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Research Article

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Isolation and Characterisation of Coumarin Derivative from *Sida rhombifolia* Linn.

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Abstract A novel coumarin derivative has been isolated from the dichloromethylene fraction of the aerial part of the *Sida rhombifolia*. The structure of the isolated compound has been established as 7-hydroxy-6-methoxy coumarin based on the spectral (UV, IR, ^{1}HNMR, ^{13}CNMR, mass) data.

Keywords *Sida rhombifolia*, Malvaceae, 7-hydroxy-6-methoxycoumarin

Introduction

Sida rhombifolia (Family–Malvaceae) commonly called "Mahabala" is used as traditional medicine in India to cure rheumatism, seminal weakness and diarrhea [1].

Sida rhombifolia is one of the 200 species in Sida genus. It is found in the temperate region and distributed throughout the tropics [2, 3].

Mostly all parts of *Sida rhombifolia* can be used for medical treatment such as the treatment of the stings and bites of scorpion, snake (its flowers), skin diseases and sore (its stem), stomach disorder, stomach pain (Its root), malaria, dysentery (roots) syndrome, gastric, fatigue, diabetes, hemorrhoids, chickenpox, cleaning of blood (its leaves), [3, 4], wounds, headache and migraine headache, gum infection, eye problem, fever, toothache [5], cuts, wound, ophthalmia and swelling [6, 7] tooth brush, wound [8].

In the previous studies have reported phytochemical isolated from this species, including ecydsteroids and their glycosides [9, 10]. The ecdysteroids were, 20-hydroxyecdysone-3-β-D-glucopyranoside, 20-hydroxy ecdysone, pterosterone-3-β-D-glucopyranoside, ecdysone and 20-hydroxy (25-acetyl)ecdysone-3-β-D-glucopyranoside. The detection and characterisation of 20-hydroxy ecdysone was previously reported by Jadhav et al [11] phenyl ethyl-β-D-glucopyranoside [12] Daucosterol [13] and alkaloid consituent such as β-phenyl ethylamine, ephectrine, ψ-ephedrine, quanazoline such as vasicine, vasicinol, vasicinone. Carboxylated tryptamine, such as S-(+) ... Nb-methyl-tryptophan methyl ester. Choline and betanin [14] sterol (β-sitosterol, stigmasterol, campesterol, spinasterol, and cholesterol), n-alkane (eg. nonacosane and hentriacontane) and n-alcohol were reported (all aerial part) from *Sida rhombifolia* [15-17].

Based on the above study, the purpose of this research work is to isolate, and, characterise, the caumarin derivative from the aerial part extract of *Sida rhombifolia* in its eluting solvent.

Result and Discussion

The dichloromethylene extract (CH_2Cl_2) of the aerial part of the *Sida rhombifolia* afforded a novel coumarin derivative, the molecular formula: $C_{10}H_8O_4$, M.P. 202 ºC, molecular ion peak [M^+] at m/z: 192, 177 (M-CH_3), 150 (M-CO), 132 (M-$COCH_2OH$), 126 (M-C_5H_8). It exhibited blue fluorescence. The UV spectrum of methanolic solution of compound have characterics bands at 338.12 nm, 296.52 nm and 226.90 nm, and 339.55 nm, 295.52 nm respectively. This lead to the conclusion that the isolated compound is coumarin derivative [18].

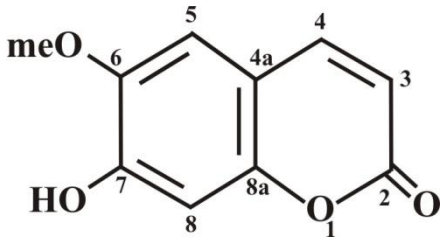
After the addition of 2-4 drops of 2M NaOH in methanolic solution of isolated compound, bathochromic shift was observed which also confirms that isolated compound is coumarin derivative is checked by standard scopoletin.

