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A New Flavone C-Glycoside from *Sida rhombifolia*

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Abstract A new flavone C-glycoside has been isolated from the dichloromethylene-ethylacetate fraction of the stem bark of the *Sida rhombifolia*. The structure of the new compound has been established as 5,7,4'-trihydroxy-3'-methoxy flavone-6-C-β-D-glucopyranoside based on the spectral data using UV, IR, ^{1}HNMR, ^{13}C-NMR and mass.

Keywords *Sida rhombifolia* and flavone-C-glycoside

Introduction

Sida rhombifolia is commonly known as bala or atibala is a perenial or some times annual plant in the family Malvaceae. The species is found in tropics and subtropics region and usually confined to waste ground such as road side and rocky area. It is used in ayurvedic and Siddha medicine, where it is known as Kurumthotti.

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 upto 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 smooth [1].

The plant is claimed by folklore for treating various diseases like rheumatism, seminal weakness, and diarrhea [2] *Sida rhombifolia* is also much used for treating ulcers, inflamation, swelling, antinoceptive [3-5].

Sida rhombifolia have a variety of phytochemicals that has led to its many use in the area of medicine. The plant is used as demulcent, diaphoretic, diureties, emollient, stomachic, tonic, sudorific, appetite and stimulant. It has significant medicinal use for which it is cultivated throughout the world. Leaves and roots are use for piles, gonorrhea, diuretic, aphrodisic. Roots are held in great repute in the treatment of rheumatism. Stem abound in mucilage are employed as demulcent and emollient both for internal and external use. This herb is also useful in calculous troubles and as a ferbuge with pepper [6]. Australian aborigines use the herb to treat diarrhoea, leaves are smoked in Mexico and a tea is prepared in India for the stimulation it provides [7].

For instance, it is used for treating stings and bites of scorpion, snake and wasp, skin disease and sore, treat stomach disorder, stomach pain, digestion problem, malaria, flatulance, dysentery, irritable bowl syndrome, gastric, enteritis, hemorrhoids, chicken pox, blood cleaning, fatigue, [8-9] migraine, headache, conjucntivitis, toothaches, fever wounds, gum infections [10].

In the previous study, the β-phenethylamine, N-methyl-β-phenethylamine, S(+)-N-β-methyl tryptophan methyltryptophan methyl ester, vasicinol, vasicinone, vasicine, choline, hypaphorine methyl ester, hypaphorine, and betain have been isolated from the *Sida rhombifolia* [11]. The plant showed antipathogenic activity like antiinflamatory, antipyretic activity [12], potent cytotoxicity [13], antimicrobial activity [14], antibacterial activity [15]. Therefore lack of studies examine the phytochemical of the stem bark extract of *Sida rhombifolia*.

Based on the above finding, the purpose of this study is to isolate, identify, characterise and evaluate the phytochemicals of stem bark of extract of *Sida rhombifolia* in its polar and nonpolar fraction.

Results and Discussion

Compound showed $[M+H]^+$ at m/z 463(100%) in its (+)- FABMS corresponding to the molecular formula $C_{22}H_{22}O_{11}$ ($[M]^+$ = 462). This was confirmed by the ^{13}C-NMR spectrum which showed signals for all the 22 carbons of molecule. The fragment at m/z 301 $[M-162+H]^+$ corresponds to the subsequent loss of hexose moiety from the compound. The UV spectrum, exhibited absorption maxima at 272, 305, and 338 nm, were typical of flavones in the 5,7,3',4'-tetraoxygenation [16-17] also the UV spectra of the compound (λ_{max} 271-273, 334-338nm) were characteristics of C-glycoside. A free C5 hydroxyl was confirmed by a bathochromic shift of 42 nm in the presence of $AlCl_3$ and a free 7-hydroxyl group was indicated by a bathochromic shift of 11 nm upon the addition of NaOAC.

Compound responded to the Molish [18] (Test for sugar) and Sinoda [19] [mg-HCl] test for flavone. It gave a positive ferric chloride test suggesting the presence of a chelated hydroxyl group and its solubility in alkali indicated its phenolic nature. The IR spectrum showed a chelated hydroxyl group at 3420 cm^{-1} and a chelated carboxyl group at 1625 cm^{-1}. It also produces copious foam on shaking with water suggesting it to be a glycoside. Compound fails to yield any sugar even on prolonged acid hydrolysis (5 hr) and this suggest that it is a C-glycosyl. Compound which is supported by the presence of two bands at 1010 and 1038 cm^{-1} in the IR spectrum [20]. Aquous $FeCl_3$ oxidation [21] of compound produces glucose. The formation of glycerol [22] when the Borohydride reduced product of periodate oxidised compound is subjected to acid hydrolysis shows the pyranose form of the glucosyl residue.

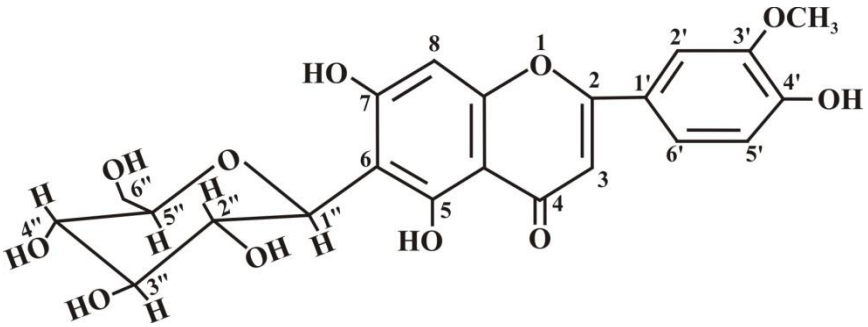
When compound is boiled with hydriodic acid in phenol, demethylation as well as decomposition of the sugar moiety occur and the resulting product is identify as orobol by authentic sample. The formation of orobol shows the presence of 3',4',5,7 tetraoxygenation pattern in compound. The 3',4'-oxygenation in the B-ring is further confirmed by the formation of veratric acid when the acetate of compound methyl ether is subjected to alkaline hydrogen peroxide oxidation.

