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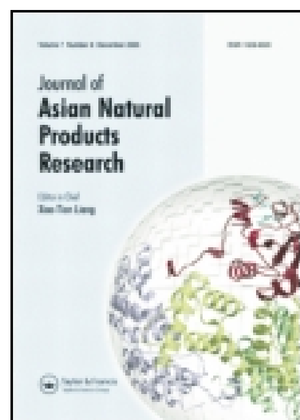

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Smilasides M and N, two new phenylpropanoid glycosides from Smilax riparia

Ting-Ting Sun [a], Dian-Wen Zhang [a], Yan Han [a], Fang-Yan Dong [a] & Wei Wang [a]

[a] Institute of Phytochemistry, Jilin Academy of Chinese Medicine Sciences, Changchun, 130012, China

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Smilasides M and N, two new phenylpropanoid glycosides from *Smilax riparia*

Ting-Ting Sun, Dian-Wen Zhang, Yan Han, Fang-Yan Dong and Wei Wang*

Institute of Phytochemistry, Jilin Academy of Chinese Medicine Sciences, Changchun 130012, China

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Two new phenylpropanoid glycosides, smilasides M and N, together with the known compound 2′,6′-diacetyl-3,6-diferuloylsucrose, were isolated and characterized from the roots and rhizomes of *Smilax riparia* A. DC. The structures of the new compounds were elucidated as 2′,6′-diacetyl-3-*Z*-feruloyl-6-feruloylsucrose (**1**) and 2′,6′-diacetyl-3-feruloyl-6-*Z*-feruloylsucrose (**2**) on the basis of extensive analysis of HR-ESI-MS, UV, IR, and 1D and 2D NMR spectroscopic data.

Keywords: *Smilax riparia*; Liliaceae; phenylpropanoid glycosides; smilaside M; smilaside N

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1. Introduction

The genus *Smilax* belongs to the family Liliaceae and comprises about 300 species, which distribute widely in the tropical and temperate regions of East Asia and South and North America [1]. The extracts of many plants from this genus have been reported to exhibit anti-hyperuricemic and nephroprotective [2], anticonvulsant and neurotoxic [3], anti-proliferative and pro-apoptotic [4], anti-inflammatory and anti-nociceptive [5], hypoglycemic [6], antioxidant [7,8], and immunomodulatory activities [9]. Phytochemical investigations on the genus *Smilax* have revealed the occurrence of steroidal saponins [10–12], phenylpropanoid glycosides [13–15], flavonoids [16–18], and phenolic compounds [19–21]. *Smilax riparia* A. DC. is a climbing herbaceous vine distributed in the south and midland of China. Its roots and rhizomes have been used as a folklore medicine for the treatment of bronchitis, lumbago of renal asthenia, traumatic injury, asthenia edema, and bronchial dilation agents [22]. Although several constituents, including four steroidal saponins, four flavonoids, a phenylpropanoid glycoside, and an aromatic compound, were isolated and identified from the roots and rhizomes of *S. riparia* previously [23,24], a systematic phytochemical investigation has not been pursued. As a part of our ongoing search for bioactive secondary metabolites from medicinal plants growing in the Changbai Mountain, Jilin Province of China, we investigated the constituents of the roots and rhizomes of *S. riparia*. Fractionation of the ethanol extract led to the isolation of two new phenylpropanoid glycosides, namely smilasides M and N (**1** and **2**), along with the known compound 2′,6′-diacetyl-3,6-diferuloylsucrose (**3**) (Figure 1). We report herein on the isolation and structural elucidation of **1** and **2** using spectroscopic data

*Corresponding author. Email: w.w.wangwei@263.net

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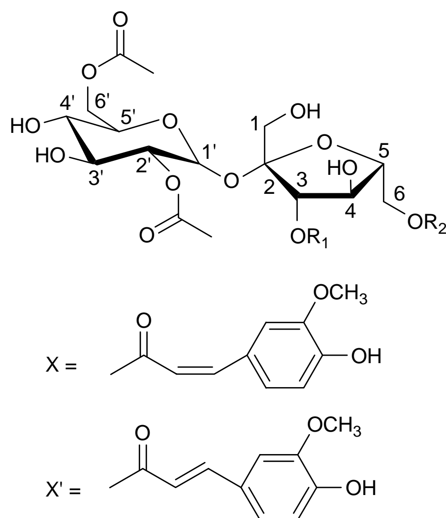

Figure 1. Chemical structures of compounds **1**–**3**.

analysis, including 1D and 2D NMR techniques.

