,3*S*,4*R*)-1,2,3,4-tetrahydro-6-hydroxy-4-(4-hydroxy-3,5-dimethoxyphenyl)-3-(hydroxymethyl)-7-methoxy-2-naphthalenyl]methyl (11).** C<sub>26</sub>H<sub>34</sub>O<sub>11</sub>, white crystals. HR-ESI-MS: *m/z* 521.2032 [M-H]<sup>-</sup> (calcd. for C<sub>26</sub>H<sub>33</sub>O<sub>11</sub><sup>-</sup>: 521.2028) and *m/z*

553.2153 [M+H]<sup>+</sup> (calcd. for C<sub>26</sub>H<sub>35</sub>O<sub>11</sub><sup>+</sup>: 523.2174). <sup>1</sup>H NMR spectrum (600MHz, CD<sub>3</sub>OD, δ, ppm, J/Hz): 4.09 (1H, m, H-1), 1.86 (1H, brtt, J = 10.5, 2.8, H-2), 4.00 (1H, dd, J = 9.8, 2.5, H-2aa), 3.21 (1H, m, H-2ab), 2.08 (1H, m, H-3), 3.77 (1H, dd, J = 11.0, 3.9, H-3aa), 3.75 (1H, dd, J = 11.0, 6.0, H-3ab), 2.85 (1H, dd, J = 16.0, 10.8, H-4a), 2.80 (1H, dd, J = 16.0, 5.4, H-4b), 6.65 (1H, s, H-5), 6.20 (1H, s, H-8), 6.50 (2H, s, H-2', 6'), 4.06 (1H, d, J = 7.6, H-1''), 3.20 (1H, m, H-2''), 3.27 (1H, t, J = 9.0, H-3''), 3.45 (1H, ddd, J = 10.2, 8.6, 5.4, H-4''), 3.81 (1H, m, H-5''a), 3.12 (1H, dd, J = 11.5, 10.3, H-5''b), 3.81 (3H, s, 6-OCH<sub>3</sub>), 3.79 (6H, s, 3', 5'-OCH<sub>3</sub>). <sup>13</sup>C NMR spectrum (150 MHz, CD<sub>3</sub>OD, δ, ppm): 48.2 (C-1), 45.6 (C-2), 69.0 (C-2a), 39.3 (C-3), 64.8 (C-3a), 33.7 (C-4), 129.0 (C-4a), 112.2 (C-5), 147.1 (C-6), 145.0 (C-7), 117.1 (C-8), 133.8 (C-8a), 137.6 (C-1'), 107.6 (C-2', 6'), 149.0 (C-3', 5'), 134.7 (C-4'), 105.7 (C-1''), 74.7 (C-2''), 77.7 (C-3''), 70.9 (C-4''), 66.8 (C-5''), 56.2 (6-OCH<sub>3</sub>), 56.5 (3', 5'-OCH<sub>3</sub>) [19].

**Kaempferol-3-O-neohesperidoside-7-O-α-rhamnopyranoside (12).** C<sub>33</sub>H<sub>40</sub>O<sub>19</sub>, yellow powder. HR-ESI-MS *m/z* 739.2090 [M-H]<sup>-</sup> (calcd. for C<sub>33</sub>H<sub>39</sub>O<sub>19</sub><sup>-</sup>: 739.2091) and *m/z* 741.2227 [M+H]<sup>+</sup> (calcd. for C<sub>33</sub>H<sub>41</sub>O<sub>19</sub><sup>+</sup>: 741.2237). <sup>1</sup>H NMR spectrum (600MHz, CD<sub>3</sub>OD, δ, ppm, J/Hz): 6.45 (1H, d, J = 2.1, H-6), 6.76 (1H, d, J = 2.1, H-8), 8.08 (2H, d, J = 8.9, H-2', 6'), 6.90 (2H, d, J = 8.9, H-3', 5'), 5.56 (1H, d, J = 1.3, H-1''), 4.02 (1H, m, H-2''), 3.82 (1H, dd, J = 9.1, 3.5, H-3''), 3.48 (1H, t, J = 9.2, H-4''), 3.58 (1H, dq, J = 9.3, 6.1, H-5''), 1.25 (3H, d, J = 6.1, H-6''), 5.76 (1H, d, J = 7.7, H-1'''), 3.62 (1H, dd, J = 9.1, 8.0, H-2'''), 3.55 (1H, brt, J = 9.1, H-3'''), 3.28 (1H, dd, J = 9.6, 8.7, H-4'''), 3.23 (1H, ddd, J = 9.6, 6.1, 2.2, H-5'''), 3.73 (1H, dd, J = 12.1, 2.2, H-6''a), 3.48 (1H, dd, J = 12.1, 6.1, H-6''b), 5.23 (1H, d, J = 1.2, H-1'''), 4.00 (1H, m, H-2'''), 3.77 (1H, dd, J = 9.6, 3.4, H-3'''), 3.34 (1H, t, J = 9.6, H-4'''), 4.03 (1H, m, H-5'''), 0.95 (3H, d, J = 6.3, H-6'''). <sup>13</sup>C NMR spectrum (150 MHz, CD<sub>3</sub>OD, δ, ppm): 159.2 (C-2), 131.8 (C-3), 180.0 (C-4), 163.5 (C-5), 100.5 (C-6), 163.2 (C-7), 95.4 (C-8), 158.0 (C-9), 107.4 (C-10), 123.0 (C-1'), 132.1 (C-2', 6'), 116.1 (C-3', 5'), 161.3 (C-4'), 99.8 (C-1''), 71.8 (C-2''), 72.1 (C-3''), 73.5 (C-4''), 71.4 (C-5''), 18.1 (C-6''), 100.2 (C-1'''), 80.1 (C-2'''), 79.0 (C-3'''), 71.9 (C-4'''), 78.6 (C-5'''), 62.6 (C-6'''), 102.7 (C-1'''), 72.4 (C-2'''), 72.4 (C-3'''), 74.0 (C-4'''), 69.9 (C-5'''), 17.5 (C-6''') [20].

