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Biflavonoids from *Stellera chamaejasme*

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Abstract

Five biflavonoids (wilstrol A, wikstrol B, neochamaejasmin A, neochamaejasmin B and chamajssmine) were isolated from the roots of *Stellera chamaejasme* L. These compounds play a very important role in the chemotaxonomy of Thymelaeaceae.

Keywords: Biflavanones, biflavonoids, chemical constituents, chemotaxonomy, *Stellera chamaejasme* L. Thymelaeaceae, wikstrol A, wikstrol B.

Introduction

Stellera chamaejasme L (Thymelaeaceae), with the Chinese name "langdu", is widespread in the north of China. It was traditionally used as a herbal remedy for scabies and tinea. Recently it has been found to show obvious antitumor activity (Feng et al., 1995; Yoshida et al., 1996).

Thymelaeaceae is divided into about 50 genera with more than 500 species. Much chemical research has been carried out on the Thymelaeaceae plants. However, little work has been done to study the chemotaxonomy of this family. The determination of taxonomic relationships until now is mainly based on morphological characters rather than on chemical criteria.

In our study, five biflavonoids were isolated from the roots of *Stellera chamaejasme* L. They were identified as wikstrol A, wikstrol B, neochamaejasmin A, neochamaejasmin B and chamajssmine. The structures were determined by comparison of the NMR data of these compounds with the literature (Niwa et al., 1984, 1986; Baba et al., 1994).

Materials and methods

General experimental procedures

^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker 300 spectrometer at 300 and 75 MHz, respectively. Melting points were determined on a Yanaco micro-melting points apparatus (uncorrected).

Plant material

The roots of *Stellera chamaejasme* L were collected in the west of Liaoning Province, northeast of China, in September 1996 and identified by professor Chunquan Xu of Shenyang Pharmaceutical University. A voucher specimen is kept in the Department of Traditional Chinese medicines of Shenyang Pharmaceutical University with the No. 19960088.

Extraction and isolation

The dry roots of *Stellera chamaejasme* (1 kg) were extracted with EtOH (95%). EtOH was evaporated and the residue was extracted with petroleum ether, ether and acetone successively. The acetone extract (20 g) was subjected to CC on silica gel and elueted with a $CHCl_3$: CH_3OH gradient from 100 : 1 to 5 : 1. The fractions elueted with $CHCl_3$: CH_3OH (10 : 1) were further purified by HPLC (RP-18 column) with H_2O : CH_3CN (7.5 : 2.5) as eluents to yield wikstrol A (10 mg), wikstrol B (10 mg), neochamaejasmin A (15 mg), neochamaejasmin B (10 mg), and chamaejasmine (10 mg).

Wikstrol A

Yellow powder, mp 243~245 °C (CH_3OH). ^{1}H NMR (DMSO-d_6): δ 2.37 (1 H, dd, J = 15.9, 7.5 Hz, H-4), 2.54 (1 H, dd, J = 15.9, 5.8 Hz, H-4), 3.81 (1 H, m, H-3), 4.38 (1 H, d, J = 6.6 Hz), 4.90 (1 H, br s, 3-OH), 5.95 (1 H, s, H-6), 6.02 (1 H, s, H-6″), 6.34 (1 H, s, H-8″), 6.63 (2 H, d, J = 8.1 Hz, H-3′, 5′), 6.67 (2 H, d, J = 8.4 Hz, H = 11″, 13″), 7.05 (2 H, d, J = 8.1 Hz, H-2′, 6′) 7.29 (2 H, d, J = 8.3 Hz, H-10″, 14″), 13.26 (5″-OH). ^{13}C NMR (DMSO-d_6): δ 27.5 (C-4), 66.3 (C-3), 80.7 (C-2), 93.8 (C-8″), 95.2 (C-6), 98.9 (C-8), 99.0 (C-4a),

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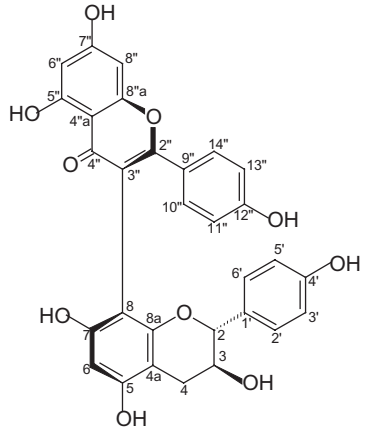
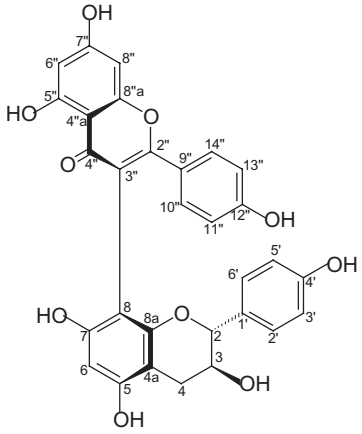
99.4 (C-6″), 102.0 (C-4″a), 112.8 (C-3″), 114.6 (C-3′, 5′), 114.8 (C-11″, 13″), 123.9 (C-9″), 128.0 (C-2′, 6′), 129.9 (C-10″, 14″), 130.1 (C-1′), 153.5 (C-7), 154.2 (C-8a), 155.8 (C-5), 156.7 (C-4′, 8″a), 157.6 (C-12″), 159.4 (C-5″), 161.4 (C-2″), 161.6 (C-7″), 181.0 (C-4″).

