



Review Article

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Chemical Constituents from the Leaves of *Machilus Bombycina* King Ex Hook.f.

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Abstract

Machilus bombycina King ex Hook. f. (*Lauraceae*) is found in China and India. Its leaves are used to cure pimples and rheumatism. Column chromatography of a methanolic extract of the leaves of *M. bombycina* afforded 8, 21-dihydroxylanost-5, 25-dien-3-olyl behenate (1) and 5,7-dihydroxy-3', 4'-dimethoxyflavanone-7-O- α -D-glucopyranosyl-(6'' \rightarrow 1''')- α -D-rhamnopyranoside (2) as the new phytoconstituents.

a. Results and Discussion: Compound 1, named 8, 21-dihydroxylanostdien-3-olyl behenate, responded positively to Liebermann-Burchard test for triterpenoids and showed IR absorption bands for hydroxyl groups (3510, 3416 cm^{-1}), ester function (1721 cm^{-1}), unsaturation (1640 cm^{-1}) and long aliphatic chain (727 cm^{-1}). On the basis of mass and ^{13}C NMR spectra the molecular ion peak of 1 was determined at m/z 780 consistent with a molecular formula of a lanostanyl ester, $\text{C}_{52}\text{H}_{92}\text{O}_4$. The ion fragments arising at m/z 457 [$\text{C}_{17}\text{-O}$ fission, $\text{C}_{30}\text{H}_{49}\text{O}_3$] $^+$, 339 [$\text{M}-457$, $\text{CH}_2(\text{CH}_2)_{20}\text{COO}$] $^+$, 440 [457-OH] $^+$, 127 [$\text{C}_{17}\text{-C}_{20}$ fission, $\text{C}_8\text{H}_{15}\text{O}$ side chain] $^+$ and 313 [440-side chain, $\text{C}_{30}\text{H}_{48}\text{O}_3$] $^+$ indicated that the attachment of a behenate group linked to the lanostandiene unit possessing a C8 side chain with a hydroxyl group and a vinylic bond. The ^1H NMR spectrum of 1 showed a one-proton multiplet at δ 5.37 assigned to vinylic H-5, two one-proton signals at δ 4.92 and 4.90 due to exocyclic methylene H_2 -26 protons, a one-proton double doublet at δ 4.38 with coupling interactions of 5.3 and 9.5 Hz attributed to oxymethine H-3 α proton, a two-proton doublet at δ 3.21 ($J=6.5$ Hz) accounted to hydroxymethylene H_2 -21 protons, six three-proton singlets between δ 1.92-1.01 accommodated to tertiary C-18 to C-30 methyl protons and a three-proton triplet at δ 0.83 ($J=6.1$ Hz) ascribed to primary C-22' methyl protons.

The other methylene and methine protons resonated as multiplets from δ 2.72 to 1.55 and as a broad signal at δ 1.28 (36 H). The ^{13}C NMR spectrum of 1 showed signals for ester carbon at δ 173.16 (C-1'), vinylic carbons between δ 142.97-116.13, oxymethine carbon at δ 79.30 (C-3), hydroxymethylene carbon at δ 63.06 (C-21), and methyl carbons from δ 21.09 to 14.04. The ^1H and ^{13}C NMR spectral data of the triterpenic unit of 1 were compared with the reported data of lanostene-type triterpenoids [1,2]. Acid hydrolysis of 1 yielded behenic acid, m. p. 79 – 80 $^{\circ}\text{C}$. On the basis of above discussion the structure of 1 has been elucidated as 8,21-dihydroxylanost-5,25-dien-3-olyl behenate, a new lanostane type-triterpenic ester.

Compound 2, named 3',4'-dimethoxyeriodictyol 7-O-glucorhamnoside, responded positively to phenolic and glycosidic tests and had UV absorption maxima at 287 and 328 nm typical for a flavanone derivative. It showed a bathochromic shift of 40 nm on addition of AlCl_3 and AlCl_3/HCl suggesting the presence of a chelated hydroxyl function at C-5. Its IR spectrum disclosed a characteristic absorption for the conjugated carbonyl group (1668 cm^{-1}) and hydroxyl groups (3510, 3418, 3217 cm^{-1}). On the basis of its mass and ^{13}C NMR spectra the molecular ion peak of 2 was determined at m/z 624 consistent with a molecular formula of a flavonoid diglycoside, $\text{C}_{29}\text{H}_{36}\text{O}_{15}$. The important ion peaks generated at m/z 609 [$\text{M}-\text{Me}$] $^+$, 581 [609-CO] $^+$, 147 [C_1''' -O fission, $\text{C}_6\text{H}_{11}\text{O}_4$] $^+$, 477 [$\text{M}-147$] $^+$, 326 [C7-O fission, $\text{C}_{12}\text{H}_{22}\text{O}_{10}$] $^+$, 298 [$\text{M}-326$, $\text{C}_{17}\text{H}_{14}\text{O}_3$] $^+$ and 315 [C_1''' -O fission, $\text{C}_{17}\text{H}_{15}\text{O}_6$] $^+$ supported that a dihexoside unit was linked with the ring A of the flavanone. The ion fragments produced at m/z 300 [315-Me] $^+$, 253 [315-2 x OMe] $^+$, 164 [$\text{C}_{3,4}\text{-C}_{2,0}$ fission] $^+$ and 150 [$\text{C}_{3,2}\text{-C}_{2,0}$ fission] $^+$ indicated the presence of two methoxy groups in ring B and methylene protons at C-3.