2. Results and discussion

Compound **1** was obtained as a yellowish gum with a molecular formula of $C_{36}H_{42}O_{19}$ on the basis of the quasi-molecular ion at *m/z* 779.2394 $[M + H]^+$ in the HR-ESI-MS. The IR and UV spectra displayed absorption bands for the hydroxyl and α,β-unsatuated aromatic ester groups. The ^{1}H NMR spectrum (Table 1) showed two pairs of olefinic protons at δ 5.92 (d, $J = 12.8$ Hz, H-8$''$) and 6.94 (d, $J = 12.8$ Hz, H-7$''$), and 6.38 (d, $J = 15.8$ Hz, H-8$'''$) and 7.65 (d, $J = 15.8$ Hz, H-7$'''$), and two aromatic moieties with ABX coupling patterns at δ 6.78 (d, $J = 8.2$ Hz, H-5$''$), 7.19 (dd, $J = 8.2$, 1.6 Hz, H-6$''$), and 7.80 (br s, H-2$''$) and at δ 6.82 (d, $J = 8.2$ Hz, H-5$'''$), 7.10 (br d, $J = 8.2$ Hz, H-6$'''$), and 7.21 (br s, H-2$'''$). The coupling constants $J = 12.8$ Hz of the proton signals at δ 5.92/6.94 and $J = 15.8$ Hz of the proton signals at δ 6.38/7.65 suggested the presence of a pair of *cis*-olefinic protons and a pair of *trans*-olefinic protons. The above ^{1}H NMR data, along with two methoxy groups at the aromatic moieties as determined from the HMBC spectrum, indicated the presence of one *Z*-feruloyl and one feruloyl units in **1**. The ^{13}C NMR chemical shifts attributable to 18 sp^2 carbons (Table 1) were in good agreement with those of one *Z*-feruloyl and one feruloyl moieties. In its ^{1}H NMR spectrum, there were signals for two acetyl groups at δ 2.04 (s) and 2.05 (s). The ^{13}C NMR spectrum also displayed resonances due to two acetyl groups. In addition, signals for eight oxygenated methines at δ 5.54 (d, $J = 8.6$ Hz, H-3), 4.45 (t, $J = 8.6$ Hz, H-4), 4.10 (m, H-5), 5.65 (d, $J = 3.8$ Hz, H-1$'$), 4.57 (d, $J = 9.8$, 3.8 Hz, H-2$'$), 3.79 (t, $J = 9.8$ Hz, H-3$'$), 3.32 (t, $J = 9.8$ Hz, H-4$'$), and 4.18 (m, H-5$'$), three oxygenated methylenes at δ 3.44 (d, $J = 11.7$ Hz, H-1a) and 3.63 (d, $J = 11.7$ Hz, H-1b), 4.47 (m, H-6a) and 4.50 (m, H-6b), and 4.16 (m, H-6$'$a) and 4.43 (m, H-6$'$b) were observed in the ^{1}H NMR spectrum. A characteristic anomeric signal at δ 5.65 with a smaller coupling constant $J = 3.8$ Hz, together with the ^{13}C NMR spectrum showing 12 oxygenated carbon resonances containing two anomeric carbons at δ 90.1 and 105.2, suggested that **1** possessed a disaccharide moiety. Detailed analysis of the ^{1}H and ^{13}C NMR spectroscopic data of **1** with the aid of 2D NMR analyses indicated that the two sugars comprise β-D-fructose and α-D-glucose moieties connected through a 2⟶1 linkages. Thus, **1** should be a phenylpropanoid sucroside. On further inspection of the HMBC spectrum, not only the *Z*-feruloyl and feruloyl moieties at C-3 and C-6 in fructose, but also each acetyl group at C-2$'$ and C-6$'$ of glucose could be assigned unambiguously, due to the correlations observed between H-3, H-6, H-2$'$, and H-6$'$,

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Table 1. ^{1}H NMR (500 MHz) and ^{13}C NMR (125 MHz) spectroscopic data for compounds **1** and **2** in CD_3OD.

