The Pharmaceutical and Chemical Journal, 2016, 3(3):158-160

Available online www.tpcj.org

Research Article

ISSN: 2349-7092
CODEN(USA): PCJHBA

A New Flavone O-Glycoside from *Sida rhombifolia*

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Abstract A new Flavone O-Glycoside has been isolated from chloroform –methanol fraction of the aerial part of the *Sida rhombifolia*. The structure of the new compound were established as *3',5,7-trihydroxy-4'-methoxy flavone-3-O-β-D-glucosyl(1''→3'')-β-D-glucopyranoside* on the basis of spectral data using UV, IR, ^{1}H NMR, ^{13}C NMR and Mass.

Keywords *Sida rhombifolia*, flavones O-glycoside

Introduction

Sida rhambifolia belong to the family malvaceae, commonly known as mahabala, found in marshy [1] place throughout India. It is use to cure fever, heart disease, burning sensation, piles, urinary disorder and all kind of inflammation.

The plant growth leads to the production of different chemical compound are called photochemicals like phenols, terpenoids, flavonoids steroids and phytosteroids [2]. It is well know that these compound have medicinal application and biological activity like antiplasmotic, antimalerial, anti-inflammatory, antimicrobial [3].

The isolated steroids are known to have cardiac impact, and to have insecticidal and antibacterial properties [4]

Due to their biological activity, they are commonly used in drugs. As a result of research, tannin have antibacterial action, antitumor's and antivirus [5]. On the basis of above finding of the plant, the aim of study was to extract, isolate, identify and characterize the isolated compound having melting point is 150 $^{\circ}$C.

Results and Discussion

Compound gave m/z 637 $[M+H]^{+}$ in the FABMS spectrum (positive mode) $C_{28}H_{32}O_{17}$ and responded to shinoda (mg-HCl) and the molish test and this molecular formula is confirmed by the ^{13}C-NMR spectrum which showed signals for all the 28 carbons of the compound. It gave a positive $FeCl_{l3}$ test suggesting the presence of a chelated hydroxyl group and its solubility in alkali indicated its phenolic behaviour.

The UV spectrum, exhibited absorption maxima at 272, 305, and 338 nm were typical of flavones in the 5, 7, 3', 4',-Tetra oxygenation. A free C5-hydroxyl were confirmed by a batho chromic shift of 42 nm in the presence of $AlCl_{l3}$ and a free 7-hydroxyl group were indicated by a batho chromic shift of 11nm upon the addition of NaOAc [6-7].

The IR spectron showed a chelated hydroxyl group at 3420 cm^{-1} and a chelated carbonyl group at 1624 cm^{-1}.

The substitution pattern of ring A and B was deduced also by ^{1}H NMR spectroscopy. Thus methoxyl group at 3.89 was assigned to C-4' and integration of signal in a complex multiplet in the 3.20-4.40 ppm region and the number of glucosyl peaks indicated the compound was a diglycoside.

The ^{1}H NMR spectrum of compound showed signals for two meta coupled aromatic protons at δ 6.35 (1H, d, J= 1.9Hz, H-6) and δ 6.63 (1H, d, J=1.9 Hz, H-8) these were suggested that A ring is disubstituted.

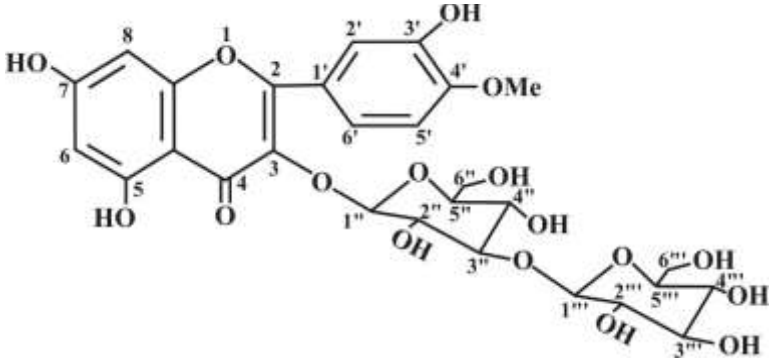
Ring-B showed a pattern of three one-proton signals at δ 7.50 (1H, d, J=1.2Hz, H-2') δ 6.88 (1H, d, J=7.2 Hz, H-6') and δ 7.42 (1H, dd, J=1.2, 7.2 Hz, H-5'), the multiplicity of which showed one proton coupled to the remaining two which were, in turn, not coupled to each other. The size of the coupling constants (1.2 and 7.2Hz) is characteristics of meta and ortho coupling as found in a 3', 4'-oxygenated flavonoid [8].

In addition, signals in the ^{13}C-NMR spectrum at δ 149.30 and 151.73 indicated that the oxygenated carbons are adjacent due to shilding effect (>10) each oxygen exert on its neighbouring ortho carbons [9]. ^{13}C-NMR of aglycon (free from sugar) showed a 3ppm downfield sift (δ 138.21) for carbon C_3 than that (135.21) of in the glycoside itself suggesting O-glycosidation at C_3 [10].