The ^1H NMR spectrum of 2 showed an ABX system of resonances as one-proton double doublets at δ 5.56 ($J=3.2, 12.9$ Hz, H-2), 3.19 ($J=2.8, 17.0$ Hz, H_2 -3a) and 2.81 ($J=3.2, 12.9$ Hz, H_2 -3b) characteristic of oxymethine H-2 and methylene H_2 -2 eq and H_2 -2 ax, respectively, of a flavanone moiety. Four one-proton doublets at δ 7.01 ($J=2.8$ Hz), 6.21 ($J=2.0$ Hz), 6.17 ($J=2.0$ Hz), 6.96 ($J=8.4$ Hz), and a one-proton double doublet at δ 6.99 ($J=2.8, 8.4$ Hz) were ascribed to meta-coupled H-2', H-8 and H-6, ortho-coupled H-5' and meta-ortho-coupled H-6' protons, respectively. Two one-proton doublets at δ 5.48 ($J=4.8$ Hz) and 5.27 ($J=5.3$ Hz), a three-proton doublet at δ 1.21 ($J=7.2$ Hz) and two three-proton broad signals at δ 3.82 and 3.44 were accounted correspondingly to anomeric H-1'' and H-1''', secondary methyl H_3 -2''' of rhamnose unit and two methoxy protons.

The ^{13}C NMR spectrum of 2 displayed 29 signals including a carbonyl carbon of a flavanone at δ 197.03 (C-4), oxymethine at δ 78.41 (C-2), methylene carbon of the flavanone at δ 42.23 (C-3), methoxy carbons at δ 55.60 and 58.57, anomeric carbons at δ 100.56 (C-1'') and 96.31 (C-1'''), methyl carbon at δ 17.81 (C-6''') and other sugar carbons between δ 76.20-65.97. The DEPT spectrum of 2 exhibited the presence of three methyl, two methylene, seven methine and eight quaternary carbons. The existence of the oxymethylene H_2 -6'' proton signal in the deshielded region as a doublet at δ 3.23 ($J=8.4$ Hz, H_2 -6'') and its C-6'' carbon signal at δ 65.97 (C-6'') suggested (6'' \rightarrow 1''') linkage of the sugar moieties.

Acid hydrolysis of 2 yielded dimethoxyeriodictyol, D-glucose, R_f 0.18 (n-butanol-acetic acid-water, 4:1:5) and D-rhamnose, R_f 0.86 (n-butanol-acetic acid-water, 4:1:1.6). On the basis of these evidences the structure of 2 was formulated as 5,7-dihydroxy-3',4'-dimethoxyflavanone-7-O- α -D-glucopyranosyl-(6'' \rightarrow 1''')- α -D-rhamnopyranoside, a new flavanone diglycoside.

Conclusion: Phytochemical investigation of a methanolic extract of the leaves of *Machilus bombycina* afforded a lanostene-type 3-olyl behenate and a flavanone-7-O- α -D-glucopyranosyl (6'' \rightarrow 1''')- α -D-rhamnopyranoside. This work has enhanced understanding about the phytoconstituents of these plants. These secondary metabolites can be used as analytical markers for quality control of these herbal drugs.

