

A new anthraquinone glycoside from the pods of *Cassia glauca*, Lam

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Abstract : The present paper deals with the isolation and structural elucidation of a new anthraquinone glycoside, 5,7-dihydroxy -4'-methoxy flavonol-3-O- β -D-glactopyranoside from the pods of *Cassia glauca*, Lam.

using TMS as internal standards and mass spectra on a JEOL JMS 300 spectrometer at 70 eV. And 300°C. Purity of the compounds were checked by paper chromatography (PC) and Thin layer chromatography (TLC) using plates coated with silica gel G.

(Key words : *Cassia glauca*, anthraquinone)

Introduction

In course of our study on species of cassia genus (Rai *et al.*¹⁻³ 1991, 1994, 2006), a systematic chemical investigation of pods of *C. glauca* (Leguminosae) has been undertaken. The plant is highly reputed for its medicinal value (Hooker^{4,5}, 1879) and colouring matters (Heines, 1961). This paper reports the isolation of a new anthraquinone glycoside on the basis of chemical and spectral evidences.

Experimental

Materials and Methods

The pods of the plants were procured from Sanjay Gandhi Jaivik Uddyan, Patna and identified in the Deptt. of Botany, Patna University, Patna.

Melting points were determined in open capillaries and are uncorrected. UV spectra (MeOH) were recorded on a Beckman DU-6 spectrophotometer, IR spectra (KBr) on a Perkin-Elmer 577 spectrometer, NMR spectra (DMSO-d₆) on a Perkin-Elmer R-32 spectrometer (300 MHz)

Results and Discussion

Air-dried and defatted powdered pods (3kg) of the plant were successively extracted with acetone and alcohol.

The alcoholic extract was concentrated to a thick syrupy mass under reduced pressure, chromatographed on silica gel column and eluted with different solvents in order of increasing polarity. Elution of the column with ethyl acetate: methanol (1:1 v/v) eluate yielded a compound which was crystallized from EtOAc-Pet. Ether mixture as orange amorphous solid (1.05gm). It responded to characteristic colour reactions of anthraquinone (Thomson⁶, 1971; Tiwary *et al.*⁷ 1979) and glycoside (Bhatt *et al.*⁸ 1973). The compound was analysed for C₂₂H₂₂O₁₁ (Elemental analysis); mp 140-41p; (Found : C, 57.88; H, 4.76. Calculated for C₂₂H₂₂O₁₁ (C, 57.14; H, 4.76%); R_f = 0.78 (PC, BAW = 4:1:5 v/v) and R_f = 0.60 (20% AcOH), $\nu_{\max}$ (MeOH) 268, 355; (+AlCl₃) 271, 401; (+AlCl₃/HCl) 272, 401; (+NaOAc) 280, 358 (NaOAc/H₃PO₃) 267, 355nm; $\nu_{\max}$ (KBr) 3400 (OH), 2875 (OMe), 1640 (chelated C=O), 1520 and 1420cm⁻¹ (aromatic). The glycoside on acid hydrolysis with 7% H₂SO₄, afforded an orange

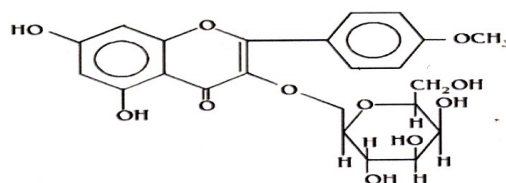
aglycone $C_{16}H_{12}O_6$ (elemental analysis), mp 228-29°C, Rf = 0.90 (PC, BAW = 4:1:5v/v) $\nu_{\max}$ (MeOH) 267, 367; (+AlCl₃) 271, 423; (+AlCl₃/HCl) 270, 422; (+NaOAc) 279, 368nm; $\nu_{\max}$ (KBr) 3405 (OH), 2850 (OMe), 1650 (chelated C=O), 1520 and 1420cm⁻¹ (aromatic) which was identified as 5,7-dihydroxy-4'-methoxy flavonol-3-O-β-D-galactopyranoside by UV, IR, NMR spectra and direct comparison with an authentic sample (Singh⁸, 1981) and co-chromatography with an authentic sample.

Position and nature of Sugar linkage

It has already been shown that the glycoside was composed with kaempferide and D-galactose. The point of attachment of sugar moiety (galactose) to the aglycone was deduced from UV spectral shift studies with MeOH diagnostic reagent. On comparing UV data of the aglycone $\lambda_{\max}$ (MeOH) 267, 367 nm with that of the glycoside $\lambda_{\max}$ (MeOH) 268, 355nm, it was found hypsochromic shift of 12nm in band-1 (from 367 to 355 nm) in MeOH indicating the glycosidation at C₃. The glycoside was methylated with (CH₃)₂SO₄/K₂CO₃ in dry acetone and the methylated product on hydrolysis with 4N-H₂SO₄ yielded a compound C₁₈H₁₆O₆ m.p 1520, identified as 5,7,4'-tri-methoxy flavonol (kaempferil, 5,7,4'-tri-O-methylether) by mmp and co-chromatography

with an authentic sample. This also confirmed the glycosidic linkage through C₃ of the aglycone. The glycoside consumed two moles of periodate per mole of glycoside and liberated one mole of formic acid showing that the sugar is monosaccharide in pyranose form. Hydrolysis of the glycoside with emulsion enzyme solution indicated the β-linkage between the sugar and the aglycone.

From the above discussion, the structure of the compound (I) can be assigned as :



(5,7-dihydroxy-4'-methoxy flavonol-3-O-β-D-galactopyranoside)

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