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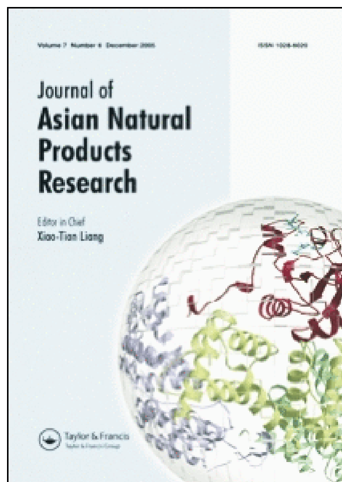

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A New Flavone Glycoside, 5-Hydroxy 7,3',4',5'-Tetra-Methoxyflavone 5-O-β-D-Xylopyranosyl-(1⟶2)-α-L-Rhamnopyranoside from *Bauhinia Variegata* Linn

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A NEW FLAVONE GLYCOSIDE, 5-HYDROXY 7,3′,4′,5′-TETRA-METHOXYFLAVONE 5-O-β-D-XYLOPYRANOSYL-(1→2)-α-L-RHAMNOPYRANOSIDE FROM *BAUHINIA VARIEGATA* LINN

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A new flavone glycoside m.f. $C_{30}H_{36}O_{15}$, m.p. 252–253°C, $[M]^+$ 636 (EIMS) was isolated from the acetone soluble fraction of the concentrated 95% ethanolic extract of the seeds of *Bauhinia variegata* (Linn). It was identified as 5-hydroxy7,3′,4′,5′-tetra-methoxyflavone 5-O-β-D-xylopyranosyl-(1→2)-α-L-rhamnopyranoside (1) by various colour reactions, chemical degradations and spectral techniques.

Keywords: *Bauhinia variegata* (Linn); Leguminosae; Flavonoid

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INTRODUCTION

Bauhinia variegata (Linn.), [1–3] belongs to Leguminosae family and is commonly known as "Kachanar" in Hindi. It is distributed almost throughout India. The plant is used for treatment of skin diseases. Its roots are also used in snake poison. The present paper deals with the isolation and structural elucidation of a new flavone glycoside, 5-hydroxy7,3′,4′,5′-tetra-methoxyflavone 5-O-β-D-xylopyranosyl-(1→2)-α-L-rhamnopyranoside on the basis of various chemical degradations and spectral analysis.

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RESULTS AND DISCUSSION

A new compound I isolated from acetone soluble fraction of the 95% exthanolic extract of the seeds of *B.variegata* has the molecular formula $C_{30}H_{36}O_{15}$, m.p. 252–253°C, $[M]^+$ 636 (EIMS) and gave all the characteristic reactions of flavone. Its IR spectrum showed a strong absorption band at 3395–3485 cm^{-1} (—OH groups), 1620 (C=O), 2985 (C—H), 2872 (OMe), 1495–1020 (O-gly) and 870 cm^{-1}. The ^{1}H-NMR spectrum of I showed four singlets at δ 3.83, 3.84, 3.70, 3.84 which were assigned to four methoxy groups at C-7, C-3′, C-4′, C-5′ positions and two aromatic protons as one singlet at δ 7.22. assigned to 2′, and 6′ positions respectively. The anomeric proton signals at δ 5.36 (1H, br, s) and δ 4.36 (1H, d, $J = 7.6$ Hz) were assigned to H-1″ and H-1‴ of rhamnose and xylose respectively and a doublet at δ 1.06 was due to the rhamnosyl methyl group.

The position of sugar moiety in compound I was established by permethylation [4] of I followed by acid hydrolysis which afforded methylated sugars identified as 3,4-di-O-methyl-L-rhamnose and 2,3,4-tri-O-methyl xylose(by Co-PC and Co-TLC) according to Petek [5], suggesting that the C-1‴ of xylose was linked with C-2″ of rhamnose and C-1″ of rhamnose was attached to C-5 of aglycone. The inter linkage ($1 \rightarrow 2$) between both sugars were further confirmed by its ^{13}C-NMR spectrum (see Experimental Section).

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Acid hydrolysis of compound I with 7% ethanolic H_2SO_4 yielded aglycone II, m.f. $C_{19}H_{18}O_7$, m.p. 263–264°C and $[M]^+$ 358 and identified as 5-hydroxy7,3′,4′,5′-tetra-methoxyflavone by comparison of its spectral data with literature values [6].

