FLAVONOIDS FROM *Gossypium hirsutum* FLOWERS

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UDC 547.972

Gossypium hirsutum L. (Malvaceae) is a cotton species that is widely cultivated worldwide. In Xinjiang Autonomous Region (PRC), flowers of *G. hirsutum* are used in folk medicine. The plant is officially listed as a Chinese Medicinal Herb and is used as a sedative agent and to treat delayed mental development and to improve brain function [1]. We studied the flavonoid composition of flowers of *G. hirsutum*.

Flowers of *G. hirsutum* (2 kg) were extracted with ethanol (95%, 3 times). The extracts were combined and evaporated to dryness at reduced pressure to afford dry powder (400 g) that was fractionated over a column of ion-exchange resin AB-8 (weakly polar). Compounds were eluted from the column by water and aqueous ethanol (50 and 70%). The alcohol fractions (90 g) were rechromatographed over a column of silica gel to produce **1**-**7**.

Kaempferol-3-*O*-β-D-(6″-*O*-*p*-coumaryl)glycoside (1), light-yellow powder, mp 246-248°C (MeOH). UV spectrum (λ_{max}, MeOH, nm): 226, 268, 316, 352sh.

PMR spectrum (600 MHz, DMSO-d_6, δ, ppm, J/Hz): 5.46 (1H, d, J = 7.8, H-1″), 6.12 (1H, d, J = 16.2, H-8‴), 6.16 (1H, d, J = 1.8, H-6), 6.39 (1H, d, J = 1.8, H-8), 6.79 (2H, d, J = 8.4, H-3‴,5‴), 6.86 (2H, d, J = 9.0, H-3′,5′), 7.35 (1H, d, J = 16.2, H-7‴), 7.38 (2H, d, J = 8.4, H-2‴,6‴), 8.00 (2H, d, J = 9.0, H-2′,6′), 10.03, 10.17 (1H each, br.s, OH on C-4‴ and C-4′, respectively), 10.86 (1H, br.s, 7-OH), 12.59 (1H, s, 5-OH).

^{13}C NMR spectrum (150 MHz, DMSO-d_6, δ, ppm): 177.6 (C-4), 166.4 (C-9‴), 164.4 (C-7), 161.4 (C-5), 160.2 (C-4′), 160.0 (C-4‴), 156.6 (C-2), 156.5 (C-9), 144.8 (C-7‴), 133.2 (C-3), 131.0 (C-2′,6′), 130.4 (C-2‴,6‴), 125.1 (C-1‴), 121.0 (C-1′), 116.0 (C-3′,5′), 115.3 (C-3‴,5‴), 113.8 (C-8‴), 104.1 (C-10), 101.1 (C-1″), 99.0 (C-6), 93.9 (C-8), 76.4 (C-3′), 74.4 (C-5″), 74.3 (C-2″), 70.1 (C-4″), 63.2 (C-6″) [2].

Quercetin (2), yellow powder, mp >300°C (acetone). UV spectrum (λ_{max}, MeOH, nm): 256, 374.

PMR spectrum (600 MHz, DMSO-d_6, δ, ppm, J/Hz): 6.19 (1H, d, J = 1.8, H-6), 6.41 (1H, d, J = 2.4, H-8), 6.88 (1H, d, J = 8.4, H-5′), 7.55 (1H, q, J = 8.4, 2.4, H-6′), 7.68 (1H, d, J = 2.4, H-2′), 9.32 (1H, s, 4′-OH), 9.39 (1H, s, 3-OH), 9.61 (1H, s, 3′-OH), 10.79 (1H, s, 7-OH), 12.50 (1H, s, 5-OH).

^{13}C NMR spectrum (150 MHz, DMSO-d_6, δ, ppm): 93.55 (C-8), 98.38 (C-6), 103.21 (C-10), 115.80 (C-2′), 116.39 (C-5′), 120.17 (C-6′), 122.15 (C-1′), 135.95 (C-3), 145.26 (C-3′), 146.99 (C-2), 147.91 (C-4′), 156.32 (C-9), 160.92 (C-5), 164.09 (C-7), 176.05 (C-4) [3].

Quercetin-3′-*O*-β-D-glucoside (3), yellow powder, mp 183-184°C (MeOH). UV spectrum (λ_{max}, MeOH, nm): 252, 370.

