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Quinovic acid glycosides from *Mussaenda pilosissima* Valeton

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Abstract

Four known quinovic acid glycosides, quinovic acid 28-*O*-*β*-D-glucopyranosyl ester (**1**), 3-*O*-*α*-L-rhamnopyranosylquinovic acid 28-*O*-*β*-D-glucopyranosyl ester (glycoside A, **2**), and 3-*O*-*β*-D-glucopyranosylquinovic acid 28-*O*-*β*-D-glucopyranosyl ester (glycoside B, **3**), 3-*O*-*α*-L-rhamnopyranosylquinovic acid 28-*O*-*β*-D-glucopyranosyl ester (**4**) were isolated from the aerial parts of *Mussaenda pilosissima* Valeton. Their chemical structures were determined using NMR spectra as well as in comparison with the reported data. Compounds **1**, **2**, and **4** were reported for the first time from *Mussaenda* genus.

Keywords. *Mussaenda pilosissima*, quinovic acid glycoside, ursane.

1. INTRODUCTION

The genus *Mussaenda* comprises about 200 species belonging to the Rubiaceae family.[1] In Vietnam, there are about 27 species of genus *Mussaenda*.[2] Of which, there are 11 species have been used as folk medicines.[3] The chemical studies of *Mussaenda* genus indicated the presence of iridoids, triterpenoids, and flavonoids. These compounds have shown the potential significant biological effects as anti-inflammatory, antioxidant, and anticancer activities.[4] *Mussaenda pilosissima* has been used for the treatment of kidney diseases and blood in urine.[5] Up to now, the chemical constituents of this plant have not been studied yet. Herein, we report the isolation and structural elucidation of four quinovic acid glycosides from the aerial parts *Mussaenda pilosissima.*

2. MATERIAL AND METHODS

2.1. Plant Material

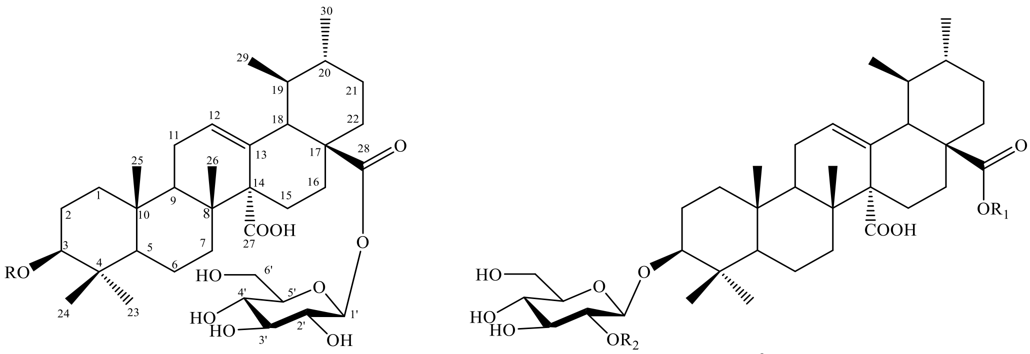
The aerial parts of *Mussaenda pilosissima* Valeton. were collected at Me Linh, Vinh Phuc, Vietnam in February 2017 and identified by Dr. Nguyen The Cuong, Institute of Ecology and Biological Resources, VAST. A voucher specimen (NCCT-P69) was deposited at the Institute of Marine Biochemistry, VAST.

2.2. General experimental procedures

All NMR spectra were recorded on a Bruker AM500 FT-NMR spectrometer (500 MHz for ^{1}H-NMR and 125 MHz for ^{13}C-NMR). NMR measurements, including ^{1}H-, ^{13}C-NMR, HSQC, and HMBC experiments, were carried out using 5-mm probe tubes at temperature of 22.2 ^{o}C. ESI mass spectra were recorded on an Agilent 6530 Accurate-Mass Q-TOF LC/MS system. Column chromatography was performed using a silica gel (Kieselgel 60, 70-230 mesh and 230-400 mesh, Merck) or RP-18 resins (150 µm, Fuji Silysia Chemical Ltd.), thin layer chromatography (TLC) using a pre-coated silica-gel 60 F_{254} (0.25 mm, Merck) and RP-18 F_{254S} plates (0.25 mm, Merck).

2.3. Extraction and isolation

The dried powder of aerial parts of *M. pilosissima* Valeton. (4.2 kg) was sonicated 3 times with hot methanol. The extract was filtered through filter paper, then solvent was removed under reduced pressure to yield 320 g of a dark solid extract. The extract was suspended in water and successively partitioned with *n*-hexane, dichloromethane, ethyl acetate giving *n*-hexane (MPA1 56.8 g), dichloromethane (MPA2 97.8 g), ethyl acetate extracts (MPA3 23 g) and water layer (MPA4).



