

NEW FLAVANONE TRIGLYCOSIDE FROM *MORINGA OLEFERA*

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ABSTRACT

From the methanolic extract of the stem of *Moringa olefera*, a new flavanone triglycoside has been isolated and elucidated as 7–O–(β –D–glucopyranosyl)–5–O–methyl naringenin–4'–[α –L–rhamnopyranosyl–(1 \rightarrow 2)]– β –D–glucopyranoside by NMR and Mass data.

Key words: Flavonone, Moringe olefera, Triglycoside

INTRODUCTION

Moringa olefera (Family Moringaceae) is a tree and used in paralytic affections, intermittent fever and epilepsy¹. A literature search revealed that no phytochemical work has been reported on stem of this plant, although few flavonoid glycosides have been isolated from its seeds^{2,3}. This paper describes the isolation and the characterization of a new flavanone triglycoside from the methanolic extract of the stem *of Moringa olefera*.

EXPERIMENTAL

General experimental procedures

The melting point was determined on a Gallenkamp melting point apparatus and is uncorrected IR spectra were recorded on Perkin-Elmer double beam spectrophotometer 580B in KBr. The ¹H NMR and ¹³C NMR were recorded on a Bruker AMX 400 spectrometer in CD₃OD with TMS as internal standard. Mass spectra was recorded on a 70 eV JEOL JMS-DX 300 spectrometer with a divert inlet. Column chromatography silica gel (Merck 60-120 mesh), TLC; Kiesel gel 60 G (Merck). The spot on TLC were visualized by spray with 5 % alcoholic FeCl₃.

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Plant material

The plant material was collected from Painula (New Tehri, Uttarakhand), India. The authentication of plant material was made at the Botany Department, Garhwal University Campus, Badshahithaul, Tehri.

Extraction and isolation

The plant material was defatted with light petroleum ether $(60-80^{0})$ followed by exhaustive extraction with MeOH. The extract was concentrated under reduced pressure to afford a light solid mass. This solid mass was subjected to column chromatography (CHCl₃ : MeOH). Several known flavonoids and flavonoids glycoside were isolated and identified^{4,5}. The last fraction, which was obtained by eluting the column with CHCl₃-MeOH (30 : 70) afforded colourless crystals of **(1)** (150 mg).

Characterization of compound 1

Crystallized from MeOH as colourless crystals, m.p. 216-218 0 C; UV (MeOH); 234(log ϵ 3.97), 224 nm (4.1); IR ν_{max} cm⁻¹ 3420, 2775, 1710, 1380, 1210; ¹H NMR (CD₃OD); δ 3.75 (3H, br s, OMe), δ 2.70 (2H, m, AB, H-3), 5.35 (1H, dd, X of ABX, H-2), 5.1, 5.3, 5.5 (1H, each, d, anomeric sugar carbons), 6.35, 6.50 (1H, br s, H-6, H-8), 6.50, 7.00 (1H, br s, H-3', H-5' and H-2' and H-6'), 2.25 - 5.5 (m, glycosyl protons), ¹³C NMR (CD₃OD) is given in Table 1.

Aglycone carbon		Sugar carbon	
		4'-O-	
		Glucose	
C-2	77.8	C-1"	101.1
C-3	42.0	C-2"	75.3
C-4	192.2	C-3"	77.3
C-5	160.5	C-4"	73.9
C-6	95.6	C-5"	77.5
C-7	166.7	C-6"	62.1
			Cont

Table 1. ¹³C NMR (CD₃OD) Data (1)

Aglycone carbon		Sugar ca	Sugar carbon	
C-8	95.0	Rhamnose		
C-9	162.7	C-1"'	104.4	
C-10	104.7	C-2"'	72.1	
C-1'	130.0	C-3"'	81.7	
C-2'	128.6	C-4"'	78.4	
C-3'	113.3	C-5"'	70.0	
C-4'	159.4	C-6"'	17.9	
C-5'	113.3	7°-O-		
C-6'	128.4	Glucose		
O-CH ₃	55.5	C - 1""	101.7	
		C - 2""	76.3	
		C -3""	78.47	
		C-4""	71.3	
		C-5""	78.3	
		C-6""	62.6	
OR				OR
	0		-0, 0, 1", 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0, 0,	OR glc (1f
RO OR	 OMe	0	1""	OR
glc (1	62)			
		1 : R = H		OR
		2 : R = Ac	OR rhan	n (146)

