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ISOLATION AND CHARACTERISATIONOF GLYCOSIDES FROM RHIZOME OF *ACORUS CALAMUS*

Deepa Chauhan*

Department of Chemistry, M.S. College, Saharanpur.

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***Corresponding Author**
Deepa Chauhan
Department of Chemistry,
M.S. College, Saharanpur.

The plant of *Acorus calamus* Linn. (Fam. Araceae), commonly known as "Butch" is a useful medicinal plant found throughout India, Ceylon and Sikkim. In India it is mostly found in marshy tracts of Kashmir and Sirmon in Manipur, and Naga hills.[1]

KEYWORDS: *Acorus calamus*, rhizome, Xanthone glycosides, spectral analysis.

MEDICINAL IMPORTANCE OF *Acorus calamus*

Its rhizome has medicinal properties against bed bugs, moths, lice, emetic stomach in dyspepsia etc. The scented leaves and more strongly scented rhizomes have traditionally been used medicinally and to make fragrances, and the dried and powdered rhizome has been used as a substitute for ginger, cinnamon and nutmeg.

Modern research

Acorus calamus shows neuroprotective effect against stroke and chemically induced neurodegeneration in rats. Specifically, it has protective effect against acrylamide-induced neurotoxicity.

Both roots and leaves of *A. calamus* have shown antioxidant, antimicrobial and insecticidal activities.

Acorus calamus may prove to be an effective control measure against cattle tick, Rhipicephalus (Boophilus) microplus.

A recent study showed that beta-asarone isolated from *Acorus calamus* oil inhibits adipogenesis in 3T3-L1 cells and thus reduces lipid accumulation in fat cells.

Chemical investigation of the rhizome of *Acorus calamus* yields two new xanthone glycosides designated as 4,5,8-terimethoxy-xanthone-2-O-β-D-glucopyranosyl (l→2)-O-β-D-galactopyranoside (A) and1, 8-dihydroxy-3, 7-dimethoxyxanthone-4-O-α-L-rhamnosyl (1→2)-O-β-D-glucopyranoside (B).

The structure of this glycoside has been established on the basis of chemical and spectral evidences.

Compound (A): $C_{28}H_{36}O_{16,}$ mp 270°C. It is on hydrolysis with 7% H_2SO_4 gave galactose and glucose (PC) as sugar moieties and an aglycone which was shown to be a xanthone by its colour reactions and UV spectral data.[3]

The aglycone, $C_{16}H_{14}O_6$, mp 257°C, showed characteristic colour reactions of xanthone. In ^{1}H NMR spectrum four aromatic protons *(meta* and *ortho* coupled) appeared at δ 6.38 (1H, d, *J*=3.0 Hz), 6.52 (1H,d *J*=3.0 Hz), 6.73(1H,d *J*=10 Hz), 7.20 (lH,d *J*=10 Hz). In addition, two singlets corresponding to three protons and six protons appeared at δ 3.87 (3H, s) and δ 3.97 (6H, s) due to three methoxy groups (-OCHs) in which two methoxyl groups were in identical environment. The appearance of four aromatic protons as four different *(ortho-meta)* split doublets were indicative of four oxygen functions (three-OCH_3 and one-OH) distributed in both rings.

Complete acetylation of aglycone with AC_2O in pyridine yielded a monoacetyl derivative showing the presence of one hydroxyl group with three methoxy groups. In ^{1}H NMR spectrum of the acetylated aglycone the downfield shift of meta-coupled aromatic pretons (δ 6.50 1H.d *J*=3.0 Hz and δ 6.90 1H, d *J*=3.0 Hz) indicated the presence of acetylated hydroxyl group and meta-coupled protons in the same ring. The aglycone did not show a bathochromic shift with $AlCl_3$ in UV spectrum indicating the absence of perihydroxyl group, i.e. at C-l, C-8. This shows that hydroxyl group is present at C-2. Absence of C-8 carbonyl deshielded protons indicates that the aglycone was oxygenated at C-8 position.

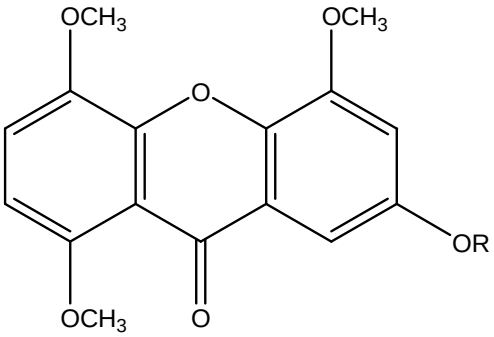
Further, ^{1}H NMR spectrum of the compound showed the presence of three -OCH_3 groups at C-4, C-5 and C-8 δ 3.97 (6H, s for C-4 & C-5) and δ 3.86 (3H, s for C-8) respectively.

The glycoside gave positive Molisch's test[4] but neither reduced Fehling's solution nor gave positive colour test with AHP reagent[5] suggesting the involvement of reducing group in

sugar Linkage. Thus, sugar moiety was attached at C-2. Such as attachment was also confirmed by positive test with Gibb's reagent.

The glycoside showed two anomeric protons at δ 5.78 (lH,d) and 8 4.90 (1H, d) for glucose and galactose, respectively. Enzymatic hydrolysis with p-glucosi-dase confirmed the presence of sugar moieties as bioside and that galactose was directly attached to aglycone with C-O-C linkage and glucose was the external sugar unit.