In Infrared spectrum analysis, the peak was at 3341.44 cm^{-1} which is the result of the –OH alcohol group present. The peak of 2875.05 is the result of the –CH group present. The peak of 1703.42 is the result of the carbonyl C═O group present. The peak of 1606.75 is the result of the alkene CH═CH group present and at the peak of 1568.83, 1511.16 is the result of the benzene ring. The peak at 861.50 is the result of disubstitution in benzene ring [19].

^{1}HNMR spectrum shows two doublet and coupling constant at δ 6.2 and 7.6 ppm as 9.0 Hz, defined as H-3 and H-4 proton respectively. At δ 6.8 and 6.9 ppm shows two singlet aromatic peak defined as H-5 and H-8 proton and δ 3.7 ppm showed methoxy group respectively [20].

The ^{13}C spectrum of the compound shows signal at δ 168.8 (C-2) 121.1 (C-3), 143.3 (C-4), 111.5 (C-4a), 104.2 (C-5), 144.4 (C-6), 152.7 (C-7), 99.2 (C-8), 148.9 (C-8a), 55.4 (OCH_3).

The mass spectra of both standard scopoletin and isolated compound shows M-1 peak at 191.10 which indicate that their molecular weight is same 192.10 so it confirm that isolated compound is 7-hydroxy-6-methoxy coumarin (scopoletin).



7-hydroxy-6-methoxy coumarin (Scopoletin)

Plant Material

The aerial part of Sida rhombifolia were collected from NRIPT (Northern Regional Institute of Printing Technology) Campus Telenganj, Prayagraj and specimen were identified by Dr. B.K. Shukla, Taxonomist, Botanical Survey of India (BSI) Prayagraj (U.P.) India.

Extraction and Isolation

The shed dried and well ground aerial part (4 kg) were refluxed with dichloromethylene (CH_2Cl_2) and the extract was concentrated under reduced pressure through rotatory evaporator. It was partitioned between DCM and ethyl acetate. The ethyl acetate soluble fraction (50 g) was chromatographed over column of silica gel and eluted with binary solution of DCM; $CHCl_3$ in the sequence of increasing polarity. The compound was isolated (petroleum ether : ethyl acetate 7 : 3 v/v) fraction as yellow needle (20 mg). M.P. 202ºC. TLC was performed on coated silica gel 60 F_{254} plates (merk) 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 min.

Experimental Section

M.P. was measured in an open capillary tube and is incorrected. UV was recorded on a Beckmans-DK2 spectophotometer. IR was recorded in KBr on a perkin Elmer spectrometer. ^{1}HNMR of compound was recorded at 500 MHz. ^{13}C NMR spectra at 125 MHz in CD_3OD using TMS as an internal reference on JEOLJNM-4 spectrometers. Mass spectra was recorded on a JEOLJNSD 300 mass spectra meters.

UV $\lambda_{max}^{CHCl_3}$ nm	:	338.12, 296.52, 226.90, 339.55 nm
IR υ_{max}^{KBr} cm^{-1}	:	3341.44, 2875.05, 1703.42, 1606.75, 1568.83, 1511.16, 861.50 cm^{-1}
^{1}H-NMR	:	(500 MHz, CD_3OD) : δ 6.2 (1H, d, J = 9.0 Hz, H-3), 7.6 (1H, d, J= 9.0 Hz, H-4), 6.8 (1H, s, H-5), 6.9 (1H, s, H-8), 3.7 (3H, s, C-6-Ome)
^{13}C-NMR	:	(125 MHz, CD_3OD) : δ 168.8 (C-2), 121.1 (C-3), 143.3 (C-4), 111.5 (C-4a), 104.2 (C-5), 144.4 (C-6), 152.7 (C-7), 99.2 (C-8), 148.9 (C-8a), 55.4 (─OCH_3).
Mass spectra	:	$[M]^+$ 192, 177, 150, 132, 126

Conclusion

From the above spectroscope data, we have conclude that the isolated compound from dichloromethylene (CH_2Cl_2) extract of aerial part of *Sida rhombifolia* is 7-hydroxy-6-methoxy coumarin scopoletin.

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