**Dunnianoside D (13).** C<sub>20</sub>H<sub>22</sub>O<sub>10</sub>, white powder. HR-ESI-MS *m/z* 421.1139 [M-H]<sup>-</sup> (calcd. for C<sub>20</sub>H<sub>21</sub>O<sub>10</sub><sup>-</sup>: 421.1140) and *m/z* 423.1286 [M+H]<sup>+</sup> (calcd. for C<sub>20</sub>H<sub>23</sub>O<sub>10</sub><sup>+</sup>: 423.1286). <sup>1</sup>H NMR spectrum (600MHz, CD<sub>3</sub>OD, δ, ppm, J/Hz): 6.71 (1H, d, J = 2.3, H-2), 6.59 (1H, d, J = 8.6, H-5), 6.57 (1H, dd, J = 8.6, 2.3, H-6), 4.76 (1H, d, J = 7.6, H-1'), 3.45 (1H, m, H-2'), 3.47 (1H, m, H-3'), 3.41 (1H, m, H-4'), 3.72 (1H, m, H-5'), 4.68 (1H, dd, J = 11.9, 2.1, H-6'a), 4.35 (1H, dd, J = 11.9, 7.3, H-6'b), 7.88 (2H, d, J = 8.9, H-2'', 6''), 6.84 (2H, d, J = 8.9, H-3'', 5''). <sup>13</sup>C NMR spectrum (150 MHz, CD<sub>3</sub>OD, δ, ppm): 152.6 (C-1), 104.0 (C-2), 149.1 (C-3), 143.1 (C-4), 116.0 (C-5), 110.1 (C-6), 103.7

(C-1'), 75.0 (C-2'), 78.0 (C-3'), 72.0 (C-4'), 75.6 (C-5'), 64.9 (C-6'), 122.5 (C-1''), 132.8 (C-2'', 6''), 116.9 (C-3'', 5''), 163.8 (C-4'') [21].

**Ssioriside (14).** C<sub>27</sub>H<sub>38</sub>O<sub>12</sub>, colorless powder. HR-ESI-MS  $m/z$  553.2292 [M-H]<sup>-</sup> (calcd. for C<sub>27</sub>H<sub>37</sub>O<sub>12</sub><sup>-</sup>: 553.2291) and  $m/z$  555.2418 [M+H]<sup>+</sup> (calcd. for C<sub>27</sub>H<sub>39</sub>O<sub>12</sub><sup>+</sup>: 555.2436). <sup>1</sup>H NMR spectrum (600MHz, CD<sub>3</sub>OD,  $\delta$ , ppm,  $J$ /Hz): 6.35 (2H, s, H-2, 6), , 2.63 (1H, dd,  $J$  = 13.5, 6.8, H-7a), 2.57 (1H, dd,  $J$  = 11.7, 8.6, H-7b), 1.94 (1H, m, H-8), 3.68 (1H, dd,  $J$  = 11.1, 5.7, H-9a), 3.55 (1H, m, H-9b), 6.33 (2H, s, H-2', 6'), 2.70 (1H, dd,  $J$  = 13.6, 6.9, H-7'a), 2.53 (1H, t,  $J$  = 13.6, H-7'b), 2.07 (1H, m, H-8'), 3.98 (1H, dd,  $J$  = 9.4, 5.7, H-9'a), 3.46 (1H, m, H-9'b), 4.19 (1H, d,  $J$  = 7.5, H-1''), 3.20 (1H, dd,  $J$  = 9.1, 7.6, H-2''), 3.30 (1H, m, H-3''), 3.47 (1H, m, H-4''), 3.85 (1H, dd,  $J$  = 11.5, 5.5, H-5''a), 3.18 (1H, dd,  $J$  = 11.4, 10.5, H-5''b), 3.75 (6H, s, 3, 5-OCH<sub>3</sub>), 3.75 (6H, s, 3', 5'-OCH<sub>3</sub>). <sup>13</sup>C NMR spectrum (150 MHz, CD<sub>3</sub>OD,  $\delta$ , ppm): 133.0 (C-1), 107.0 (C-2), 148.7 (C-3), 134.0 (C-4), 148.7 (C-5), 107.0 (C-6), 35.8 (C-7), 43.7 (C-8), 62.4 (C-9), 132.8 (C-1'), 106.8 (C-2', 6'), 148.7 (C-3', 5'), 134.0 (C-4'), 36.0 (C-7'), 41.1 (C-8'), 70.6 (C-9'), 105.1 (C-1''), 74.8 (C-2''), 77.7 (C-3''), 71.0 (C-4''), 66.8 (C-5''), 56.3 (3,5-OCH<sub>3</sub>), 56.3 (3', 5'-OCH<sub>3</sub>) [22].

**Luteolin (15).** C<sub>15</sub>H<sub>10</sub>O<sub>6</sub>, yellow powder, HR-ESI-MS  $m/z$  285.0406 [M-H]<sup>-</sup> (calcd. for C<sub>15</sub>H<sub>9</sub>O<sub>6</sub><sup>-</sup>: 285.0405) and  $m/z$  287.0537 [M+H]<sup>+</sup> (calcd. for C<sub>15</sub>H<sub>11</sub>O<sub>6</sub><sup>+</sup>: 287.0550). The compound is identical to the authentic sample [23].

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