	1		**2**	
No.	δ_H	δ_C	δ_H	δ_C
β-D-Fru				
1	3.44 (d, *J* = 11.7 Hz)	64.9 (t)	3.42 (d, *J* = 12.0 Hz)	64.8 (t)
	3.63 (d, *J* = 11.7 Hz)		3.60 (d, *J* = 12.0 Hz)	
2		105.2 (s)		105.4 (s)
3	5.54 (d, *J* = 8.6 Hz)	78.1 (d)	5.54 (d, *J* = 8.2 Hz)	78.4 (d)
4	4.45 (t, *J* = 8.6 Hz)	73.7 (d)	4.43 (t, *J* = 8.5 Hz)	74.4 (d)
5	4.10 (m)	81.1 (d)	4.11 (m)	81.2 (d)
6	4.47 (m)	65.0 (t)	4.44 (m)	65.3 (t)
	4.50 (m)		4.48 (m)	
α-D-Glc				
1′	5.65 (d, *J* = 3.8 Hz)	90.1 (d)	5.57 (d, *J* = 3.8 Hz)	90.4 (d)
2′	4.57 (d, *J* = 9.8, 3.8 Hz)	74.4 (d)	4.59 (d, *J* = 10.0, 3.8 Hz)	74.4 (d)
3′	3.79 (t, *J* = 9.8 Hz)	72.0 (d)	3.81 (t, *J* = 10.0 Hz)	72.1 (d)
4′	3.32 (t, *J* = 9.8 Hz)	71.8 (d)	3.36 (t, *J* = 10.0 Hz)	71.9 (d)
5′	4.18 (m)	71.9 (d)	4.18 (m)	72.0 (d)
6′	4.16 (m)	65.1 (t)	4.15 (m)	65.6 (t)
	4.43 (m)		4.40 (m)	
Feruloyl-3				
1″		127.9 (s)		126.9 (s)
2″	7.80 (br s)	115.2 (d)	7.26 (br s)	112.0 (d)
3″		148.4 (s)		148.0 (s)
4″		149.9 (s)		149.5 (s)
5″	6.78 (d, *J* = 8.2 Hz)	115.8 (d)	6.81 (d, *J* = 8.2 Hz)	115.6 (d)
6″	7.19 (dd, *J* = 8.2, 1.6 Hz)	127.1 (d)	7.12 (dd, *J* = 8.2, 1.6 Hz)	124.5 (d)
7″	6.94 (d, *J* = 12.8 Hz)	146.9 (d)	7.71 (d, *J* = 15.8 Hz)	148.0 (d)
8″	5.92 (d, *J* = 12.8 Hz)	115.5 (d)	6.45 (d, *J* = 15.8 Hz)	114.6 (d)
9″		167.4 (s)		168.9 (s)
OCH_3	3.89 (s)	56.5 (q)	3.90 (s)	56.5 (q)
Feruloyl-6				
1‴		127.8 (s)		127.5 (s)
2‴	7.21 (br s)	111.8 (d)	7.83 (br s)	115.2 (d)
3‴		149.4 (s)		148.0 (s)
4‴		150.8 (s)		148.4 (s)
5‴	6.82 (d, *J* = 8.2 Hz)	116.5 (d)	6.77 (d, *J* = 8.2 Hz)	115.7 (d)
6‴	7.10 (br d, *J* = 8.2 Hz)	124.2 (d)	7.14 (dd, *J* = 8.2, 1.9 Hz)	124.2 (d)
7‴	7.65 (d, *J* = 15.8 Hz)	147.1 (d)	6.88 (d, *J* = 12.8 Hz)	146.0 (d)
8‴	6.38 (d, *J* = 15.8 Hz)	115.3 (d)	5.84 (d, *J* = 12.8 Hz)	115.2 (d)
9‴		168.8 (s)		167.9 (s)
OCH_3	3.90 (s)	56.6 (q)	3.88 (s)	56.5 (q)
OAc-2′		172.3 (s)		172.4 (s)
	2.05 (s)	20.9 (q)	2.09 (s)	21.0 (q)
OAc-6′		172.9 (s)		172.8 (s)
	2.04 (s)	20.8 (q)	2.07 (s)	21.0 (q)

and the corresponding carbonyl carbons of *Z*-ferulate, ferulate, and acetates (Figure 2). From the above evidence, the structure of **1** was established as 2′,6′-diacetyl-3-*Z*-feruloyl-6-feruloylsucrose, and named smilaside M.

