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Research Article

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Acylated Flavone O-Glycoside from *Sida rhombifolia*

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Abstract A new acylated flavone O-glycoside has been isolated from the methanol fraction of the root part of the *Sida rhombifolia*. The structure of the new compound has been established as, 4',6-dihydroxy-7-methoxy flavone-3-O-α-(6'''-acetyl glucosyl-β-1,4-rhamnopyranose) based on the spectral (UV, IR, ^{1}H, ^{13}C NMR and Mass) data.

Keywords Acylated Flavone, O-Glycoside, *Sida rhombifolia*

Introduction

Sida rhombifolia is the member of the genus sida belonging to the family malvaceae. Local names are called bala, mahabala (India), guri, sida guri, sali-guri (Sumatra) sada guri, otok-otok, taghuri (Java), Kahindu, mistaken (Musatenggara) and hutugamo, bitumudigo, sosapu (maluku) [1].

The plant is a small, erect woody, under shrub about 1.5 meters high with rough branches and stellate hairs. Leaves are very variable in shape up to 5 mm by 18 mm, short petioled, rhomboid-lanceolate, serrated towards the tops entire towards the base. The flowers are yellow coloured while seed are black and smooths [2].

The roots, leaves, fruits, stem, flower of *Sida rhombifolia* is used in traditional medicine against chronic diseases like skin diseases, sore, stomach disorder, digestion problem, malaria, diarrhea, dysentery, gastric, diabetes, chicken pox, blood cleaning [3, 4], headache migraine, eye problem, fever, gum infection, swelling [5] ophthalmia, swelling, cuts and wounds [6-8].

In the previous studies have reported phytochemicals isolated from this species, including ecdysteroids and their glycosides [9-10], Daucosterol [11], alkoloid [12], steroid and n-alkane [13].

Base on the above study, the aim of this research work is to isolate, characterise, the acylated flavone glycoside from the aerial part of extract of *Sida rhombifolia* in its eluting solvent.

Results and Discussion

The compound was obtained as yellow amorphous powder mp 175-178 °C based on the FAMBS ion peak at m/z 651 $[M+H]^{+}$, the molecular formula of compound is $C_{30}H_{34}O_{16}$ was determined, The UV spectrum exhibited absorption maximum at 248 and 305 nm, 338nm, which is typical of flavone. The molecular formula was corroborated by the ^{13}C NMR spectrum, which showed signals for all the 30 carbons of the molecule.

It gave positive colour reaction with aqueous NaOH (yellow) conc. H_2SO_4 (yellow to orange) and Mg-HCl solution (yellow to red) for flavone [14].

The IR spectrum of compound displayed absorption at 3430 (OH) and 1662 (CO, α, β-unsaturated) cm^{-1}.

The substitution pattern of ring A and B was deduced by ^{1}H NMR spectroscopy, thus the integration of the signals in a complex multiplet in the 3.20-4.40 ppm region and the number of glucosyl peaks indicated that the compound was a diglycoside.

An extra singlet signals for three protons at δ 2.04ppm was observed suggesting that one of the sugar hydroxyl was acylated.

Acetylatron of compound gave the acetate, which showed signals in ^{1}H NMR for six aliphatic and two aromatic acetyl groups. Acid hydrolysis of the compound yielded glucose, rhamnose and aglycone.

The anomeric proton (H-1''') of the glucose appeared as a doublet at 4.28 (J=8Hz) this chemical shift confirmed that glucose is not attached to the aglycone nucleus The diaxial coupling (J=8Hz) between H-1''' glucose and H-2''' glucose indicated that the glucose has a β-configuration.

The signals at 5.52 (J=2Hz) was assigned to H-1'' of rhamrose, confirming the position of linkage between the sugar and aglycone at C-3, and the diequitorial compiling (J=2Hz) between H-1'' rhamrose and H-2'' rhamnose indicated the α-configuration [15].

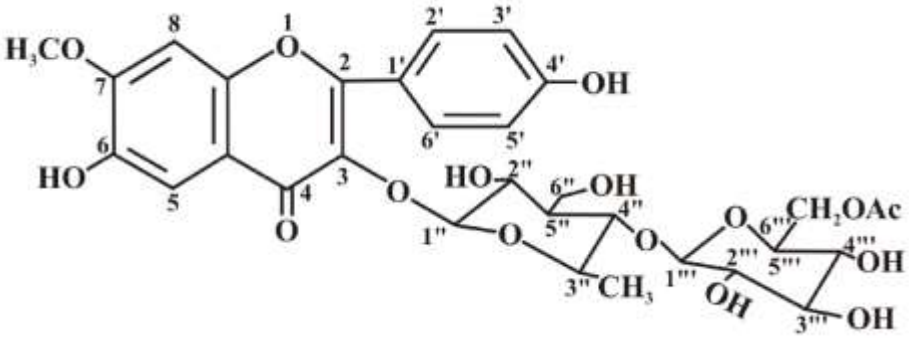
The position of the linkage between aglycone and the other moiety, occur at C-3. It is noteworthy that the λ_{max} of band I at 316nm shows a low value, a typical compound C-3 linked glycoside shows a corresponding λ_{max} located about 350-360 nm which appears only as shoulder in the ethanol UV spectrum of compound This Hypsochromic shift bond I is due to the presence of the acetyl group in the side chain.

