A NEW GLYCOFLAVANONE FROM *CLEOME VISCOSA* WHOLE PLANT

S. K. SRIVASTAVA AND (SMT.) S. D. SRIVASTAVA
Department of Chemistry, University of Saugar, Sagar 470 003, *M.P., India*

ABSTRACT

A new glycoflavanone has been isolated from the whole plant of *Cleome viscosa*. It has been assigned as 3′, 4′-dihydroxy-5-methoxy flavanone-7-0-*a*-L-rhamnopyranoside on the basis of spectroscopic and chemical studies.

CLEOME VISCOSA (No. Capparidaceae) is reputed for its medicinal importance[1,2] in our indigenous system of medicines. Leaves are used as rubefacient, vesicant, sudorific and external application for wounds and ulcers. Seeds are carminative and anthelmintic. The plant was selected for chemical examination because no work is reported in the literature.

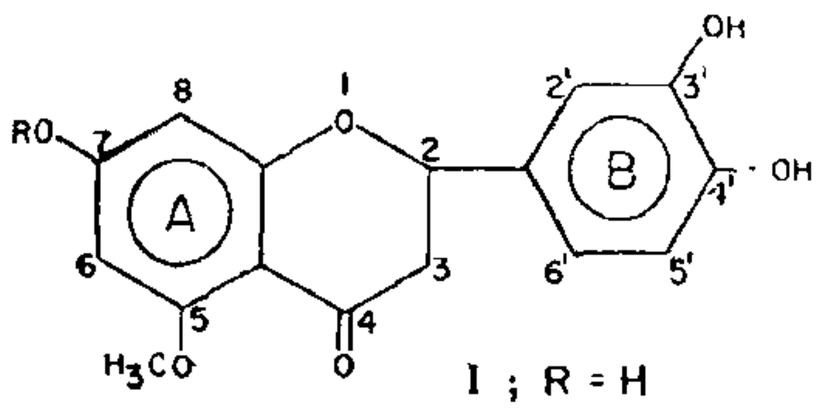
RESULTS AND DISCUSSION

From the water soluble part of the ethanolic extract of air-dried powdered plant, a dark yellow coloured compound, $C_{22}H_{24}O_{10}$ was isolated which gave the characteristic reactions of flavanone and a glycoside. Acid hydrolysis of the glycoside gave one mole of rhamnose and an aglycone, named as 7, 3′, 4′-trihydroxy-5-methoxy flavanone (I).

The aglycone, $C_{16}H_{14}O_6$, m.p. 110° (d), ν_{max}^{KBr} 3400 cm^{-1} (OH) was analysed for three OH groups (acetate) and one $-OCH_3$ group (Zeisel; ν_{max}^{KBr} 2870 and 1170 cm^{-1}). On demethylation (HBr/AcOH) aglycone yielded a tetrahydroxy compound, m.p. 267–68° which was found to be identical (m.p., m.m.p., R_f, UV and degradation studies) with eriodictyol[3]. The methoxyl group was located at position-5, since the aglycone and the glycoside separately on alkaline degradation[4] yielded protocatecheuic, m.p. 219° and phloroglucinol monomethyl ether, m.p. 75° and protocatecheuic acid respectively. The position of the $-OCH_3$ group was further confirmed by the absence of bathochromic shift with $AlCl_3$[5]. Thus the aglycone was assigned the structure of 7, 3′, 4′-trihydroxy-5-methoxy flavanone (I), new one.

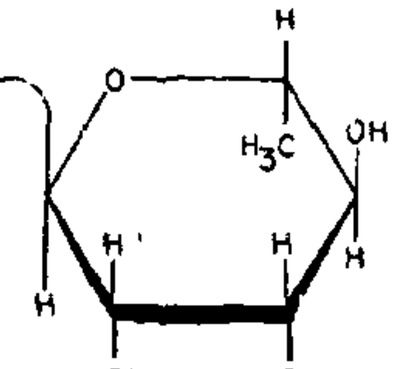
The periodate oxidation[6] showed the consumption of 2·00 moles of periodate with the production of 1·00 mole of formic acid per mole of the glycoside suggesting the presence of only one unit of rhamnose in pyranose form.

The position of the sugar in the glycoside was determined by the comparison of the properties of the glycoside with those of the aglycone. The aglycone gave a positive bathochromic shift with fused NaOAc[7] for free OH group at position-7 but not the glycoside. The glycoside was methylated with $(CH_3)_2SO_4/K_2CO_3$ and followed by acidic hydrolysis to get 5, 3′, 4′-trimethyl ether of eriodictyol[3]. Thus obviously the sugar moiety is attached at position-7 of the aglycone. The result of tokadiastase enzymatic hydrolysis revealed that rhamnose is *a*-linked. Hence, the glycoside is 3′, 4′-dihydroxy 5-methoxy flavanone-7-0-*a*-L-rhamnopyranoside(II).