Compound **2** showed a quasi-molecular ion at *m/z* 779.2402 by HR-ESI-MS, which is consistent with the molecular formula $C_{36}H_{42}O_{19}$. The ^{1}H and ^{13}C NMR spectra suggested that **2** possessed a structure similar to **1**, containing glucose

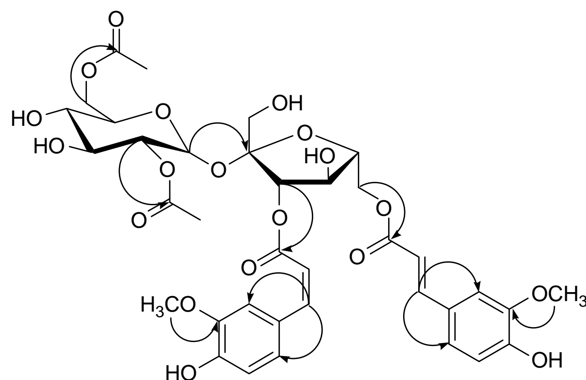

Figure 2. Key HMBC correlations of compound **1**.

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and fructose units, one *Z*-feruloyl and one feruloyl moieties, and two acetyl groups. Furthermore, by comparing the HMBC spectrum of **2** with that of **1**, the position of feruloyl moiety was determined at C-3 of fructose in **2** instead of C-6 in **1**, due to the long-range correlation between the ester carbonyl carbon at δ 168.9 of feruloyl moiety and the H-3 at δ 5.54 of fructose for **2**, while that of position of *Z*-feruloyl moiety was determined at C-6 of fructose in **2** instead of C-3 in **1**, due to the long-range correlations between the ester carbonyl carbon at δ 167.9 of *Z*-feruloyl moiety and H-6 at δ 4.44 and 4.48 of fructose for **2**. Accordingly, the structure of **2** was determined as 2′,6′-diacetyl-3-feruloyl-6-*Z*-feruloylsucrose, and named smilaside N.

The known compound 2′,6′-diacetyl-3,6-diferuloylsucrose (**3**) was also isolated and identified by comparison of their spectral data with those reported in the literature [25].

3. Experimental

3.1 General experimental procedures

UV spectra were measured with a Shimadzu UV-2100 spectrometer in methanol with absorption given in nm and IR spectra were measured as KBr pellets with a Perkin-Elmer FT-IR spectrometer with absorption given in cm^{-1}. Optical rotations were measured by using a HORIBA SEPA-300 digital polarimeter. NMR spectra were measured on a Bruker AV-500 FT-NMR in CD_3OD, using visual CD_3OD resonances (^{1}H δ 3.31, ^{13}C δ 49.0) for internal reference. All chemical shifts (δ) are given in ppm. HR-ESI-MS and ESI-MS were obtained with a Bruker microTOFQ mass spectrometer (Bruker Daltonics, Bremen, Germany). Column chromatography was obtained with macroporous resin D101 (Haiguang Chemical Ltd, Tianjin, China), silica gel (200–300 mesh, Qingdao Marine Chemical Factory, Qingdao, China), RP-18 reversed-phase silica gel (S-50 μm, YMC, Kyoto, Japan), and Sephardex LH-20 (GE Healthcare, Uppsala, Sweden). TLC analysis was carried out on pre-coated TLC plates with silica gel 60 F_{254} and silica gel RP-18 60 F_{254} (Merck, Darmstadt, Germany, 0.25 mm). Detection was achieved by spraying with 10% H_2SO_4 in MeOH followed by heating. Preparative HPLC was carried out on a Shimadzu LC-6AD pump connected with a Shimadzu SPD-20A UV–vis detector (at 220 nm) and a SHODEX RI-102 detector, using Shim Pak ODS column (250 mm × 21.2 mm, i.d., 5 μm, Shimadzu, Kyoto, Japan) and Kromasil silica column (250 mm × 10 mm, i.d., 5 μm,

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Fluka BioChemika, Buchs, Switzerland), respectively. All solvents used for chromatographic separations were distilled before use.

