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Research Article

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Flavonol Glycoside from *Sida rhombifolia*

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Abstract A novel flavonol glycoside has been isolated from the ethyl acetate fraction of the stem bark of the *Sida rhombifolia*. The structure of the new compound has been established as 5, 7-dihydroxy-4'-methoxy flavonol-*3-O-β-D*-glucopyranoside based on the spectral data using UV, IR, ^{1}H-NMR, ^{13}C-NMR and Mass.

Keywords *Sida rhombifolia*, Flavonol Glycoside

Introduction

Sida guri or *Sida rhombifolia* Linn. belong to the Malvaceae Family locally known as Bala, Mahabala (India), Lal Barela Atibala (Bangladesh), Huang HuaMu (China) chitta madi (Srilanka), Escobilla (Panama), Petoria-bossie (Africa), it is short–lived perennial sub shrub commonly growing to 60cm, but sometimes reaching 1.5m in height, distributed through Bangladesh [1] it also distributed throught the tropical, subtropical, warm temperate region [2-3].

Whole part of *Sida rhombifolia* were used as traditional medicine in India as diuretic, diaphoretic, demulcent, emollient, stomachic, tonic, sudorific, appetite and stimulant. Leaves and roots are used for piles, gonorrhea, ant soud, the roots of these herbs are of great importance in the treatment of rheumatism [4] the stems are employed as demulcent and emollient both for external and internal use. Mucilage is used as an emollient and for scorpion sting. An Australion aborigine use the herb to treat diarrhea, leaves are used as smoke in Mexico and a tea is prepared in India for the stimulation it provides [5].

Sida rhombifolia have significant activity such as antibacterial [6] anti arthritic [7] antiinflamatory [8] antidiarrheal [9] antidiabetic [10] wound healing [11] hypotensine [12] nephrotoxicity [13] antimalerial [14] antioxidant [15] hepatoprototective [16] Larvicidal.

In the former investigation, the presence of some acdysteroid and their glycoside in *S. rhombifolia* extract has been reported. They are of four type, ecdysone, 20-hydroxy ecdysone, 2-deoxy-20-hydroxy-*3-0-β-D*-glucopyranoside, 20-hydroxy ecdysone-*3-0-β-D*-glucopyranoside. These are reported for the first time. Sterol (β-Sitosterol, stigmasterol, compesterol, spinastrol, cholesterol.n-alkane (nonacosane, hentricontane) an n-alcohol were also identified from the whole air dried and aerial part of *S.rhombifolia*. β-phenethylamine, ψ-ephedrine, quinazoline like vasicine, vasicinol, vasicinone, carboxylated tryptamine such as S-(+)-N-methyl tryptophan methyl ester, choline and betain were isolated from aerial part [17] however no were found to examine the chemical compound of stem bark extract of *S. rhombifolia*.

Based on the above finding, the purpose of my research is to isolate, identify, characterize and elucidate the active constituent of stem Bark extract of *S. rhombifolia* in its polar and non polar fraction.

Result and Discussion

The molecular formula of compound was deduced as $C_{22}H_{16}O_{11}$ from ^{13}C-NMR and FABMS and their molecular ion M^+ at m/z 456. It responds to the molish and shinoda [mg-HCl] test. It gave a positive ferric chloride test suggesting the presence of chelated hydroxyl group and its solubility in alkali indicated its phenolic nature [18].

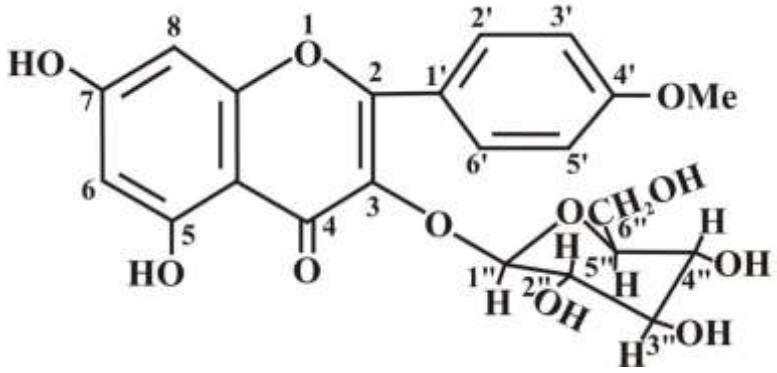
It the UV spectrum of compound the absorption maximum at 265 nm suffers bath chromic shift of 12 and 14 nm with Aluminium chloride and sodium acetate respectively, indicating the presence of free hydroxyls at 5 and 7-position [19].

The IR spectrum showed a chelated hydroxyl group at 3418 cm^{-1} and a chelated carbonyl group at 1624 cm^{-1}. The UV spectrum in methanol showed no bathochromic absorption in band I and II, this fails to examine hydroxyl group at C-3 of the flavonoid skeleton [20].