I ; R = H

II ; R =



EXPERIMENTAL

Isolation and Purification

The air-dried and powdered whole plant (2 kg) of *Cleome viscosa* was extracted with EtOH under reflux for 150 h and the extract was concentrated in a rotavapour to 100 ml. It was then poured into excess of H_2O (500 ml). The water insoluble material was filtered off and the filtrate was extracted with a number of organic solvents in the order of their increasing polarities in liquid-liquid extractor. The EtOAc soluble fraction deposited the reported glycoside in the refrigerator. It was filtered and purified over the column of magnesol, crystallised in yellow needles from petroleum ether: EtOAc mixture, m.p. 68–70°. The homogeneity of the glycoside was checked by TLC (R_f 0·28 in MeOH : C_6H_6, 5 : 5 v/v; 0·81 in *n*-BAW 4 : 1 : 5 v/v) and PC (R_f 0·91 in *n*-BAW, 4 : 1 : 5 v/v), yield, 0·039%, λ_{max}^{EtOH} 290 nm; $\lambda_{max}^{EtOH+AlCl_3}$ 288 nm; $\lambda_{max}^{EtOH+NaOAc}$ 290 nm. ν_{max}^{KBr} 825, 1025, 1120, 1170, 1280, 1360, 1460, 1510, 1600, 1685, 2870, 2900 and 3450 cm^{-1} (Found; C, 58·89, H, 5·35; $C_{22}H_{24}O_{10}$ requires; C, 58·90; H, 5·35%).

Acidic Hydrolysis

The glycoside (500 mg) was hydrolysed with boiling with 7% ethanolic H_2SO_4 (50 ml) for 6 h. After cooling, the contents were diluted to 50% with H_2O,

The precipitated aglycone was separated by filtration. The aqueous solution was neutralised ($BaCO_3$), filtered and evaporated under reduced pressure to a syrup which was found to contain rhamnose by paper cochromatography with an authentic sample and osazone formation, m.p. 191°. Quantitative analysis[6] revealed the presence of 1 mole of sugar.

The aglycone was purified by paper chromatography and crystallised from EtOAc: petroleum ether mixture as yellow needles, m.p. 110° (d). The homogeneity was checked by TLC and PC in different organic solvent systems. λ_{max}^{EtOH} 290, 330 (sh) nm; $\lambda_{max}^{EtOH+AlCl_3}$ 288 nm; $\lambda_{max}^{EtOH+NaOAc}$ 323 nm.; ν_{max}^{KBr} 1020, 1120, 1170, 1205, 1265, 1340, 1360, 1420, 1450, 1500, 1550, 1600, 1680, 2870, 2920 and 3400 cm^{-1} (Found: C, 63·55; H, 4·62; OCH_3, 10·25; $C_{16}H_{14}O_6$ requires; C, 63·57; H, 4·63. $-OCH_3$; 10·26%). Acetyl derivative prepared by Ac_2O/Py method and crystallised from acetone: methanol as pale yellow needles melted at 130–35° (d) and did not give any colour with $FeCl_3$ (Found; C, 61·67; H, 4·66; acetyl, 30·14; $C_{22}H_{20}O_9$ requires; C, 61·68; H, 4·67; 3 × acetyl, 30·14%).

Demethylation of the Aglycone

The aglycone (150 mg) in glacial acetic acid (10 ml) was refluxed with 50% HBr (10 ml) for 10 h and worked up as usual. The resultant product was crystallised from EtOAc: Petroleum ether 1 : 1 v/v) as light yellow needles, m.p. 267–68°; λ_{max}^{EtOH} 290 nm; $\lambda_{max}^{EtOH+AlCl_3}$ 315 nm; $\lambda_{max}^{EtOH+NaOAc}$ 325 nm; R_f 0·85 in butanol: 27% gl. ACOH; (1 : 1 v/v). It was identified as eriodictyol by spectral means, colour reactions and degradation studies.

NMR Spectrum of the Aglycone

It was recorded at 100 MHz; $(CD_3)_2CO$; −60° C; signals at τ, 7·21 (d, Jgem 17Hz), 3H (eq); 6·70 (q Jgem 2Hz), 3H (ax); 6·20 (s), OMe; 2·91 (s) 6 and 8–H; 3·20 (5′–H); 2·2 (2′ and 6′–H).

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ADENOSINE TRIPHOSPHATASE ACTIVITY IN THE SPERMATOGENIC AND ANDROGENIC COMPONENTS OF THE TESTIS OF *TAPHOZOUS LONGIMANUS* HARDWICKE (MICROCHIROPTERA : MAMMALIA)

D. R. SWAMI* AND S. B. LALL

Department of Zoology, University of Udaipur, Udaipur 313 001

ABSTRACT

Histochemical site and distribution of adenosine triphosphatase (ATPase) activity in the spermatogenic and androgenic cells of the testis of sexually mature males of an insectivorous Microchiroptera, *Taphozous longimanus* Hardwicke exhibited differences of intensity. It is suggested that varying levels of activity of this enzyme represent the metabolic levels of testicular cell populations and energy requirements in relation to the transport of chemicals, secretion, meiosis and process of differentiation and maturation during spermatogenesis. ATPase may also serve as one of the "link" enzymes which regulate the catalysis of spermatozoal fructolysis and glycolysis.

HISTOCHEMICAL and biochemical evidence indicates that cellular specialisation during spermatogenesis is accompanied by molecular individualisation. Enzyme studies have offered new insight into the biochemical changes associated with meiosis, differentiation and maturation in the mammalian testes[1–9]. Once the profile of various testicular enzymes is known, it would be possible to use them as "finger prints" for elucidating control and regulatory mechanism(s) of spermatogenesis. However, despite their worldwide distribution, unique aerial life and curious reproductive mechanisms, there is no study on testicular

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