Acid hydrolysis of compound (1)

Compound (1) (3 mg) was heated with aq. 10 % HC1 (2 mL) in a sealed tube at $100 \,{}^{0}$ C for 4 hr. The aglycone was extracted with ether and identified by TLC analysis with authentic samples using CHCl₃-Me₂CO (50 : 6). The aqueous layer was neutralized with Ag₂CO₃ and dried. This concentrated aqueous hydrolysate showed the presence of D-

glucose and L-rhamnose [Paper chromatography EtOAC- $C_5H_5N-H_2O$ (10 : 4 : 30)], R_f values are 0.23 and 0.42, respectively.

RESULTS AND DISCUSSION

Compound (1), molecular formula $C_{34}H_{44}O_{19}$ (EI MS); m.p. 216-218 °C gave positive Shinoda test⁶ suggesting the presence of a flavone or flavanone. Its UV spectrum was typical of flavone or flavanone⁷. The IR spectrum showed peaks at 3420 (br, polyhydroxy system), 2775 (methoxy group) and 1710 cm⁻¹ (carboxyl group).

Acid hydrolysis of (1) afforded an aglycone. D-glucose and L-rhamnose in the ratio⁸ of 2 : 1. The aglycone vielded a diacetate on acetvlation, m.p. 172-174 ^oC. The ¹H NMR spectrum of aglycone showed a signal for a methoxy group at 3.75 (s), a typical double doublet for 4'-oxygenated β -ring at δ 7.25, 6.80 (J = 9.0 Hz each), two-metacoupled doublets at δ 5.83, 6.03 (J = 2.2 Hz each) and a typical ABX pattern of protons at δ 2.70 (m) and 5.35 (1H, dd, J = 5 Hz) similar to that of naringenin-5-methyl ether ⁹. The existence of a methoxy group on the A ring was confirmed by the presence of ions at m/z286. 167 and 120 in the EIMS (1). The ¹³C NMR spectrum (1) (Table 1) showed signals at δ 101.1 (anomeric glucose C-1"), 104.4 (anomeric rhamnose C-1"), 101.6 (anomeric glucose C-1""). On acetylation with $Ac_2O/pyridine$ (1) gave a deca-acetate (2) (δ 1.95-2.45). The ¹H NMR spectrum showed three anomeric protons at δ 5.1, 5.5 and 5.3, which appeared as doublets each (J = 3 Hz). Thus, on the basis of hydrolytic and spectral data (1) was 5-methoxy naringenin triglycoside. The sugar moieties of which consist of two Dglucose units which has β linkages with the aglycone and rhamnose (δ 1.76 due to secondary methyl), linked with a glucose unit at α position¹². The attachment of sugar units at C-7 and C-4', respectively on the aglycone was apparent from UV spectrum (1) which showed a bathochromic shift of 50 nm in the presence of NaOMe without increase in intensity. The ¹H NMR spectrum (1) showed singlets for glycosyl protons at 3.23 - 5.5 (m), two broad singlets at δ 6.35, 6.50 for C-6, C-8 protons, two broad signals each at 5 6.50, 7.00 for C-3', C-5' and C-2', C-6' protons. FAB-MS (1) showed a molecule ion peak at m/z756; 708.6, 286 (M⁺-Sugar units), 167 and 120 due to cleavage of the aglycone. Based on the above evidence, the structure (1) was elucidated as 7-O-(B-D-glucopyranosyl)-5-Omethyl naringenin- 4'- $[\alpha$ -L-rhamnosyl-(1 \rightarrow 2)]- β -D-glucopyranoside.

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