Wikstrol B

Yellow powder, mp 232~234 °C (CH_3OH). ^{1}H NMR (DMSO-d_6): δ 2.35 (1 H, dd, *J* = 15.9, 8.4 Hz, H-4), 2.62 (1 H, dd, *J* = 15.9, 4.8 Hz, H-4), 3.45 (1 H, m, H-3), 4.44 (1 H, d, *J* = 7.5 Hz), 4.90 (1 H, br s, 3-OH), 5.77 (1 H, s, H-6″), 6.00 (1 H, s, H-6), 6.17 (1 H, s, H-8″), 6.52 (2 H, d, *J* = 8.4 Hz, H-3′, 5′), 6.64 (2 H, d, *J* = 8.4 Hz, H = 2′, 6′), 6.74 (2 H, d, *J* = 8.7 Hz, H-11″, 13″) 7.38 (2 H, d, *J* = 8.7 Hz, H-10″, 14″), 13.17 (5″-OH). ^{13}C NMR (DMSO-d_6): δ 28.1 (C-4), 66.8 (C-3), 80.9 (C-2), 93.9 (C-8″), 95.1 (C-6), 98.7 (C-8), 99.0 (C-6″), 99.5 (C-4a), 102.4 (C-4″a), 112.5 (C-3″), 114.6 (C-3′, 5′), 114.9 (C-11″, 13″), 123.9 (C-9″), 128.0 (C-2′, 6′), 129.9 (C-10″, 14″), 130.0 (C-1′), 152.7 (C-7), 155.0 (C-8a), 155.7 (C-5),



Wikstrol A



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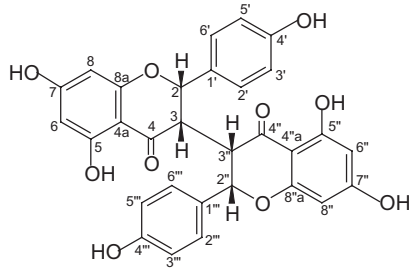
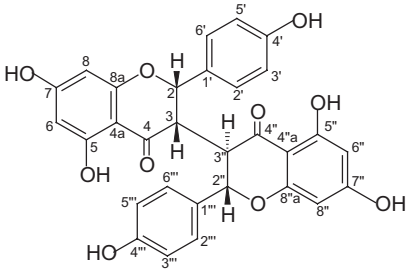
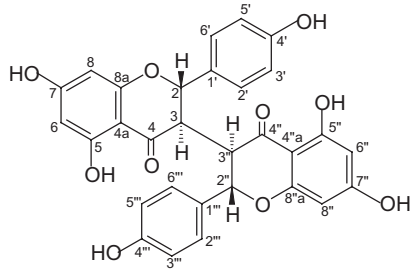


Neochamaejasmin A



Neochamaejasmin B



Chamaejasmine

Figure 1. Structures of biflavonoids isolated from *Stellera chamaejasme*.

Table 1. The distribution of biflavonoids in *S. chamaejasme*, *W. sikokiana*, *W. indica*, and *E. siamensis*.

	wikstrol A	wikstrol B	neochamaejasmin A	neochamaejasmin B	chamaejasmine
S. chamaejasme	+	+	+	+	+
W. indica	+	+			
W. sikokiana	+	+	+	+	
E. siamensis					+

156.7 (C-4′, 8″a), 157.5 (C-12″), 159.7 (C-5″), 161.0 (C-2″), 161.5 (C-7″), 180.8 (C-4″).

