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CHEMICAL CONSTITUENTS FROM *Feronia limonia* ROOTS

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Feronia limonia belongs to the Rutaceae family, commonly known as Ma-kuid in Thai. All parts of the plant have been used for the treatment of various ailments. For example, the fruits are considered to be a stomachic, astringent, diuretic, cardiotonic, and tonic to the liver and lungs [1]. The bark is occasionally used for biliousness, whereas the leaves are used for the treatment of indigestion and minor bowel infections of children [1]. In addition, the roots are sometimes used for the treatment of snake bites [1]. In previous phytochemical investigations, coumarins, alkaloids, flavanones, lignans, sterols, and triterpenes have been isolated from different parts of the plant [2]. Many of them show interesting biological activity, including antimicrobial and antitumor activity [1]. As part of our further continuing studies on Rutaceae plants [3–6], we investigated the chemical constituents of this plant. This paper describes the isolation and identification of seven constituents, including six coumarins, 6-methoxy-7-geranyloxycoumarin (**1**) [7], auraptene (**2**) [8], osthol (**3**) [9], osthenol (**4**) [10], xanthyletin (**5**) [11], isopimpinellin (**6**) [9], and a quinolone alkaloid, 1-methyl-4-methoxy-2-quinolone (**7**) [12] from the roots of *F. limonia*. Their structures were determined on the basis of spectroscopic data. To the best of our knowledge, compounds **1**, **3–5**, and **7** were reported for the first time from *F. limonia* roots.

The roots of *F. limonia* were collected from Phitsanulok Province, Thailand in April 2009. A specimen (MFU-NPR0013) is deposited in the Natural Products Research Laboratory, School of Science of Mae Fah Luang University, Thailand.

The roots of *F. limonia* (4 kg) were extracted with acetone over a period of 3 days at room temperature. The solvent was removed under reduced pressure to provide the acetone extract. The crude acetone (43.5 g) extract was chromatographed by quick column chromatography (QCC) over silica gel and eluted with a gradient of hexane–EtOAc (100% hexane to 100% EtOAc) to give 15 fractions (A–O). Fraction E (1.07 g) was resubmitted to QCC eluting with hexane–EtOAc (100% hexane to 100% EtOAc) to afford 12 fractions (E1–E12). Fraction E4 (115.5 mg) was further purified by column chromatography (CC) with 10% EtOAc–hexane to yield three subfractions (E4-1–E4-3). Compound **3** (13.0 mg) was derived from subfraction E4-2 (39.6 mg) by CC developed with 50% CH_2Cl_2–hexane. Fraction E6 (210.0 mg) was subjected to CC eluted with 20% $CHCl_3$–hexane to give five subfractions (E6-1–E6-5). Compound **1** (29.8 mg) was obtained from subfraction E6-4 (66.6 mg) by repeated CC with 30% acetone–hexane. Purification of fraction B (6.83 g) by repeated CC yielded compound **2** (320.4 mg). Fraction F (5.1 g) was resubmitted to QCC and eluted with CH_2Cl_2 to afford six subfractions (F1–F6). Subfraction F2 (204.3 mg) was further purified by repeated CC with CH_2Cl_2 to yield **4** (5.5 mg).The isolation of fraction F1 (241.8 mg) by CC with 20% EtOAc–hexane yielded seven subfractions (F1-1–F1-7). Compound **5** (4.1 mg) was obtained from subfraction F1-5 (23.6 mg) by repeated CC with 50% $CHCl_3$–hexane. Fraction F5 (1.2 g) was separated by QCC with 30% EtOAc–hexane to afford four subfractions (F5-1–F5-4). Compound **7** (4.0 mg) was obtained from subfraction F5-4 (114.7 mg) by repeated CC with 5% acetone–hexane. Fraction D (590.0) was recrystallized with hexane to give a yellow solid (459.3 mg), which was further purified by CC to yield compound **6** (28.7 mg).

