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A new chromone from *Cassia nodosa*

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A new chromone named as 5,4'-dihydroxy-7-methyl-3-benzyl chromone (**1**) along with three known flavonoid compounds as unsubstituted flavone, kaempferol-3-o-rhamnoside and quercetin-3-o-arabino- side have been isolated from the leaves of *Cassia nodosa*. Their structures have been established by means of chemical and spectral evidences (IR, UV, ¹H-NMR, ¹³C-NMR and mass spectra).

Keywords: *Cassia nodosa*; Leguminosae; Chromone; 5,4(-dihydroxy-7-methyl-3-benzyl chromone; Unsubstituted flavone; Flavonoid glycosides

1. Introduction

Chemical investigations on plants belonging to Leguminosae have indicated that flavonoids are their important components [1–3]. Plants of *Cassia* genus have been used as remedies in traditional systems of medicines. *Cassia nodosa* is used as a folk medicine for the treatment of cheloid tumour, ring-worm, insect bite and rheumatism [4–5]. Earlier work includes the isolation and characterization of 2,3-dihydrokaempferol-3-o-rhamnoside, quercetin-3-o-rhamnoside, kaempferol-3-o-rhamnoside, nodosin, 8-c-glucosyl genistein and dalpanitin [6–7] from the flowers of *Cassia nodosa*. Now we are reporting the isolation and characterization of a new chromone (**1**) along with three known flavonoid compounds, the unsubstituted flavone, kaempferol-3-o-rhamnoside and quercetin-3-o-arabino- side from the leaves of *Cassia nodosa*.

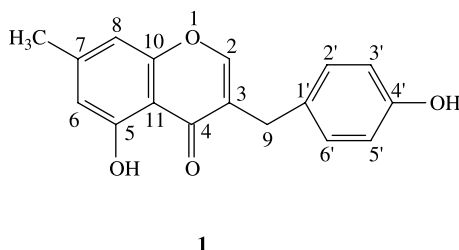
2. Results and discussion

The air-dried and powdered leaves (2.5 kg) of *Cassia nodosa* after being defatted with light petroleum (60–80°C) were exhaustively extracted with methanol. The methanolic extract was concentrated under reduced pressure, to yield a reddish brown gummy mass, which responded positively to colour tests of flavonoids [8–9]. It was chromatographed over silica gel column. Elution of the column with different solvents gave various fractions which upon crystallization afforded four TLC homogenous compounds marked as compounds **1**, **2**, **3**, **4**. Compound **1** was found to be a novel flavone and is characterized as 5,4'-dihydroxy-7-methyl-3-benzyl chromone (**1**) as discussed below, while compounds **2**, **3**, **4** were identified as unsubstituted flavone [10], kaempferol-3-o-rhamnoside [11] and quercetin-3-o-arabino- side [12].

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Compound **1** was obtained by eluting the column with benzene-ethylacetate (8:1). It was crystallized with chloroform-methanol as pale yellow granular crystals (35 mg) mp 290–92°C. It gave a greenish brown colour with alcoholic FeCl₃, indicating the presence of free phenolic hydroxyl group. It responded negatively to Shinoda's test [9], which supported the absence of the flavone nucleus. Elemental analysis along with the molecular ion peak at *m/z* 282 agreed with the molecular formula of C₁₇H₁₄O₄. The characteristic IR spectrum showed a broad band at 3100–3300 cm⁻¹ corresponded to chelated OH group and a carbonyl group appeared at 1655 cm⁻¹. The UV spectrum exhibited absorption maxima at 235 (4.21) and 340 nm (4.50) corresponding to homoisoflavone structure [13]. Analysis with shift reagents [14] gave a bathochromic shift of 30 nm in band II pointing out the presence of a hydroxyl group at the 5-position.

¹H-NMR spectrum (Table 1) of compound **1** showed a singlet of three protons at δ 2.27 assigned to the methyl group, while a two proton singlet at δ 3.16 assigned to the benzyl methylene group. A pair of meta-coupled doublets indicative of one proton each at δ 6.20 (*J* = 2.0 Hz) and δ 6.45 (*J* = 2.0 Hz) were attributed to H-6 and H-8, respectively. Another pair of ortho-coupled doublets indicative of two protons each at δ 6.94 (*J* = 9.0 Hz) and δ 7.76 (*J* = 9.0 Hz)