The ^{13}C NMR spectrum of the sugar moiety of the compound shows a down field shift of C-6 glucose (Δ+2.1) from the chemical shift values reported for the corresponding carbon resonance of unlinked C-6 glucose (such as flavonol-3-0-glucophyronoside) [16] these shifts are expected from the substituent effect of C-6 glucose acylation [17] otherwise, in the ^{13}CNMR spectrum of Kaempferol-3-O-(rhamno(1-6) glucoside) [18] the glucose C-6'' signals shift downfield from δ 61.0 to 67.1 due to rhamnosilation at C-6'', this evidence exclude other possible acylation site in the glucose moiety of compound and & fixed the acylgroup to C-6 glucose, The chemical shift for C-4 rahmnose in compound resonate at δ 82.6 indicating that glucose unit must be linked the 4-OH group of the rhamnose moiety.

In ^{1}H NMR spectra of the compound contained signals for the para coupled protons at δ 6.80 and 6.70 (IH, each, s) due to H-5 and H-8 respectively [19] and compound showed the signal at δ 3.68 (3H, s) relative to one methoxyl group, this was also supported by ^{13}C NMR, which showed the signal at δ 57.00 respectively.

Furthermore, four aromatic proton signals appeared as an A_2B_2 type (AA'BB') at δ 7.20 and 8.12 (J=9.0 Hz) and were assigned to the B ring protons of a 4'-substituted flavone.

On the basis of these data, we can conclude that the structure of the compound is 4', 6-dihydroxy-7-methoxy flavone–3-O-α-(6'''-Acetylglucosyl-β-1,4-rhamnoside)



Experimental

Apparatus

The UV and IR spectra were recorded on a shimadzu UV-Visible spectrophotometer Model UV1601 PC and shimadzu FTIR Model 8400 Respectively. The ^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker BPX-200 Spectrometer Operating at 400 MHz for ^{1}H NMR and 100 MHz for ^{13}C NMR Spectra. The FABMS were obtained on a varian MAT CH5 instrument. Column Chromatography was performed on silicagel (200-400) mesh TLC was carried out on silicagel (merk, 10-40 μ) p recoated plat paper chromatography (PC) was done on Whatmann No. 1 filter pepar. And the sports were visualized by spraying with 7% H_2SO_4.

Plant Material

The leaves of *S. rhombifolia* were collected from NRIPT, Telear Gaj, Proyagraj, India and identified by Dr. B.K. Shukla, Taxonomist, Botanical survey of India (BSI) Prayagraj. It is widely distributed through India and Nepal, specially in most region ascending to an altitude of 1800 cm in the Himalayas

Extraction and Isolation

The dried and powdered root part of *S. rhombifohia* (4 kg) were extracted in the MeOH (5L) under reflux (x4), The combined extract were concentrated and the residue (50g) suspended in H_2O and extracted in the n-BuOH, the organic layer was chromatographed on silica gel eluted in the a (C_6H_6:MeOH solvent system. A fraction (C_6H_6: MeOH, 8:2 V/V) containing flavone glycoside was rechromatographed on silica gel using $CHCl_3$: MeOH: H_2O, 10:8:2 as eluent and crystalize to give 4',6-dihydroxy-7-methoxy flavone–3-O-α-(6'''-Acetylglucosyl-β-1,4-rhamnoside)

UV λ_{max}^{MeOH} nm	:	248, 305, 338
IR υ_{max}^{KBr} cm^{-1}	:	3440, 2930, 1640, 1250, 1185
^{1}H NMR $CdCl_3$) 400 MHz δ ppm	:	3.92 (3H, s, 7-OCH_3) 12.56 (1H, s, 6-OH) 8.57 (1H, s, 4'-OH) 6.80 (1H, s, H-5) 6.70 (1H, s, H-8) 7.20 (2H,d, J=9.0Hz, H-3', H5') 8.12 (2H, d, J = 9.0 Hz, H-2', H-6') 0.91 (3H, d, J = 6.0 Hz, me-rhamonse) 3.03-4.15 (m sugar proton) 4.28 (1H, d, J=8.0Hz, H-1 glu) 5.52 (1H, d, J = 2.0H_z, H-1 rham) 2.04 (3H, s, acetyl)
^{13}C NMR ($CdCl_3$) 100 MHz δ ppm	:	180[2](C-4, s) 129.2 (C-5, d) 143.8 (C-6, s) 147.2 (C-7, s) 130.2 (C-8, d) 57.82 (q, -OCH_3) 20.9 (C-6'''-Acetyl) 164.0 (C-2, s) 102.8 (C-3, s) 144 (C-9, s) 105.5 (C-10, s) 18.3 (C-6rha) 63.8 (C-6 glu) 70.5 (C-2 rha, C-5 rha) 71.11 (C-4 glu) 72.7 (C-3 rha) 74.7 (C-2 glu) C-5 glu) 76.9 (C-3 glu), 82.4 (C-4 rha) 99.6 (C-1 rha) 101.6 (C-1 glu) 114.8 (C-2') 116.5 (C-5') 121.5 (C-1') 121.8 (C-6') 125.9 (C-1).
FABMS;		m/z 651[M+H],$^+$ 489 [m-162+H] 479 [M-180+H] 309 [m-162+180+H^+]

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