PMR spectrum (600 MHz, DMSO-d_6, δ, ppm, J/Hz): 3.17-5.09 (sugar protons), 5.15 (1H, d, J = 4.2, H-1″), 6.19 (1H, d, J = 1.8, H-6), 6.49 (1H, d, J = 1.8, H-8), 6.98 (1H, d, J = 8.4, H-5′), 7.85 (1H, q, J = 8.4, 2.4, H-6′), 7.96 (1H, d, J = 1.8, H-2′), 9.22 (1H, s, 4′-OH), 9.42 (2H, s, 3-OH), 10.67 (1H, s, 7-OH), 12.46 (1H, s, 5-OH).

^{13}C NMR spectrum (150 MHz, DMSO-d_6, δ, ppm): 60.76 (C-6″), 69.73 (C-4″), 73.49 (C-2″), 76.14 (C-3″), 77.42 (C-5″), 93.90 (C-8), 98.44 (C-6), 102.59 (C-1″) 103.22 (C-10), 115.93 (C-2′), 116.20 (C-5′), 122.42 (C-6′), 123.77 (C-1′), 136.20 (C-3), 145.49 (C-3′), 146.43 (C-2), 148.98 (C-4′), 156.39 (C-9), 160.84 (C-5), 164.22 (C-7), 176.13 (C-4) [4, 5].

Quercetin-3-*O*-β-D-glucoside (4), yellow powder, mp 249-250°C (MeOH). UV spectrum (λ_{max}, MeOH, nm): 257, 361.

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 0009-3130/08/4403-0370

PMR spectrum (600 MHz, DMSO-d_6, δ, ppm, J/Hz): 3.07-5.29 (sugar protons), 5.47 (1H, d, J = 7.2, H-1″), 6.20 (1H, d, J = 2.4, H-6), 6.40 (1H, d, J = 1.8, H-8), 6.84 (1H, d, J = 8.4, H-5′), 7.58 (1H, d, J = 2.4, H-6′), 7.56 (1H, br.s, H-2′), 9.24 (1H, s, 4′-OH), 9.74 (1H, s, 3′-OH), 10.86 (1H, s, 7-OH), 12.65 (1H, s, 5-OH).

^{13}C NMR spectrum (150 MHz, DMSO-d_6, δ, ppm): 61.97 (C-6″), 70.13 (C-4″), 74.29 (C-2″), 76.69 (C-3″), 77.81 (C-5″), 91.02 (C-8), 98.85 (C-6), 101.01 (C-1″), 104.18 (C-10), 115.40 (C-2′), 116.39 (C-5′), 121.37 (C-1′), 121.82 (C-6′), 133.50 (C-3), 145.02 (C-3′), 148.66 (C-4′), 156.36 (C-9), 156.52 (C-2), 161.45 (C-5), 164.32 (C-7), 177.65 (C-4) [6].

Hyperoside (5), yellow powder, mp 234-236°C (MeOH). UV spectrum (λ_{max}, MeOH, nm): 257, 305sh, 361.

PMR and ^{13}C NMR spectra of **5** agreed fully with those published [3]. Acid hydrolysis of **5** produced quercetin and D-galactose [6].

Astragalin (6), yellow needles (MeOH). UV spectrum (λ_{max}, MeOH, nm): 266, 300sh, 351.

PMR and ^{13}C NMR spectra of **6** agreed fully with those published [3]. Acid hydrolsis of **6** gave kaempferol and D-glucose [7].

Quercetin-7-*O*-β-D-glucoside (7), yellow powder, mp 210-212°C (MeOH). UV spectrum (λ_{max}, MeOH, nm): 256, 373.

PMR spectrum (400 MHz, DMSO-d_6, δ, ppm, J/Hz): 3.20-3.72 (sugar protons), 5.07 (1H, d, J = 7.2, H-1′), 6.43 (1H, d, J = 2, H-6), 6.78 (1H, d, J = 2, H-8), 6.94 (1H, d, J = 8.4, H-5′), 7.58 (1H, d, J = 8.4, 2, H-6′), 7.72 (1H, d, J = 2, H-2′), 9.39 (1H, s, 4′-OH), 9.55 (1H, s, 3-OH), 9.76 (1H, s, 3′-OH), 12.49 (1H, s, 5-OH).

^{13}C NMR spectrum (100 MHz, DMSO-d_6, δ, ppm): 61.11 (C-6″), 70.04 (C-4″), 73.60 (C-2″), 76.78 (C-3″), 77.69 (C-5″), 94.99 (C-8), 99.35 (C-6), 100.47 (C-1″), 105.23 (C-10), 115.87 (C-2′), 116.16 (C-5′), 120.76 (C-6′), 122.44 (C-1′), 136.59 (C-3), 145.50 (C-3′), 148.08 (C-2), 148.31 (C-4′), 156.38 (C-9), 160.70 (C-5), 163.28 (C-7), 176.51 (C-4) [5].

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