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A new constituent from prunus cerasoides

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ABSTRACT KEYWORDS

The present study deals the isolation of a new compound from fruits of Prunus *cerasoides* and characterized as 2,4,4'-dihydroxy-6-methoxy chalcone-4-O-[β -D-glycopyranosyl ($1\rightarrow4$)] α -L-rhamnopyranoside by NMR and MS data. © 2007 Trade Science Inc. - INDIA

Prunus cerasoides; Chalcones; α-L-rhamnopyranoside; Column chromatography.

INTRODUCTION

Prunus cerasoides is a tree distributed in temperate Himalaya to an altitude of 1700m^[1,2]. The plant is reported to be antipyretic, refrigerant and useful in asthma, leprosy and leucoderma^[1,2]. The stem bark has been investigated and is found to contain sakuranetin, prunetin, genestein and genkwanin^[3]. We have shown that the sapwood of plant contain some flavonoids^[4] and flavanone glucoside^[5]. This paper describes the isolation and characterization of a chalcone glycoside (1) from the methanolic extract of the fruits of Prunus cerasoides.

EXPERIMENTAL

Material and Method

The Fruits of Prunus cerasoides were collected from Kamand (Tehri,Uttrakhand India) and authenticated by Dr.P.Uniyal, Department of Botany,Garhwal university campus, .Badhshahithaul, Tehri. A voucher specimen (No-121) has been deposited in the herbarium of the department.

Extraction and isolation

Dried and powdered fruits were soxhlet-extracted with MeOH. The extract was concentrated under reduced pressure afforded a light yellow solid mass. Whicsh on repeated column chromatography(CHCl₂-MeOH) gave compound1 (110mg). Column chromatography was carried out on Kiesel gel (230-240mesh MERK), TLC was performed on Kiesel gel 60 GC (Merk). The melting point is uncorrected. UV spectra(MeOH) were obtained Hitachi 320 Spectrophotometer. IR spectra were recorded on KBr discs (JASCO-IR-810 specrophotometer). The ¹H-NMR and ¹³C-NMR spectra were obtained on a JEOLJNM-MH200 at 300MHz and JNM-FXFOO at 100MHz in DMSO, D₆ and CDCl₂ using TMS as internal standard (Chemical shift in δppm). Mass spectra (70 ev JEOL-JMS-DX300 spectrometer) were taken with a direct inlet.

Acid hydrolysis of 1

Compound 1(15mg) was hydrolysed with 7% H_2SO_4 in MeOH at 100°C for 2hr to afford a yellow compound identified as 2, 4, 4'- trihydroxy-6-methoxy chalcone. The filtrate from the hydrolysate was neutralized with Ag_2CO_3 and filtered, the filtrate was concentrated under reduced pressure and the residue tested

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Compound 1

for the presence of D.glucose and L-rhmnose (PC, solvent EtOAc-pyridine-H₂O, 10:4:3 values 0.23 and 0.42 respectively).

Partial hydrolysis of 1

Compound 1(15mg) was hydrolysed with 1% MeOH- H₂SO₄ to afford another compound. The residue obtained after neutralization and concentration of hydrolysate showed the presence of rhamnose (PC, Rf 0.42).

Compound 1:Crystallised from MeOH as yellow granules (110mg), mp 188-190°C, UV(MeOH) 245sh(log ε 4.25) and 364nm(4.42). IR 3400(br), 2775 and 1685 cm-1. 1 H-NMR δ7.80(1H, d, J=16Hz), 7.75(2H, m), 7.60(1H, d, J=16Hz), 7.00(2H, m), 6.50 (1H, br, s), 6.35 (1H, br, s), 3.25-5.30(12H, m) 8.0, 7.80, 7.60(Phenolic–OH, s) 13 C-NMR: 114.5(C-1)164.4(C-2), 99.7(C-3), 166.4 (C-4), 95.9 (C-5), 163.1(C-6), 144.6(C-α), 126.4(C-β), 9.4(C-1'), 132.6(C-2'), 115.0(C-3'), (C-4'), 115.4(C-5'), 131.7(C-6'), (C-O), 55.0 (OMe), Suger carbons glc1" to 6": 102.4, 74.8,75.0, 62.7, 78.4, 58.9, rham 1"' to 6"'', 102.2,70.2, 81.7,77.7, 70.0, 17.9, FAB-MS (m/z), 594 (M+), 287,168 and 120.

RESULTS AND DISCUSSION

Repeated column chromatography of the methanolic extract of the fruits gave a compound (1). It gave a positive ${\rm FeCl}_3$ test but a negative Shinoda test suggesting the absence of a flavone or an isoflavone nucleus^[6]. Its UV specrum showed absorption bands characteristic of chalcone^[7]. The IR spectrum showed characteristic absorption at 3400(br) for a polyhydroxy system, 2775 for a methoxy group and 1645cm⁻¹ for a conjugated carbonyl group, Acid hydrolysis of compound1 gave an aglycone ${\rm C_{16}H_{14}O_5}$, which on acety-

lation with Ac_2O / pyridine, gave triacetyl derivative, m.p. $173\text{-}174^0\mathrm{C}$, $C_{26}H_{21}O_8$. The presence of one methoxy group was determined with the help of Zeisels method^[8]. The ¹H-NMR spectrum of 1 showed a methoxy group signal at $\delta 3.72$ (br, s), a typical doublet for 4-oxygenated a ring ($\delta 7.25$, $\delta .80$, J= 9Hz each) three one- proton signal at 8.0, 7.80, 7.60 (exchangeable with D_2O) attributed to phenolic OH. The presence of AB quartet (J=16 Hz) for -HC=CH protons at 7.60-7.80, which is characteristic of ArCH=CH-CO-Ar system in chalcone^[9] confirmed that 1 was a chalcone glycoside. The existence of a methoxy group on the ring was confirmed by the presence of ions at m/z 286, 168 and 120 in the EIMS of aglycone^[10].

The proton multiplets between $\delta 3.4-4.0$ represent the position of suger protons, whereas a singlet at 0.8 and 4.0 showed the position of α - linked rhamnose. The appearance of doublet at 5.8(d, J=6.8Hz) further confirmed the position of β - linked anomeric proton of D-glucose. The attachment of sugar unit at C-4 was apparent from the UV spectrum of 1 which showed a bathochromic shift of 50nm with NaOMe. The ¹³C-NMR spectrum showed twenty carbon peaks resonated at aromatic and carbohydrate region. The downfield peak at 165.0 confirmed the presence of carbonyl function, whereas the peak at 165.0 (C-4), 163.1(C-6), 164.4(C-2), 161.0(C-4') substitution at these positions. The appearance of peak at $\delta 55.0$ was assigned for the methoxy carbon atom. FAB-MS of 1 showed a molecular ion peak at m/z 599 and significant peaks at m/z 287 due to loss of one glucose and one rhmnose units, m/z 168 and 120 due to cleavage of the aglycone in to two halves. Partial acidic hydrolysis (1% MeOH- H₂SO₄) showed the loss of rhmnose (co-pc, co-TLC). This further confirmed the linear attachement of sugar with aglycone. Thus the structure of aglycone was elucidated as 2,4,4'- trihydroxy-6-methoxy chalcone and thus that of 1, a new glycoside, 2,4'-dihydroxy-

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6-methoxy chalcone -4-O-[β-D glycopyranosyl(1 \rightarrow 4)] α-L- rhamnopyranoside.

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