Neochamaejasmin A

Brown powder, mp 270 °C (dec). ^{1}H NMR (DMSO-d_6): δ 3.71 (1 H, br s, H-3, 3″), 4.79 (1 H, br s, H-2, 2″), 5.77 (1 H, d, J = 1.6 Hz, H-6, 6″), 5.90 (1 H, d, J = 1.6 Hz, H-8, 8″), 6.79 (2 H, d, J = 8.4 Hz, H-3′, 3‴, 5′, 5‴), 7.01 (2 H, d, J = 8.4 Hz, H-2′, 2‴, 6′, 6‴), 11.84 (1 H, s, 5, 5″-OH). ^{13}C NMR (DMSO-d_6): δ 47.4 (C-3, 3″), 80.8 (C-2, 2″), 95.3 (C-8, 8″), 96.5 (C-6, 6″), 101.3 (C-10, 10″), 115.7 (C-3′, 3‴, 5′, 5‴), 126.9 (C-1′, 1‴), 129.7 (C-2′, 2‴, 6′, 6‴), 158.5 (C-4′, 4‴), 162.7 (C-9, 9″), 163.7 (C-5, 5″), 167.2 (C-7, 7″), 195.1 (C-4, 4″).

Neochamaejasmin B

Brown powder, mp 225 °C (dec). ^{1}H NMR (CD_3OD): δ 3.13 (1 H, m, H-3), 3.26 (1 H, dd, J = 9.0, 3.4 Hz, H-3″), 5.13 (1 H, d, J = 9.0 Hz, H-2″), 5.52 (1 H, d, J = 4.8 Hz, H-2), 5.75 (1 H, br s, H-6), 5.77 (1 H, br s, H-6″), 5.86 (1 H, br s, H-8), 5.97 (1 H, br s, H-8″), 6.64 (2 H, d, J = 8.3 Hz, H-3′, 5′), 6.78 (2 H, d, J = 8.3 Hz, H-3‴, 5‴), 6.92 (2 H, d, J = 8.3 Hz, H-2′, 6′), 7.13 (2 H, d, J = 8.3 Hz, H-2‴, 6‴). ^{13}C NMR (CD_3OD): δ 49.6 (C-3), 50.7 (C-3″), 81.4 (C-2), 83.2 (C-2″), 96.0 (C-8), 96.4 (C-8″), 97.0 (C-6), 97.3 (C-6″), 103.8 (C-10), 105.0 (C-10″), 116.1 (C-3′, 5′), 116.4 (C-3‴, 5‴), 128.5 (C-2′, 6′), 128.7 (C-1′), 129.0 (C-1‴), 130.2 (C-2‴, 6‴), 158.5 (C-4′), 158.9 (C-4‴), 163.3 (C-9), 165.1 (C-5, 9″), 165.4 (C-5″), 168.2 (C-7), 168.4 (C-7″), 196.1 (C-4), 198.5 (C-4″).

Chamaejasmine

Brown powder, mp 235~237 °C (CH_3OH). ^{1}H NMR (DMSO-d_6): δ 2.77 (1 H, d, J = 12.0 Hz, H-3, 3″), 5.69 (1 H, d, J = 12.0 Hz, H-2, 2″), 5.77 (1 H, s, H-6, 6″), 5.85 (1 H, s, H-8, 8″), 6.79 (2 H, d, J = 8.2 Hz, H-3′, 3‴, 5′, 5‴), 6.86 (2 H, d, J = 8.2 Hz, H-2′, 2‴, 6′, 6‴), 11.78 (1 H, br s, 5, 5″-OH). ^{13}C NMR (DMSO-d_6): δ 49.1 (C-3, 3″), 83.2 (C-2, 2″), 95.3 (C-8, 8″), 96.4 (C-6, 6″), 101.5 (C-10, 10″), 115.5 (C-3′, 3‴, 5′, 5‴), 126.6 (C-1′, 1‴), 129.3 (C-2′, 2‴, 6′, 6‴), 158.4 (C-4′, 4‴), 162.5 (C-9, 9″), 163.6 (C-5, 5″), 167.8 (C-7, 7″), 196.0 (C-4, 4″).

Results and discussion

From *Stellera chamaejasme*, five biflavonoids were obtained, of which wikstrol A and wikstrol B were isolated from this plant for the first time. Wistrol A and wikstrol B were biflavonoids formed by the connection of C-3 of a flavone with C-8 of a flavan-3-ol and the other three were C-3 dimeric flavanones. Wikstrol A and wikstrol B were isomers with axial chirality of C-8/C-3″.

Recently, such biflavonoids have also been found in *Wikstroemia sikokiana* (Baba et al., 1994), *W. indica* (Hu et al., 2000), and *Enkleia siamensis* (Boonyaratanakornkit et al., 1991), all of which belong to Thymelaeaceae. The distribution of biflavonoids in these four plants, listed in Table 1, appears to indicate that the chemical relationship between the genera *Stellera* and *Wikstroemia* is closer than that between *Stellera* and *Enkleia*.

Further study should be conducted by examining biflavonoids from more plants of different genera of this familia to find authentic evidence of chemotaxonomy. The extension of chemical research from qualitative identification to quantitative determination will be necessary.

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