In ^{1}H NMR spectrum H-6 doublet at δ 6.89 (J= 2Hz), an H-8 doublet at δ 7.1 (J=2Hz) metacoupled, and an orthocoupled system (A_2B_2) (B-ring protons) at δ 7.95 (J=10Hz) and δ 7.22 (J=10Hz)[21] were assigned to the B-ring proton of 4'- substituted flavonol. In addition the compound exhibited a bathochromic shift (50 nm) on addition of Aluminium chloride/Hydrochloric acid in their UV spectra, these results strongly suggested that compound were flavonoid glycoside with chelated hydroxyl group at C-5 [22].

A fragment ion at M/z 132 confirms the presence of a methoxyl group in ring B in the flavonol glycoside, compound gave positive color reaction for flavonol [23] it gave positive test for sugar [24]. It also produced copious foam on shaking with water suggesting it to be a glycoside. The acid hydrolysis of compound gave Aglycon and glucose. Compound showed one anomeric proton doublet at δ 5.48 in dicaling the presence of sugar moieties.

^{13}C-NMR of Aglycon showed a 3PPM downfield shift (δ: 138.21) for carbon C-3 than that of (δ 135.21) in the glycoside itself, suggesting –O-glycosidation. On the basis of above evidence, the compound is characterized as 5,7-dihydroxy-4'-methoxy flavonol-*3-O-β-D*-gluco-pyranoside.



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 400MHz for ^{1}H NMR and 100MHz 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) precoated plat, Paper chromatography (PC) was done on whatman no-1 filter pepar. And the sports were visunalized by spraying with 7% H_2SO_4.

Plant Material

The stem bark of *S. rhombifolia* were collected from NRIPT, Telear Ganj, Prayagraj, India and identified by Dr. B.K. Shukla, Taxanomist, Botanical Survey of India (BSI) Prayagraj. It is widely distributed through India and Nepal, specially in moist region ascending to an altitude of 1800cm in the Himalayas.

Extraction and Isolation

The stem bark was cut in to small pieces dried at room temperature and ground in to powder, the dried powder (5Kg) of *S. rhombifolioa* was extracted in the 95% ethanol (3x24h). On removal of the solvent under reduced pressure at the temp <40 °C the C_2H_5OH extract gave a brown mass (200g). The ethanol extract was triturated with n-hexane (3x500 ml) each time for 1h at room temp. The n-hexane soluble fraction was collected by filtration and the residue was treated twice more with n-hexane in a similar fashion, the combined n-hexane triturate was evaporated to dryness when a solid mass (4.9g) was obtained which do not show any good

resolution on TLC. The left over residue from n-hexane triturating mass (20g) was adsorbed on silica gel and place over a column of silica gel a and eluted with n-hexane: EtOAC (9:1) and divided into different fraction. The fraction of same nature were mix. On repeated fractional crystallization from C_6H_6: EtOAC (9:1) one pure compound (125mg) was isolated.

5,7-dihydroxy-4'- methoxy-*3-O-β-D*-glucopyranoside

UV λ_{max}^{EtOH} nm	:	360, 316, 300, 268, 258 nm
IR υ_{max}^{KBr} cm^{-1}	:	3418, 2930, 1624
^{1}NMR	:	(400MHz) δ 6.8 (1H, d, J=2.0Hz, H-6) δ 7.1 (1H, d j=2.0Hz, H-8.0) δ 3.9, (3H, s, -O CH_3) δ 6.88 (1H,d J= 8.4Hz , H-5) δ 7.25 (1H,dd, J=2.0, 8.6 Hz, H-6) δ 7.36 (1H, d, J= 2H z H-2) δ 6.70 (1H, d ,J= 8.6, Hz H-3) δ 5.48 (1H, d, H-1 glu) δ 3.57 (1H, m, H-2) δ 3.57 (1H, m,H-3) δ 3.68 (1H , m, H-4) δ 34.8 (1H , m, H-5) δ 3.56 (2H, m, H-6)
^{13}C-NMR (100MHz)	:	δ 63.8 (C-6glu) 71.1 (C-4glu) 74.7 (C-2 glu) δ 76.9 (C-3 glu) 94.5 (C-8) 101.6 (C-1glu) 114.8 (C-2) 116.5 (C-5 glu) 121.5 (C-1) 121.8 (C-6) 135(C -3) 145 (C-3') 146.1 (C-4') 149.5 (C-7) 157.3 (C -1') 157.5 (C-2') 160.6 (C-4) 178.6 (C-5') 165 (C-5) 170.8 (C-6')
FABMS	:	M+, 456, 293, 132

Acid hydrolysis

Compound (7mg) was refluxed with 2N HCl in aqueous MeOH (4ml) for 6h. The reaction mixture was then concentrated under reduced pressure to remove methanol, this was then diluted with H_2O (4ml) and the aqueous layer was adjusted to pH 7 with Ag_2CO_3 and filtered. The supernatant liquid was concentrated and compare with reference sugar on TLC and spot were visualized by spraying aniline hydrogen phthalate.

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