Keywords: *Machilus Bombycina* Leaves; Chemical Constituents; Isolation; Characterization

Introduction

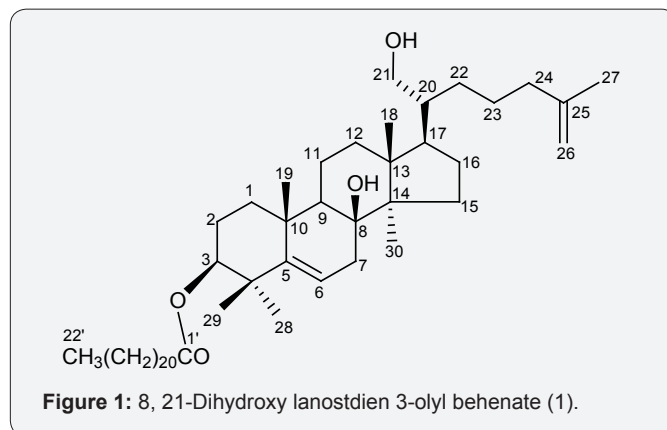
Machilus Bombycina King ex Hook. f. (*Lauraceae*), known as som, is distributed in China, India and other south eastern countries. It is one of the primary food plants of *Antheraea assama* Westwood, the silkworm that produces muga or golden colour natural silk. Its leaf paste is applied to relieve rheumatism. Juice of the leaves of *M. bombycina* and *Achyrenthus aspera* is applied to cure pimples [3]. Chlorogenic acid, phytic acid, tannins, catechol, morin, gallic acid, β -sitosterol and its 3-O-glucoside are reported from the leaves [4]. The plant essential oil was mainly consisted of decanal, 11-dodecenal and dodecanal [5]. The major constituents of the flower oil were caryophyllene oxide, (E)-nerolidol, 11-dodecenal and 11-dodecenoic acid. The fruit oil was composed of the furanoid forms of *trans*- and *cis*-linalool oxides [6,7]. The manuscript describes isolation and characterization of one each of triterpenic ester and flavanone glycoside from the leaves of *M. bombycina*.

Extraction and Isolation

The leaves of *M. bombycina* (1 kg), procured from Dibrugarh, Assam were coarsely powdered and extracted exhaustively separately with methanol in a Soxhlet apparatus. The extracts were concentrated under reduced pressure to get a dark brown mass (115.6). The dried residue (100 g) was dissolved in minimum amount of methanol and adsorbed on silica gel column grade (60-120 mesh) to prepare a slurry. It was air-dried and chromatographed over silica gel columns loaded in chloroform. The column was eluted with chloroform and chloroform - methanol mixtures to isolate the compounds 1 and 2.

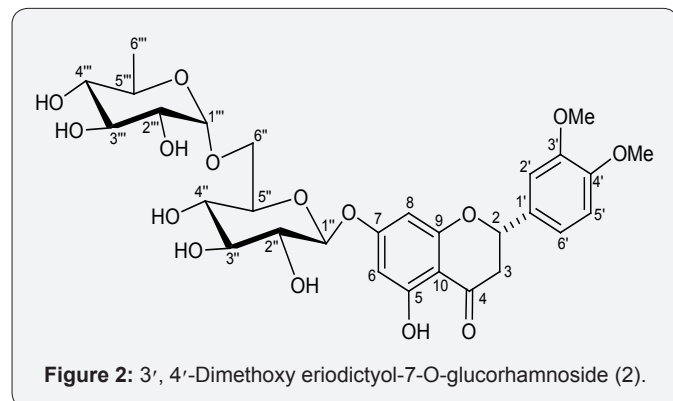
a. 8,21-Dihydroxylanostdien-3-olyl behenate (1): Elution of the column with chloroform-methanol (49:1) produced colorless crystals of 1, yield 163 mg, m. p. 233-235 °C; IR ν_{\max} (KBr): 3510, 3416, 2918, 2854, 1721, 1640, 1461, 1373, 1272, 1123, 727 cm^{-1} ; ^1H NMR (CDCl_3): δ 5.37 (1H, m, H-5), 4.92 (1H, s, H_2 -26a), 4.90 (1H, s, H_2 -26b), 4.38 (1H, dd, J = 5.3, 9.5 Hz, H-3 α), 3.21 (2H, d, J = 6.5 Hz, H_2 -21), 1.92 (3H, brs, Me-27), 1.21 (3H, s, Me-28), 1.19 (3H, brs, Me-29), 1.16 (3H, brs, Me-19), 1.14 (3H, brs, Me-30), 1.01 (3H, brs, Me-18), 2.72 (2H, m, H_2 -7), 2.02 - 1.33 (21H, m, $9 \times \text{CH}_2$, $3 \times \text{CH}$), 2.35 (2H, t, J = 7.5 Hz, H_2 -2'), 1.55 (2H, m, H_2 -3'), 1.28 (36 H, brs, $18 \times \text{CH}_2$), 0.83 (3H, t, J = 6.1 Hz, Me-22'); ^{13}C NMR (CDCl_3): δ 39.34 (C-1), 25.75 (C-2), 79.30 (C-3), 42.32 (C-4), 142.97 (C-5), 121.71 (C-6), 28.26 (C-7), 71.81 (C-8), 50.12 (C-9), 36.79 (C-10), 23.07 (C-11), 29.13 (C-12), 45.42

