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Flavonoids in aerial parts of *Persicaria mitis* (Schrank) Holub

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ABSTRACT

The study presents the isolation of seventeen polyphenols from aerial parts of *Persicaria mitis* comprising one simple chromone derivative (**1**) and sixteen flavonols (**2**–**17**). Compounds (**1**, **3**–**4**, **8**, **10**, **12** and **15**–**17**) were detected and isolated for the first time from investigated plant material. The chemotaxonomic value of all isolated compounds within *Persicariai* genus is discussed.

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1. Subject and source

Persicaria mitis (Schrank) Holub (*Persicaria dubia* Stein, *Polygonum mite* Schrank), commonly known as tasteless water pepper, is an erect to suberect, 35–60 cm tall, branched, annual herbaceous weed occurring in the central temperate region of Europe (Webb, 1964; Jalas and Suominen, 1972). Recent phylogenetic studies on *Polygonum* L. *sensu lato* confirmed that *Persicaria mitis* is closely related to *Persicaria minor* (Huds.) Opiz and *Persicaria hydropiper* (L.) Delarbre (Galasso et al., 2009). The aerial parts of *P. mitis* were collected at the banks of Vistula river in Warsaw (N 52°13′36″, E 21°03′06″, altitude: 82 m). The plant material was identified by Dr. Sebastian Granica and confirmed by Prof. Christian Zidorn (Institute of Pharmacy, Department of Pharmacognosy, University of Innsbruck, Austria) and Prof. Dr. Konrad Pagitz (Institute of Botany, University of Innsbruck, Austria). A voucher specimen (No. 20140910_C) was deposited in the herbarium of Department of Pharmacognosy and Molecular Basis of Phytotherapy, Medical University of Warsaw, Poland.

2. Previous work

Previous investigations on the chemical composition of taxa belonging to Polygonaceae family showed that flavonoids are valuable chemotaxonomical markers within this family (Kawasaki et al., 1986). In the literature there are three chromatographic studies describing the phytochemical composition of *P. mitis* (Smolarz, 1999, 2002a,b). The TLC analysis of phenolic acids revealed that *P. mitis* contained chlorogenic, protocatechuic, caffeic, gentysic, *p*-hydroxyphenyl acetic, *p*-hydroxybenzoic, *p*-coumaric, melillotic, syringic, vanillic and salycilic acids (Smolarz, 1999). The HPLC investigation of flavonoid aglycones showed that aerial parts of *P. mitte* contain taxifolin, luteolin, quercetin-3-*O*-methyl ether, quercetin, kaempferol and rhamnetin (Smolarz, 2002a). Smolarz (2002b) also reported thirteen flavonoid glycosides including quercetin 3-*O*-

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rutinoside (rutin), quercetin 3-*O*-galactoside (hyperoside, hyperin), quercetin 3-*O*-glucoside (isoquercitrin), quercetin 3-*O*-rhamnoside (quercitrin), quercetin 3-*O*-arabinopyranoside (guaijaverin), quercetin 3-*O*-arabinofuranoside (avicularin), quercetin-3-*O*-glucuronide (miquelianin), quercetin 3-*O*-(6″-galloyl)-glucoside, quercetin 8-*O*-glucoside, quercetin 4′-*O*-glucoside (spireoside), kaempferol 3-*O*-glucoside (astragalin) and isorhamnetin 3-*O*-glucoside. It must be noted that none of the mentioned compounds have been isolated and that their identity was confirmed basing on R_f values or retention times compared to available standards. Thus all reported studies should be considered as preliminary.

3. Present study

Shade dried aerial parts of *P. mitis* (800 g) were exhaustively extracted with a mixture of acetone:methanol:water (3:1:1, *v*/*v*/*v*) by sonication (10 × 3 L, 30 min each time). Organic solvents were evaporated under reduced pressure and the aqueous residue was partitioned between diethyl ether (7 × 0.75 L), ethyl acetate (5 × 0.75 L) and finally *n*-butanol (5 × 0.75 L). Combined fractions were evaporated to dryness under vacuum affording 1.11, 4.86 and 13.53 g of each fraction, respectively.

The diethyl ether residue (1.11 g) was subjected to a flash chromatography system (PuriFlash 430evo, Interchim, France, silica gel column – 40 mm × 50 mm, Interchim, France, λ = 254, 350 nm, flow 5 mL/min, mobile phase: chloroform (A) and methanol (B); elution program: 0% B – 50% B (0–480 min) then 50% B – 75% B (480–510 min)) fractions were collected continuously 20 mL each to obtain 128 fractions which were then pooled into 12 main fractions E_1-E_{12} basing on TLC profile (silicagel, mobile phase: ethyl actete:formic acid:water, 18:1:1, *v*/*v*/*v*, derivatized with 1% Naturstoffreagenz A, Carl Roth, Germany). Fraction E_2 (33 mg) was subjected to preparative HPLC system (Shimadzu LC10vp, Japan, Zorbax C_{18} – 5 μm, 150 mm × 21.2 mm, Agilent, CA, USA, λ = 280 nm, flow 7 mL/min, mobile phase: 0.1% HCOOH in water (A) and 0.1% HCOOH in acetonitrile (B); elution program: 10% B – 30% B (0–35 min)) fractions were collected based on UV–Vis chromatograms) to give pure compound **1** (t_r = 33.3–34.5 min, 2.1 mg). Fraction E_7 (200 mg) was subjected to column chromatography on Sephadex LH-20 (150 cm × 2.5 cm) and eluted with methanol to give 108 fractions combined to 8 main fractions ES_1-ES_8 basing on TLC profile (the same as above). Fraction ES_4 (68 mg) and fraction ES_7 (14 mg) were separated using preparative HPLC system (instrumentation and conditions as above) to give pure compounds **2** (t_r = 28.7–30.8 min, 12.5 mg) and **3** (t_r = 29.3–30.7 min, 5.5 mg), respectively.

