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Chemical Examination of the Seeds of Trichosanthes anguina Linn

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The present paper deals with the isolation and characterization of kaempferol, quercetin and kaempferol 3-O-β glucoside from the seeds of *Trichosanthes anguina.*

Trichosanthes anguina (family Cucurbitaceae) is commonly known as *chachida* in Hindi. It is cultivated throughout the hotter parts of India. The seeds are considered cooling. It is used as a purgative and vermifuge. It also lessens thirsts increases appetite, act as a tonic and is good for the stomach[1].The present paper deals with the isolation and characterization of Kaempferol quercetin and Kaempferol 3-O-β glucoside from the seeds of *Trichosanthes anguina* which are identified by various chemical degradations and spectroscopic techniques.

The seeds of *T. anguina* Linn. were collected from United Chemicals and Allied, Calcutta. The fresh seeds were then defatted by extraction with pet. ether (40–60°C) and the residue was extracted with MeOH. The combined methanolic extract was concentrated under reduced pressure to give a brown gummy mass which was dissolved in hot H_2O. After cooling it was filtered and the water-insoluble part was dissolved in ether. The water-soluble portion was extracted with Et_2O, EtOAc and MeOH. The methanolic extract of the water-soluble part, on TLC examination over Si-gel (benzene-pyridine-formic acid 36 : 9 : 5) gave three spots. These were separated by CC and assigned TC-1, TC-2 and TC-3.

Compounds TC-1, TC-2 and TC-3 were identified by various chemical and spectral analyses.

TC-1

It was obtained as pale yellow granules. It gave Shinoda test[2], m.pt. 280–281°C. Elemental analysis agreed to the molecular formula $C_{15}H_{10}O_6$. Found C, 62.80 H, 3.44% whereas $C_{15}H_{10}O_6$ requires C, 62.93%, H, 3.49%. ν_{max}^{KBr} cm^{-1}: 3550 (OH), 1675 (C=O), 2945 (C–H), 850 (C=C, aromatic), 1240; UV_{max}; $^{+}$MeOH 249 sh, 265, 290, 370; + $AlCl_3$ 259 sh, 266, 300 sh, 315, 430; + NaOMe 277, 300, 398. On acetylation with Ac_2O/py, it gave a tetraacetate derivative. ^{1}H-NMR (270 MHz, $CDCl_3$): 8.00 (2H, d, J = 8.5 Hz, H-2′,6′), 6.92 (2H, d, J = 8.5 Hz, H-3′,5′), 6.18 (1H, d, J = 2.5 Hz, H-6), 6.44 (1H, d, J = 2.5 Hz, H-8), 2.48 (3H, S, OAc, S), 2.39 (9H, S, OAc-3, 4 : 7). MS data: m/z 286.

On the basis of above data and by m.m.pt. and Co-TLC with authentic sample[3], TC-1 was characterised as kaempferol.

TC-2

It was obtained as yellow powder, which on cystallization with $CHCl_3$–MeOH gave yellow needle-shaped crystals. It gave all tests of flavonoid, m.pt. 280–282°C, molecular formula $C_{15}H_{10}O_7$. Found C, 52.60, H, 3.35, whereas $C_{15}H_{10}O_7$ requires C, 59.62, H, 3.31%. IR^{KBr}_{max} cm^{-1}: 3465 (O-H), 1700 (C=O), 2945 (C–H), UV_{max}: MeOH 258, 302 sh, 370, + $AlCl_3$ 272, 333, 458, + $AlCl_3$–HCl 265, 350, 428, + $AlCl_3$–HCl 265, 350, 428, + NaOAc 258 sh, 322, 390 (Dec), NaOAc–H_3BO_3 261, 380. On acetylation with Ac_2O/py, it gave a pentaacetate derivative. ^{1}H-NMR (270 MHz, $CDCl_3$): 7.67 (1H, d, J = 2.5 Hz, H–2′), 7.60 (1H, d, J = 8.5 H and 2.5 Hz, H–6′), 7.16 (1H, d, J = 8.5 Hz, H–5′), 7.34 (1H, d, J = 2.0 Hz, H–8), 6.86 (1H, d, J = 20 Hz, H–6), 2.47 (3H, S, OAc–5), 2.40 (3H, S, OAc-7), 2.35 (9H, 3, OAc-3,4′,3′). MS data: m/z M^+ 302. It was characterized as quercetin by comparison of its spectral data and co-chromatography with authentic sample[4].

TC-3

It was obtained as yellow needles on crystallization with MeOH, m.pt. 176-178°C. It gave Shinoda test[2], Molish test and showed high solubility in water[5]. Elemental analysis agreed to the molecular formula $C_{21}H_{20}O_{11}$. Found C, 56.35, H, 4.40% of whereas $C_{21}H_{20}O_{11}$ requires C, 56.25, H, 4.46%. $IR\nu^{KBr}_{max}$ cm^{-1}. 3420 (OH), 1650 (C=O), 1110–1020 (C–O, gly), 850 (aromatic C=C). $UV\lambda^{MeOH}_{max}$ MeOH 244, 265, 350. λ^{MeOH}_{max} + NaOAc 265, 348, 398. λ^{MeOH}_{max} + $AlCl_3$ 255, 301, 354. λ^{MeOH}_{max} + $AlCl_3$–HCl 274, 298 sh, 398, + NaOMe 246, 249, 350 sh, 380. UV spectral data gave a bathochromic shift 44 nm in band I and 19 nm in band II with $AlCl_3$/HCl, in comparison with $AlCl_3$. It means C-3 position in the above glycoside is blocked. On acetylation it gave a heptaacetate derivative. ^{1}H-NMR ($CDCl_3$, 90 MHz): 8.07 (2H, d, J = 9 Hz, H-2′,6′), 7.26 (2H, d, J = 9 Hz, H–3′,5′), 7.30 (1H, d, J = 2.5 Hz,H–8), 6.79 (1H, d, J = 2.5 Hz, H–6), 5.51 (1H, d, J = 9 Hz glu, aromatic proton), 2.47 (3H, S, OAc–5), 2.35 (3H, OS, OAc–7), 2.32 (3H, S, OAc, 4′), 1.72, 2.15 (12H, M, OAc, Sugar). MS data: m/z 618 M^+, 286 aglycone moiety$^+$; 337 acetylated hexopyranoside$^+$, 153 $A_1 + H^{++}$, 121 B_1^+. Acid hydrolysis of glycoside with 7% HCl gave an equimolar quantity of aglycone, m.pt. 280–281°C, which was characterized as kaempferol by direct comparison with anthentic sample[3]. The sugar was identified as glucose by Co–PC (R_f 0.21, 0.31 (EtOAC-Py-H_2O 12 : 5 : 4) and B : A : W 6 : 1 : 2). Methylation of glycoside (CH_3I/Ag_2O/DMF) followed by acid hydrolysis gave 3-OH,5,7,4′-Trimethoxy flavone[6], m.pt. 135–36°, (Cal. for $C_{18}H_{16}O_6$: C, 65.00, H, 4.87%; found: C, 65.90, H, 4.88% and 2,3,4,6-tetra-O-methoxy-D-glucose. This confirmed the location of glucose in the glycoside at C-3 position. The enzymatic hydrolysis with almonds emulsion confirmed the presence of sugar as glucose as well as β-linkage between sugar and aglycone.

Thus it was identified as Kaempferol 3-O-β-glucoside.

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