The aqueous hydrolysate obtained after acid hydrolysis of compound I was neutralised with $BaCO_3$ and $BaSO_4$ filtered off. After concentration, it was subjected to PC and sugars were identified as rhamnose (R_f 0.34) and xylose (R_f 0.28) (Co-PC and Co-TLC). Periodate oxidation [7] of compound I further confirmed that both sugars were present in pyranose form.

Enzymatic hydrolysis of compound I with almond emulsion liberated xylose first showing the presence of β-linkage between xylose and rhamnose, and on hydrolysis with Takadiastase liberated rhamnose confirming the presence of the α-linkage between aglycone and rhamnose.

On the basis of above evidences, the structure of compound I was assigned as a new flavone glycoside 5-hydroxy7,3′,4′,5′-tetra-methoxy-flavone 5-O-β-D-xylopy-ranosyl-($1 \rightarrow 2$)-α-L-rhamnopyranoside.

EXPERIMENTAL SECTION

General Experimental Procedures

Melting points are uncorrected. The IR spectra were recorded in KBr disc. ^{1}H-NMR spectra were run at 400 MHz using TMS as internal standard and $CDCl_3$ as solvent. ^{13}C-NMR spectra were run at 100 MHz using DMSO-d_6 as solvent.

PLANT MATERIAL

The seeds of *Bauhinia variegata* (Linn) were collected around Sagar region and was Taxonomically authenticated by Taxonomist of Botany Department of Dr. H. S. Gour University, Sagar. The voucher specimen was deposited in the Natural Products Laboratory, Department of Chemistry, Dr. H. S. Gour University, Sagar (M.P.).

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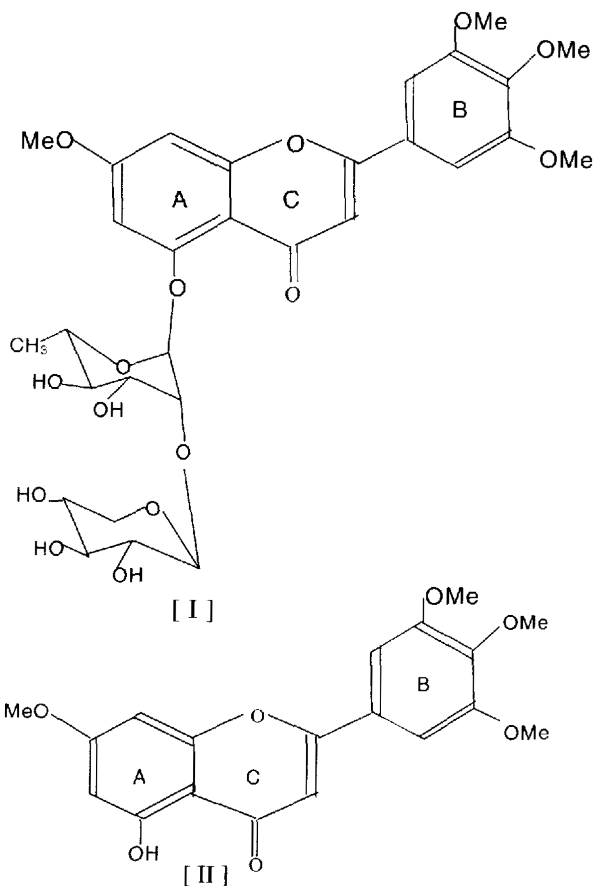
EXTRACTION AND ISOLATION