The presence of two anomeric signals in the 'H-NMR spectrum (δ 0 4.88, d, J=7.6 Hz, δ 4.60, d, J=7.6 Hz) and ^{13}C NMR (104.38, 105.14) spectra suggested compound to be disaccharide derivative. The diaxial coupling (J-= 7.6Hz) between H-1" to H-2" and H-1"' to H-2"' of glucose indicated that the glucose has a β-configuration.

Therefore it was found that both sugar were β-D-glucose in pyranose form [11] Carbon-13 chemical shift for the sugar, i.e. glucose were also similar to their reported resonance value. This down field shift showed that they are linked to each other at C-3.

Based on the above observation, the structure of the compound were established as *3',5,7-trihydroxy-4'-methoxy flavone 3-O-β-D-glucosyl- (1"'→3") –β-D-glucopyranoside*.



Experimental Section

Apparatus: M.P. was measured in an open capillary tube and it is uncorrected UV were recorded on a Beckman's DK_2 spectrometer. IR was recorded in KBr on a Perkin-Elmer Spectrometer. 1HNMR of compound were recorded at 300 MHz. $^{13}CNMR$ Sectra at 100 MHz in $CdCl_3$ using TMS as an internal reference a jeol JNM-A500 spectrometer. Mass spectrum was recorded on a jeoLMSD 300 mass spectrometer. TLC were performed on a coated silica gel 60F254 (merck) and the spot were visualised by exposure to iodine vapour or spraying with 5% H_2SO_4 in methanol followed by heating the plate at 110^0C for 5 minutes.

Plant Material

The stem bark of *S. rhombifolia* was collected from Northern Regional Institute of Printing Technology (NRIPT), Teliargaj, Prayagraj, and the plant was identified by Dr. B.K. Shukla, Taxonomist, Botanical survey of India (BSI) Prayagraj. It is widely distributed through India and Nepal, especially in most region ascending to an altitude of 1800cm in the Himalayas.

Extraction and Isolation

The shed dried well ground stem bark of *S. rhombifolia* (4kg) were refluxed with (90%) ethanol and the extract were concentrated under reduced pressure through rotatory evaporator. It was partition between DCM: Ethylacetate. The chloroform soluble fractions (50g) were chrometographed over column of silica gel and eluted with binary solution of n-Hexane: Chloroform (9:1) in sequence of increasing polarity; The compound was isolated chloroform:

methanol (7:2) fraction as pale-yellow solid (20mg) 3',5,7-*trihydroxy-4'methoxy flavone-3-O-B-D-glucosyl-(1'''→3'')-B-D-glucopyranaside*.

UV λ_{max}^{MeOH} nm	:	272, 305, 338
IR λ_{max}^{KBr} cm^{-1}	:	3420, 1624
$^{1}HNMR_{300mHz}^{CDCl_3}$ δ ppm	:	3.89 (1H, s, -OCH_3, C-4') 3.20-4.40(14H, m, glu) 7.50 (1H, d, J=1.2Hz, H-2') 6.88 (1H, d, J=7.2Hz, H-6') 7.42(1H, dd, J-=1.2 ,7.2Hz, H-5') 13.02 (1H, s,5-OH) 10.56 (1H, s, 7-OH) 8.97 (1H, s,3'-OH) 7.69 (1H, d,H-6) 7.06 (1H, d,H-8)4.88 (1H,d, J=7.6 Hz, H-1'') 4.60(1H, d, J=7.6 Hz,H-1''')
$^{13}CNMR_{100mHz}^{CDCl_3}$ δ ppm	:	176.0 (s, C-4) 163.6 (s, C-7) 159.6 (s,C-5) 155.0 (s,C-9) 147.7 (C-3',C-5', s, 146.2 (s, C-2)138.2 (s,C-4') 135.8(s,C-3) 120.7 (s, C-1') 105(s, C-6) 102.7 (s, C-10) 106 (d, C-2', C-6') 93.3 (d, C-8) 81.4 (d, C-5'') 80.8 (d, C-3'') 73.1(d, C-1'') 70.5 (d,C-2'') 70.3 (d, C-4'') 63.3 (t, C-6) 56.2 (q, 3-Ome).
FABMS	:	637 $[M+H]^+$, 474 [M–162+H], 313 [M–2x 162+H]

Acid hydrolysis

Compound is treated with 8% HCl (2ml) and MeOH (20ml) were reflexed for 2hr. The reaction mixture was refluxed in vacuo to dryness, dissolve in water (3ml) and neutralized with NaOH. The neutralized product were subjected to TLC analysis (EtOAC:MeOH : H_2O : HOAc, 6:2:1:1). The chromatograms were sprayed with Aniline hydrogen phthalate followed by heating at 100°C. The sugar was identified after comparison with authentic sample.

Acknowledgement

The Author thanks to UGC New Delhi India for providing teacher research fellowship (TRF) and also the Author wish express their gratitude to Dr. B.K. Shukla, Taxonomist, Botanical Survey of India (B.S.I.) Prayagraj (U.P.) India for Identification of the plant *S. rhombifolia*.

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