3.2 Plant material

The roots and rhizomes of *S. riparia* were collected from Changbai Mountain, Jilin Province of China, in October 2007, and were authenticated by Prof. Zhongkai Yan, Jilin Academy of Chinese Medicine Sciences, China. A voucher specimen (SRNWC-2007-10) is deposited at the Institute of Phytochemistry, Jilin Academy of Chinese Medicine Sciences, China.

3.3 Extraction and isolation

The air-dried and ground roots and rhizomes (23.5 kg) of *S. riparia* were extracted twice with 95% ethanol under reflux for 2 h and the solvent was evaporated under reduced pressure to give a brown residue (1.85 kg). The residue (1.8 kg) was diluted with distilled water and subjected to D101 macroporous resin column chromatography and eluted with H_2O and 30% and 70% ethanol, successively. The fraction eluted with 70% ethanol (200 g) was suspended in water and successively partitioned with chloroform, ethyl acetate, and *n*-butanol.

The EtOAc-soluble fraction (15 g) was chromatographed over a silica gel column (8 × 60 cm, 1.0 kg) eluting with $CHCl_3$–MeOH (49:1, 19:1, 9:1, and 1:1) and separated into nine fractions (Fractions I–IX) on the basis of TLC analyses. Fraction III (2 g) was subjected to RP-18 reversed-phase silica gel column (3 × 25, 100 g) chromatography with a MeOH–H_2O (50:50) solvent system to afford six subfractions (Subfractions III-1–III-6). Subfraction III-3 (166 mg) was isolated by preparative HPLC (RP-18 column: 250 mm × 21.2 mm, i.d., 5 μm; flow rate: 2 ml/min) using CH_3CN–H_2O (28:72) as the mobile phase to yield compounds **1** (15.2 mg, $t_R = 197$ min), **2** (10.2 mg, $t_R = 252$ min), and **3** (40.4 mg, $t_R = 230$ min).

3.3.1 Smilaside M (1)

A yellowish gum; $[\alpha]_D^{25}$ + 18.4 ($c = 0.56$, CH_3OH); UV λ_{max} (CH_3OH): 322, 299 (sh), 233, 219; IR υ_{max} (KBr): 3364, 2943, 2837, 1704, 1603, 1448, 1278, 1029 cm^{-1}; ^1H NMR (500 MHz, CD_3OD) and ^{13}C NMR (125 MHz, CD_3OD) spectroscopic data see Table 1; HR-ESI-MS: *m/z* 779.2394 $[M + H]^+$ (calcd for $C_{36}H_{43}O_{19}$, 779.2399).

3.3.2 Smilaside N (2)

A yellowish gum; $[\alpha]_D^{25}$ + 10.5 ($c = 0.78$, CH_3OH); UV λ_{max} (CH_3OH): 320, 299 (sh), 231, 217; IR υ_{max} (KBr): 3365, 2945, 2837, 1700, 1598, 1450, 1278, 1030 cm^{-1}; ^1H NMR (500 MHz, CD_3OD) and ^{13}C NMR (125 MHz, CD_3OD) spectroscopic data see Table 1; HR-ESI-MS: *m/z* 779.2402 $[M + H]^+$ (calcd for $C_{36}H_{43}O_{19}$, 779.2399).

3.3.3 Alkaline hydrolysis of 1 and 2

Compounds **1** (5.2 mg) and **2** (4.8 mg) were hydrolyzed with 5% KOH/MeOH (3 ml) at room temperature for 20 min. The reaction mixture was neutralized with 2 M HCl and filtered. The filtrate was concentrated under reduced pressure to give a residue that was chromatographed over a Sephardex LH-20 column (MeOH) and preparative HPLC (silica gel column: 250 mm × 10 mm, i.d., 5 μm; flow rate: 2 ml/min) using $CHCl_3$–MeOH–H_2O (90:70:15) as the mobile phase to yield sucrose (1.5 mg from **1** and 1.4 mg from **2**, $t_R = 17.6$ min). The sucrose was detected by co-injection of an authentic sample on preparative HPLC in the same manner described above, giving a single peak.

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