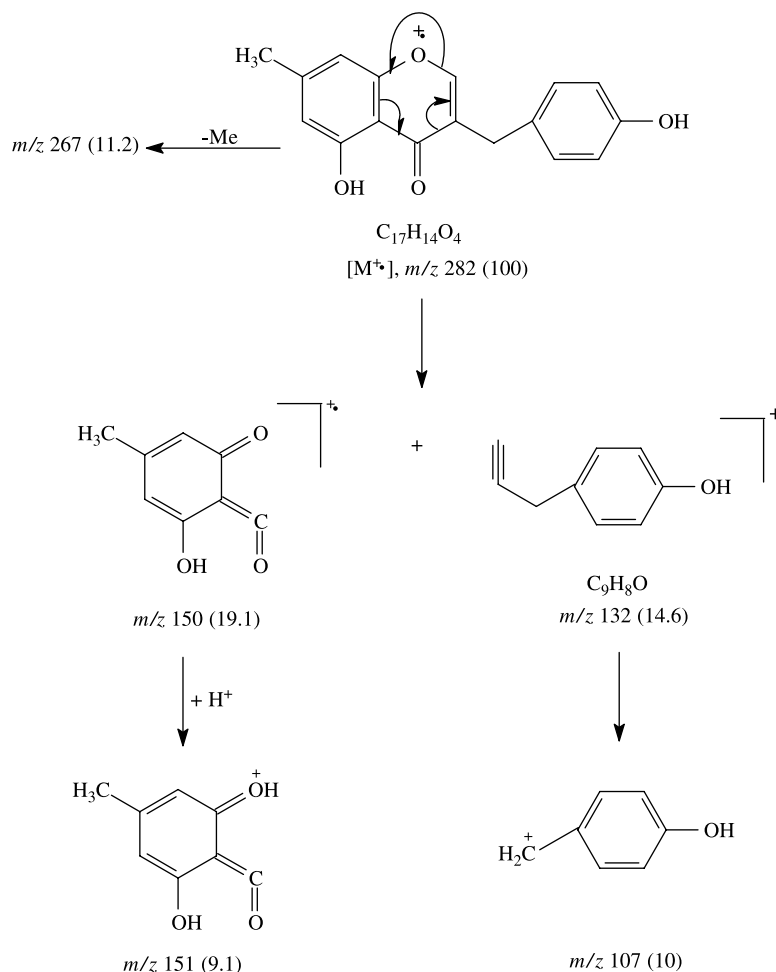


were ascribed to H-3',5' and H-2',6', respectively. The olefinic proton appearing at δ 8.02 was assigned to H-2 proton. The assignments of corresponding carbons according to ¹³C-NMR spectrum are given in Table 1. The above assignments were further confirmed by mass spectrum, which showed the molecular ion peak at *m/z* 282. The fragment ions are rationalized in Scheme 1.

Table 1. ¹H-NMR and ¹³C-NMR spectral data of compound 1.

Assignments	δH (ppm)	Assignments	δC (ppm)
CH ₃	2.27 (3H, s)	CH ₃	18.16
Ar-CH ₂	3.16 (2H, s)	Ar-CH ₂ (C-9)	29.0
H-6	6.20 (1H, d, <i>J</i> = 2.0 Hz)	C-6	98.01
H-8	6.45 (1H, d, <i>J</i> = 2.0 Hz)	C-8	93.02
H-3',5'	6.94 (2H, d, <i>J</i> = 9.0 Hz)	C-3',5'	115.21
H-2',6'	7.76 (2H, d, <i>J</i> = 9.0 Hz)	C-2',6'	128.96
H-2	8.02 (1H, s)	C-1'	131.5
OH-4'	10.13 (1H, s)	C-4'	159.30
OH-5	12.46 (1H, s)	C-2	151.20
		C-3	121.06
		C-4	180.0
		C-5	156.10
		C-7	104.2
		C-8	93.02
		C-10	162.01
		C-11	117.0

Spectrum recorded in DMSO-d₆ at 300 MHz, using TMS as internal standard.



Scheme 1.

On the basis of above evidences, compound **1** was identified as 5,4'-dihydroxy-7-methyl-3-benzyl chromone (**1**).

3. Experimental

3.1 General experimental procedure

Melting points were recorded on a Koflerblock and are uncorrected. IR spectra were taken on a Shimadzu IR-408 Perkin Elmer 1800 (FTIR). The MS and 1H -NMR spectra were obtained from a different institution. MS spectra were mostly measured in E I mode at 70 eV with a JEOL D-300; while the NMR spectra were usually recorded on a JEOL 4H-100 MHz, Bruker dpx 200 MHz and DRx 300 MHz in DMSO- d_6 using TMS as internal standard. Silica gel G (Merk, 60–120 mesh) was used for column chromatography. Pre-coated silica gel plates (Merk) were used for analytical TLC.

3.2 Plant material

The leaves of *Cassia nodosa* were procured from Botanical Garden of A.M.U., Aligarh, India and were identified by taxonomist Prof. Wazahat Hussain, Department of Botany, A.M.U., Aligarh, India. Its voucher specimen is deposited in the same department.

3.3 Extraction and isolation

Air-dried and powdered leaves of *Cassia nodosa* (2.5 kg) were defatted with light petroleum (60–80°C) and then extracted thoroughly thrice with methanol, which upon concentration under reduced pressure gave a reddish brown gummy residue. Usual colour tests were given for flavonoids. TLC examination in different solvent systems [benzene-pyridine-formic acid (36:9:5) and toluene-ethyl formate-formic acid (5:4:1)] showed it to be a complex mixture. Therefore, it was chromatographed over silica gel column. The column was eluted successively with light petroleum ether, light petroleum ether-benzene mixtures (9:1–1:1), benzene, benzene-ethylacetate mixtures (9:1–7:3), ethyl acetate, ethyl acetate-methanol mixture (9:1–1:1) and methanol, respectively. Fractions showing similar behaviour on TLC examination and the same IR spectra were combined. Repeated column chromatography of the fractions followed by fractional crystallization afforded four TLC homogeneous compounds, labelled as compounds **1**, **2**, **3**, **4**.

3.3.1 Compound 1. Compound **1** was obtained by the elution of column with benzene-ethylacetate (8:2). It was crystallized from chloroform-methanol as pale yellow granular crystals (35 mg), mp 290–292°C; it gave a greenish brown colour with alcoholic FeCl₃. Elemental analysis for C₁₇H₁₄O₄: found C, 72.31; H, 4.92%; calcd for C, 72.34; H, 4.96%. IR (KBr) ν_{\max} (cm⁻¹): 3100–3300 (OH), 1655 (C = O). UV (MeOH) λ_{\max} (nm, log ϵ): 235 (4.21), 285 (3.96), 340 (4.50, inf); (+ NaOAc) 235, 284, 338; (+ AlCl₃) 265, 306, 355 (inf.); (+ AlCl₃/HCl) 264, 306, 355; (+ NaOMe) 236, 286, 340. ¹H-NMR (300 MHz, DMSO-d₆) and ¹³C-NMR (300 MHz, DMSO-d₆) (see Table 1).

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