Due to the presence of free hydroxyl at 5 and 7-position indicated by UV shift, the -OMe group is present only in the side phenyl ring either at 3' or 4'-position that it is at 3'-position is revealed by the formation of vanillic acid, when the hepta acetate of compound is oxidise by potassium permanganate.

Oxidative degradation leading to the formation of veratric acid and vanillic acid clearly shows that the side phenyl ring is devoid of the C-glycosyl residue. Hence either 6 or 8-position of the condensed benzene ring is involve in the C-glycosylation.

The anomeric protons in the ^{1}HNMR spectrum of the compound were observed at 4.59 (1 H, d, J = 9.9Hz, H-1") demonstrated the β-configuration of the glucosyl residue, long range correlations in the HMBC spectrum from 4.59 to C^{13} resonances at 110.0 (C-6) respectively the assignment of C-5 and C-6 were also confirmed by long range correlations from δ 13.56 (s, 5-OH) to these carbons. ^{1}H NMR spectrum of compound showed one proton singlet at δ 6.53 (1H, s, H-8) indicating that A ring were trisubstituted [23].

Three B-ring proton are shown up as a typical ABX splitting pattern with aromatic proton signal at δ 6.76 (d, J=1.6, Hz, H-3') 6.61 (dd, J=1.6, 8.0 Hz, H-5') and 6.68 (d, J=8.0 Hz, H-6') and at δ 3.99 (3H, s, $-OCH_3$) respectively on the basis of above data. The structure of the compound is Identified as 5,7,4'-trihydroxy 3'-methoxy flavone -6-C-β-D-glucopyranoside.



5,7,4'-trihydroxy 3'-methoxy flavone -6-C-β-D- glucopyranoside

Experimental Section

Apparatus: M.P. was measured in an open capillary tube and it is uncorrected UV were recorded on a Beckmans DK_2 spectrophotometer. IR were recorded in KBr on a Perkin-Elmer spectrometer. ^{1}HNMR of compound were recorded at 300 MHz. ^{13}CNMR spectra at 100 MHz in $CdCl_3$ using TMS as an internal reference an a Jeol JNM-A500 spectrometer. Mass spectrum were 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 110ºC for 5 minutes.

Plant Material

The stem bark of *S. rhombifolia* were collected from Northern Regional Institute of Printing Technology (NRIPT) Teliarganj, Prayagraj and the plant were identified by Dr. B.K. Shukla, Taxonomist, Botanical Survey of India (BSI) Prayagraj. It is widely distributed through India and Nepal specially in moist region ascending to an attitude of 1800 cm in the Himalyas.

Extraction and Isolation

The shed dried and well ground stem bark of *S. rhombifolia* (4 kg) 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 fraction (50g) were chromatographed over a column of silica gel and eluted with binary solution of DCM. Methanol, in sequence of increasing polarity. The compound were isolated (C_6H_6 : $CHCl_3$, 8 : 2 v/v) fraction as pale yellow solid (20 mg) 5,7,4'-trihydroxy-3'-methoxy flavone-6-C-β-D-glucopyranoside m.p. 215ºC.

UV λ_{max}^{MeOH} nm	:	272, 305, 338 nm
IR υ_{max}^{KBr} cm^{-1}	:	3420, 1625, 1038, 1010
^{1}HNMR ($CdCl_3$) 300 MHz δ ppm	:	13.02 (1H, s 5-OH), 10.56 (1H, s, 7-OH) 9.18 (1H, s, 4'-OH) 6.53 (1H, s, H-8) 7.05 (1H, s, H-3), 6.67 (1H, d, J = 1.6. Hz, H-3') 6.61 (1H, dd, J=1.6, 8.0Hz, H-5') 6.68 (1H, d, J = 8.0 Hz, H-6'), 3.99 (3H, s, 3'-OCH_3) 4.59 (1H, d, J=9.9Hz, H-1") 7.25 (1 H, d, H-2"), 3.57 (1 H, m, H-3") 3.68 (1 H, m, H-4") 3.48 (1H, m, H-5") 3.76 (2H, m, H-6")
^{13}C-NMR ($CdCl_3$) 100 MHz 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', 5') 146.2 (s, C-2) 138.2 (s, C-4') 135.8 (s, C-3) 120.7 (s, C-1') 108 (s, C-6) 102.7 (s, C-10) 106.0 (d, C-2', C-6') 93.3 (d, C-8) 81.4 (d, C-5") 8.8 (d, C-3") 73.1 (d, C-1") 70.5 (d, C-2") 70.3 (d, C-4") 61.3 (t, C-6") 56.2 (q, 3'-OMe)

Acid hydrolytic the compound (20 mg) in 7% aq. alcoholic H_2SO_4 (20ml) was heated at 100 ºC for 5hr cooled, extracted with EtOAc, the extract on evaporation yielded no aglycone, the aqueous portion was treated with excess of $BaCO_3$, the ppt filtered off and the filtrate concentrated. Examination of the concentrate by paper chromatography revealed the presence of unchanged starting material without the formation of any free sugar.

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