6-Methoxy-7-geranyloxycoumarin (1). ^{1}H NMR (400 MHz, $CDCl_3$, δ, ppm): 1.58 (3H, s, H-9′), 1.64 (3H, s, H-8′), 1.76 (3H, s, H-10′), 2.10 (4H, m, H-4′ and 5′), 3.89 (3H, s, 6-OCH_3), 4.68 (2H, d, J = 6.4, H-1′), 5.06 (1H, m, H-6′), 5.47 (1H, m, H-2′), 6.25 (1H, d, J = 9.6, H-3), 6.81 (1H, s, H-8), 6.85 (1H, s, H-5), 7.61 (1H, d, J = 9.6, H-4).

Auraptene (2). ^{1}H NMR (400 MHz, $CDCl_3$, δ, ppm, J/Hz): 1.60 (3H, s, H-9′), 1.66 (3H, s, H-8′), 1.76 (3H, s, H-10′), 2.11 (4H, m, H-4′ and 5′), 4.60 (2H, d, J = 6.4, H-1′), 5.05 (1H, m, H-6′), 5.47 (1H, m, H-2′), 6.25 (1H, d, J = 9.6, H-3), 6.81 (1H, d, J = 2.4, H-8), 6.85 (1H, dd, J = 8.2, 2.4, H-6), 7.36 (1H, d, J = 8.4, H-5), 7.64 (1H, d, J = 9.6, H-4).

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Osthol (3). ^{1}H NMR (400 MHz, $CDCl_3$, δ, ppm, J/Hz): 1.67 (3H, s, H-4′), 1.84 (3H, s, H-5′), 3.54 (2H, d, J = 7.2, H-1′), 3.92 (3H, s, 7-OCH_3), 5.22 (1H, m, H-2′), 6.24 (1H, d, J = 9.6, H-3), 6.66 (1H, d, J = 8.4, H-6), 7.30 (1H, d, J = 8.4, H-5), 7.62 (1H, d, J = 9.6, H-4).

Osthenol (4). ^{1}H NMR (400 MHz, $CDCl_3$, δ, ppm, J/Hz): 1.76 (3H, s, H-4′), 1.86 (3H, s, H-5′), 3.62 (2H, d, J = 7.2, H-1′), 5.28 (1H, m, H-2′), 6.16 (1H, br.s, 7-OH), 6.24 (1H, d, J = 9.6, H-3), 6.75 (1H, d, J = 8.4, H-6), 7.23 (1H, d, J = 8.4, H-5), 7.63 (1H, d, J = 9.6, H-4).

Xanthyletin (5). Light viscous oil. ^{1}H NMR (400 MHz, $CDCl_3$, δ, ppm, J/Hz): 1.47 (6H, s, H-4′ and 5′), 5.65 (1H, d, J = 10, H-2′), 6.22 (1H, d, J = 9.6, H-3), 6.33 (1H, d, J = 10, H-1′), 6.72 (1H, s, H-5), 7.04 (1H, s, H-8), 7.58 (1H, d, J = 9.6, H-4).

Isopimpinellin (6). ^{1}H NMR (400 MHz, $CDCl_3$, δ, ppm, J/Hz): 4.16 (3H, s, 5-OCH_3), 4.18 (3H, s, 8-OCH_3), 6.28 (1H, d, J = 9.9, H-3), 7.01 (1H, d, J = 2.4, H-2′), 7.63 (1H, d, J = 2.4, H-3′), 8.12 (1H, d, J = 9.9, H-4).

1-Methyl-4-methoxy-2-quinolone (7). ^{1}H NMR (400 MHz, $CDCl_3$, δ, ppm, J/Hz): 3.95 (3H, s, 4-OCH_3), 3.68 (3H, s, N-CH_3), 6.07 (1H, s, H-3), 7.24 (1H, dd, J = 8.0, 8.2, H-6), 7.34 (1H, d, J = 8.4, H-8), 7.59 (1H, dd, J = 8.2, 8.4, H-7), 7.97 (1H, d, J = 8.0, H-5).

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