The glycoside was completely methylated, hydrolysed and the resulting partially methylated sugars were identified as 3,4,6-tri-*O*-methyl-D-galactose and 2,3,4,6-tetra-*O*-methyl-D-glucose. This established that the two sugar units were present in the form of (l→2) bioside. Based on the chemical and spectral evidences, the compound has been assigned the structure as 4,5,8-trimethoxyxanthone-2-O-β-D-glucopyranosyl(l→2)-O-β-D-galactopyranoside **(A).**



R= glucose(1-2)galactosesugar

compound 1

4,5,8-trimethoxy xanthone-2-O-β-D-glucopyranosyl(l-2)-O-β-D-galactopyranoside

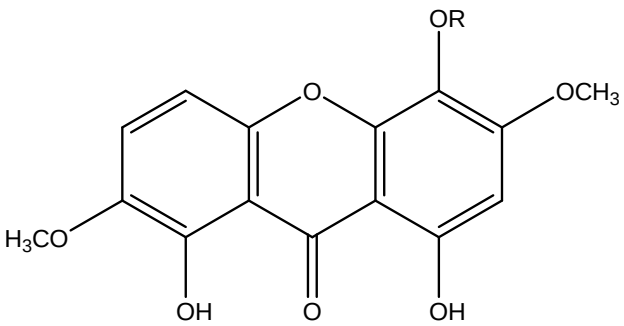
The glycoside was obtained from benzene-DCM (9:1 v/v) fraction and crystallised from ethyl acetate at 10 °C, mp 270 °C; R_f 0.51 (ethyl acetate-methanol; 7:3 v/v); UV (MeOH): 220, 240, 255, 265, 360 nm; IR: 1635, 1620, 1575 on'; 'H NMR (200 MHz, $CDC1_3$): δ 3.5-3.85(12H, br s, sugar protons), 3.86 (3H, s, -OCH_3), 3.97 (6H, s, 2x-OCH_3) 6.38 (IH, d, *J*=3.0 Hz) 6.52 (IH, d, *J* =3.0 Hz), 6.73 (IH, d *J* =10 Hz), 7.20 (IH, d, *J*=10 Hz), 4.90 (IH, d, *J*=5.2 Hz, H-l" galactosyl) 5.78 (IH, d, *J*=7.5 Hz, H-l" glucosyl).

Compound (B): $C_{27}H_{32}O_{16}$, m.p. 217°, was also glycosidic in nature and on hydrolysis with 7% H_2SO_4 gave glucose and rhamnose (PC) as sugar residues and an aglycone which was

shown to be a xanthone by its colour reactions and UV spectral data. The aglycone was characterised as 1, 4, 8-trihydroxy-3, 7-dimethoxyxanthone on the basis of colour reactions and spectral data.

The PMR spectrum of the bioside showed that glucose and rhamnose had the neohesperidoside type linkage which was further confirmed by permethylation of glycoside and identification of permethylated sugars after hydrolysis.

Both the aglycone and the glycoside showed bathochromic shifts in UV spectra with $A1C1_3$ but no shift was observed on addition of NaOAc and $NaOAc/H_3BO_3$. This showed that hydroxyl groups at positions-1 and -8 were free and not involved in the glycosidic linkage. Thus, only position left for the attachment of the sugar moiety was C-4. Such an attachment was also cooijr. med by positive test with Gibb's reagent and UV spectral data. The a-nature of the rhamnose and β-nature of the glucose were confirmed by enzymatic hydrolysis of the glycoside. Based on the chemical and spectral evidences, compound B has been assigned the structure as l, 8-dihydroxy-3, 7-dimethoxyxanthone-4-O- α -L-rhamnosyl (1→2)-O-β-D-glucopyranoside (**B**)



R= rhmnose(1-2)glucose sugar

compound 2

l, 8-dihydroxy-3, 7-dimethoxyxanthone-4-O- α -L-rhamnosyl (1-2)-O-β-D-glucopyranoside

Glycoside (B): Rf 0.31, solvent-ethyl acetate: methanol (8:2, v/v); UV(MeOH): 235, 265, 310 (sh), 341, 400 (sh) nm; $+A1C1_3$: 236, 278, 324, 387 nm; IR(KBr): 1640, 1620, 160p, 1575 cm^{-1}; PMR (90 MHz, $CDC1_3$): 6 1.38 (3H, rhamnose $-CH_3$), 3.50-3.76 (br, 10H, sugar protons), 3.80 (s, 3H, $–OCH_3$), 3.96 (s, 3H, $-OCH_3$), 4.96 (s, IH, H-1" rhamnosyl), 5.24 (bs, IH, H-1" glucosyl), 6.58 (s, IH, H-2), 7.01 (d, J = 9 Hz, IH, H-5), 7.62 (d, J = 9 Hz, IH, H-6), 11.72 (s, IH, C-l OH), 11.77 (s, 1H.C-80H).

Aglycone: UV (MeOH): 237, 269, 309, 343, 400 (sh) nm; +A1C1$_3$: 245, 280, 327, 383 nm; IR(KBr): 1640, 1620, 1600, 1580 cm^{-1}; PMR (90 MHz, CDC1$_3$): δ 3.82 (s, 3H, -OCH$_3$), 3.98 (s, 3H, –OCH$_3$), 6.60 (s, IH, H-2), 7.12 (d, J = 9 Hz, 1H, H-5), 7.65 (d, J = 9 Hz, 1H, H-6); MS: m/z 304 (M$^+$), 289, 275, 259, 231.

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