The ethyl acetate residue (4.86 g) was subjected to a flash chromatography system (the same instrumentation, silica gel column – 45 mm × 50 mm, flow 5 mL/min, mobile phase: chloroform (A) and methanol (B); elution program: 0% B – 50% B (0–360 min) then 50% B – 70% B (360–420 min) and finally 70%B – 100% B (420–480 min)) fractions were collected continuously 15 mL each to obtain 160 fractions which were pooled into 13 main fractions O_1–O_{13} basing on TLC profile (the same as above). Fraction O_2 contained pure compound **4** (43 mg). Fractions O_5 (60 mg) and O_6 (83 mg) were separated using preparative HPLC system (instrumentation as above, different gradient elution: 20% B – 45% B, 0–45 min, then 45%–60%, 45–60 min) to give pure compounds **5** (t_r = 37.0–37.7 min, 1.1 mg), **6** (t_r = 37.9–38.6 min, 8.8 mg), **7** (t_r = 45.5–46.2 min, 1.1 mg) and **8** (t_r = 54.8–55.5 min, 0.6 mg) from fraction O_5 and compound **9** (t_r = 28.0–29.6 min, 3.6 mg) from fraction O_6. Fraction O_7 (700 mg) was subjected to column chromatography on Sephadex LH-20 (150 cm × 2.5 cm) eluted with methanol to give 110 fractions combined to 10 main fractions OS_1–OS_{10} based on TLC profile (the same as above). Fraction OS_6 (150 mg) was subjected to preparative HPLC system (instrumentation as above, different gradient elution: 10% B – 35% B, 0–35 min) to yield pure compounds **10** (t_r = 29.3–30.0 min, 1.9 mg), **11** (t_r = 34.7–35.4 min, 1.5 mg), and **12** (t_r = 35.6–36.2 min, 2.2 mg). Fraction OS_8 (38 mg) was subjected to preparative HPLC system (instrumentation as above, different gradient elution: 12% B – 40% B, 0–45 min) to give compounds **13** (t_r = 24.2–25.0 min, 5.8 mg) and **14** (t_r = 31.5–36.5 min, 4.9 mg). Fraction O_9 (320 mg) was subjected to column chromatography on Sephadex LH-20 (150 cm × 2.5 cm) eluted with methanol to give 144 fractions combined to 8 main fractions OSS_1–OSS_8 based on TLC profile (the same as above). Fraction OSS_4 (25 mg) was subjected to preparative HPLC system (instrumentation as above, different gradient elution: 12% B – 35% B, 0–45 min) to give compound **15** (t_r = 22.0–22.4 min, 4.5 mg). Fraction OSS_7 (60 mg) was subjected to preparative HPLC system (instrumentation as above, different gradient elution: 10% B – 35% B, 0–35 min) to give compound **16** (t_r = 23.6–24.3 min, 6.9 mg). Fraction O_{11} (1.01 g) was subjected to preparative HPLC system (instrumentation as above, 15 runs 67 mg each time, different gradient elution: 10% B – 30% B, 0–35 min) to obtain pure compound **17** (t_r = 29.0–30.0 min, 99 mg).

Structures of the isolated compounds have been determined on the basis of the UV–Vis, MS and ^{1}H spectra. If needed, additional experiments including ^{13}C, HSQC, HMBC and chromatographic experiments have been performed. Isolated flavonoids were identified as 7,5-dihydroxychromone (**1**) (Simon et al., 1994), quercetin 3-*O*-α-L-rhamnopyranoside (quercitrin) (**2**) (Peng et al., 2003), quercetin 3-O-α-(2″-*O*-galloyl)-L-rhamnopyranoside (**3**) (Peng et al., 2003), rhamnazin (**4**) (Barbera et al., 1986), kaempferol (**5**) (Yang et al., 2012), isorhamnetin (**6**) (Su et al., 2008), rhamnetin (**7**) (Yamauchi et al., 2014), kaempferide (**8**) (Ji et al., 2014), quercetin (**9**) (Yang et al., 2012), gossypetin 8-methyl ether 3-*O*-β-D-glucopyranoside (**10**) (Bennini et al., 1992), kaempferol 3-*O*-β-D-glucopyranoside (**11**) (astragalin) (Peng et al., 2003), isorhamnetin 3-*O*-β-D-glucopyranoside (**12**) (Sang et al., 2002), quercetin 3-*O*-α-L-arabinopyranoside (**13**) (Kwon and Bae, 2013), rhamnazin 3 *O*-sulphate (**14**) (Haraguchi et al., 1996), quercetin 3-*O*-β-D-glucopyranoside (**15**) (isoquercitrin) (Wu et al., 2008), quercetin 3-*O*-β-(2″-*O*-galloyl)-D-glucopyranoside (**16**) (Kawakami et al., 2011) and quercetin 3-*O*-(2″-O-β-D-xylopyranosyl)-α-L-rhamnopyranoside (**17**) (Nielsen et al., 2005).

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