(C-13), 56.76 (C-14), 33.94 (C-15), 31.60 (C-16), 59.55 (C-17), 11.79 (C-18), 21.09 (C-19), 30.79 (C-20), 63.06 (C-21), 37.25 (C-22), 24.14 (C-23), 42.83 (C-24), 140.74 (C-25), 116.13 (C-26), 19.40 (C-27), 19.15 (C-28), 19.04 (C-29), 18.79 (C-30), 173.16 (C-1'), 56.05 (C-2'), 47.87 (C-3'), 30.68 (C-4'), 29.72 (C-5'), 29.69 (C-6'), 29.64 (C-7'), 29.45 (C-8'), 29.28 (C-9' to C-14'), 29.13 (C-15' to C-18'), 27.23 (C-19'), 24.31 (C-20'), 21.11 (C-21'), 14.04 (C-22'); ESI MS m/z (rel. int.): 780 [M]⁺ ($\text{C}_{52}\text{H}_{92}\text{O}_4$) (16.2), 457 (12.5), 440 (20.5), 339 (32.8), 313 (31.8), 127 (23.6) (Figure 1).



b. 3',4'-Dimethoxyeriodictyol 7-O- glucorhamnoside (2): Elution of column with chloroform-methanol (3:1) furnished yellow crystals of 2, yield 155 mg, m.p. 174-176 °C, UV λ_{\max} (MeOH): 287, 328 nm; IR ν_{\max} (KBr): 3510, 3418, 3217, 2927, 2835, 1668, 1601, 1453, 1405, 1326, 1127, 1076 cm^{-1} ; ^1H NMR ($\text{DMSO}-d_6$): δ 7.01 (1H, d, J = 2.8 Hz, H-2'), 6.99 (1H, dd, J = 2.8, 8.4 Hz, H-6'), 6.96 (1H, d, J = 8.4 Hz, H-5'), 6.21 (1H, d, J = 2.0 Hz, H-8), 6.17 (1H, d, J = 2.0 Hz, H-6), 5.56 (1H, dd, J = 3.2, 12.9 Hz, H-2), 3.19 (1H, dd, J = 2.8, 17.0 Hz, H_2 -3a), 2.81 (1H, dd, J = 3.2, 12.9 Hz, H_2 -3b), 3.82 (3H, brs, OMe), 3.44 (3H, brs, OMe), 5.48 (1H, d, J = 4.8 Hz, H-1''), 4.84 (1H, m, H-5''), 4.71 (1H, dd, J = 4.8, 6.4 Hz, H-2''), 3.69 (1H, m, H-3'), 3.42 (1H, m, H-4'), 3.23 (2H, d, J = 8.4 Hz, H_2 -6''), 5.27 (1H, d, J = 5.3 Hz, H-1'''), 4.79 (1H, m, H-5'''), 4.58 (1H, m, H-2'''), 3.51 (1H, m, H-3'''), 3.35 (1H, m, H-4'''), 1.21 (3H, d, J = 7.2 Hz, Me-6'''), ^{13}C NMR ($\text{DMSO}-d_6$): δ 78.41 (C-2), 42.23 (C-3), 197.03 (C-4), 162.96 (C-5), 99.14 (C-6), 165.07 (C-7), 95.48 (C-8), 162.50 (C-9), 103.25 (C-10), 130.89 (C-1'), 114.09 (C-2'), 146.35 (C-3'), 147.90 (C-4'), 117.94 (C-5'), 111.98 (C-6'), 55.60 (OMe), 58.57 (OMe), 100.56 (C-1''), 72.01 (C-2''), 70.63 (C-3''), 69.52 (C-4''), 76.20 (C-5''), 65.97 (C-6''),

96.31 (C-1'''), 72.92 (C-2''), 70.22 (C-3'''), 68.29 (C-4'''), 75.44 (C-5'''), 17.81 (C-6'''); ESI MS m/z (rel. int.): 624 [M]⁺ (C₂₉H₃₆O₁₅) (5.2), 609 (5.7), 581 (12.1), 477 (14.9), 326 (25.8), 315 (11.9), 300 (33.6), 298 (32.1), 253 (38.7), 164 (21.2), 150 (25.5), 147 (22.3), 105 (35.8) (Figure 2).



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