The air-dried and powdered seeds (2.5 kg) of *B. variegata* were extracted with 95% EtOH in a Soxhlet extractor. The total ethanolic extract was concentrated under reduced pressure to give a brownish viscous mass which was successively extracted with petroleum ether (60–80°C), chloroform, benzene, ethylacetate, acetone and methanol. The acetone soluble fraction of the ethanolic extract of the plant was concentrated under reduced pressure to give compound I as light yellow needles, which showed single spot on TLC examination using solvent system (C_6H_6–MeOH–H_2O, 5 : 3 : 1) m.p. 252–253°C. It has the molecular formula $C_{30}H_{36}O_{15}$, $[M]^+$ 636 (EIMS) (Elemental analysis calcd. for $C_{30}H_{36}O_{15}$: C 56.60, H 5.60, found: C 56.38, H 5.30), IR ν_{max}^{KBr}: 3395–3485 cm^{-1}. (—OH groups), 1620 (C=O), 2985 (C—H), 2872 (OMe), 1495–1020 (O-gly) and 870 cm^{-1}. ^{1}H-NMR (400 MHz – $CDCl_3$) at δ 3.83 (3H, s, C-7 OMe); 3.84 (3H, s, C-3′ OMe); 3.70 (3H, s, C-4′ OMe); 3.84 (3H, s, C-5′ OMe); δ 6.78 (1H, s, H-3); δ 6.86 (1H, d, J = 2.5 Hz, H-6); δ 6.98 (1H, d, J = 2.5 Hz, H-8); δ 7.22 (1H, s, H-2′); δ 7.22 (1H, s, H-6′); 5.36 (1H, br, s, H-1″); 4.14 (1H, br, d, J = 3.8 Hz, H-2″); 3.83 (1H, dd, H-3″); 3.26 (1H, dd, H-4″); 3.68 (1H, d, H-5″); 1.04 (3H, d, J = 6.0 Hz, Rham – Me); 4.36 (1H, J = 7.6 Hz, H-1‴); 3.26 (1H, dd, H-2‴); 3.34 (1H, dd, H-3‴); 3.36 (1H, H-4‴); 3.14 (2H, dd, H-5‴). ^{13}C-NMR



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(100MHz, DMSO-d_6) 161.2 (C-2); 108.3 (C-3); 177.6 (C-4); 158.3 (C-5); 103.2 (C-6); 164.6 (C-7); 96.7 (C-8); 159.3 (C-9); 109.6 (C-10); 126.2 (C-1′); 104.3 (C-2′); 153.5 (C-3′); 141.3 (C-4′); 153.3 (C-5′); 104.4 (C-6′); 56.2 (OMe-7); 56.4 (OMe-3′); 60.4 (OMe-4′); 56.8 (OMe-5′); 103.4(C-1″); 82.2(C-2″); 71.6 (C-3″); 74.3 (C-4″); 72.2 (C-5″); 18.6 (C-6″); 108.3 (C-1‴); 74.6 (C-2‴); 77.3 (C-3‴); 70.4 (C-4‴); 67.3 (C-5‴).

ACID HYDROLYSIS OF COMPOUND I

100 mg of compound I was dissolved in EtOH (20 ml) and refluxed with 15 ml of 7% H_2SO_4 on water bath for 9–10 h. The contents were concentrated and allowed to cool and the residue was extracted with

Et_2O. The ethereal layer was washed with water and the residue was chromatographed over silica-gel using $CHCl_3$–MeOH (5:3) to give compound II, $C_{19}H_{18}O_7$, m.p. 263–264°C, $[M]^+$ 358 (EIMS) (Elemental analysis; found C 63.89, H 5.20; calcd. for $C_{19}H_{18}O_7$, C 63.68, H 5.02).

The aqueous hydrolysate obtained after acid hydrolysis was neutralised with $BaCO_3$, and the $BaSO_4$ filtered off. The filtrate was concentrated and subjected to paper chromatography examination (n-BuOH–AcOH–H_2O 4:1:5) as solvent and Ninhydrin as detecting agent showed the presence of L-rhamnose (R_f 0.34) and xylose (R_f 0.28) (Co-PC and Co-TLC).

PERMETHYLATION FOLLOWED BY ACID HYDROLYSIS OF COMPOUND I

Compound I was refluxed for 24 h with MeI (4 ml) and Ag_2O (40 mg) in DMF (5 ml) and then filtered. The filtrate dried in vacuum and hydrolysed with 10% ethanolic H_2SO_4 for 8–10 h, yielded methylated aglycone identified as 5,7,3′,4′,5′-pentamethoxy flavone and methylated sugars, which were identified as 3,4-di-O-methyl-L-rhamnose and 2,3,4-tri-O-methyl-xylose according to Petek.

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PERIODATE OXIDATION OF COMPOUND I

Compound I was dissolved in MeOH and treated with sodium meta periodate for two days. The liberation of formic acid and consumed periodate were estimated by Jones method, which also showed that both the sugars were present in pyranose form.

ENZYMATIC HYDROLYSIS OF THE COMPOUND I

The compound I (40 mg) was dissolved in MeOH (15 ml) and on hydrolysis with equal volume of almond emulsion at room temperature yielded D-xylose indicating the presence of β-Linkage between D-xylose and L-rhamnose and on hydrolysis with Takadiastase yielded L-rhamnose and aglycone showing the presence of α-linkage between L-rhamnose and aglycone.

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