1 R = H
2 R = Rha

Rha: rhamnopyranosyl
Glc: glucopyranosyl

3 R_1 = Glc R_2 = H
4 R_1 = H R_2 = Glc

Figure 1: Chemical structures of compounds **1-4** from *M. pilosissima*

The MPA3 extract was chromatographed on a silica gel column eluting with dichloromethane: methanol (100:0 → 0:1, v/v) to give 8 subfractions, MPA3A-MPA3H. MPA3F was chromatographed on a RP-18 column eluting with methanol/water (1/1.5, v/v) to give eight smaller fractions, MPA3F1-MPA3F8. Compound **1** (17.0 mg) was yielded from MPA3F2 fraction using a silica gel column eluting with dichloromethane/acetone/water (1/2.5/0.15, v/v/v). MPA3F6 fraction was chromatographed on a silica gel column eluting with dichloromethane/acetone/water (1/2.5/0.15, v/v/v) to give two fractions, MPA3F6A and MPA3F6B. MPA3F6B was chromatographed on a RP18 column eluting with acetone/water (1/1.3, v/v) to yield **2** (30.0 mg). MPA3F8 was continued to fractionate on a RP18 column eluting with acetone/water (1/1.3, v/v) to give two fractions, MPA3F8A and MPA3F8B. MPA3F8A was chromatographed on a silica gel column eluting with dichloromethane/acetone/water (1/2.5/0.15, v/v/v) to yield **3** (10.0 mg). MPA3H was chromatographed on a silica gel column to give three smaller fractions, MPA3H1-MPA3H3. Fraction MPA3H3 was chromatographed on a RP-18 column eluting with acetone/water (1/1.3, v/v) to obtain **4** (25.0 mg).

Quinovic acid 28-*O*-*β*-D-glucopyranosyl ester (**1**): White amorphous powder; $[\alpha]_D^{25}$ + 60.0 (*c* 0.1, MeOH); ESI-MS *m/z* 649 $[M+H]^+$, $C_{36}H_{56}O_{10}$, M = 648; ^{1}H- and ^{13}C-NMR (CD_3OD), see table 1.

3-*O*-[α-L-rhamnopyranosyl]-quinovic acid-28-*O*-[*β*-D-glucopyranosyl] ester (**glycoside A, 2**): White amorphous powder; $[\alpha]_D^{25}$ + 15.0 (*c* 0.1, MeOH); ESI-MS *m/z* 795 $[M+H]^+$, $C_{42}H_{66}O_{14}$, M = 794; ^{1}H- and ^{13}C-NMR (CD_3OD), see table 1.

3-*O*-[*β*-D-glucopyranosyl]-quinovic acid-28-*O*-[*β*-D-glucopyranosyl] ester (glycoside B, 3): White amorphous powder; $[\alpha]_D^{25}$ + 30.0 (*c* 0.1, MeOH); ESI-MS *m/z* 811 $[M+H]^+$, $C_{42}H_{66}O_{15}$, M = 810; ^{1}H- and ^{13}C-NMR (CD_3OD), see table 2.

Quinovic acid 3-*O*-*β*-D-glucopyranosyl-(1→2)-*β*-D-glucopyranoside (**4**): White amorphous powder; $[\alpha]_D^{25}$ + 82.0 (*c* 0.1, MeOH); ESI-MS *m/z* 811 $[M+H]^+$, $C_{42}H_{66}O_{15}$, M = 810; ^{1}H- and ^{13}C-NMR (CD_3OD), see table 2.

3. RESULTS AND DISCUSSION

Compound **1** was obtained as a white amorphous powder. The ^{1}H-NMR spectrum of **1** showed the following signals: one olefin proton at δ_H 5.64 (br s); six methyl groups at δ_H 0.78 (3H, s), 0.90 (3H, s), 0.95 (3H, s), 0.99 (3H, s), 0.92 (3H, d, J = 6.0 Hz), and 0.94 (3H, d, J = 6.0 Hz), suggesting the presence of the ursane aglycone. In addition, one anomeric proton at δ_H 5.39 (d, J = 8.0 Hz) suggested the appearance of a sugar moiety. The ^{13}C-NMR spectrum of **1** revealed the signals of 36 carbons, including eight non-protonated carbons at δ_C 38.2, 40.3, 40.8, 49.9, 57.4, 133.4, 178.0, and 179.0; twelve methines at δ_C 38.1, 40.0, 48.1, 55.4, 56.6, 71.3, 73.9, 78.3, 78.6, 79.7, 95.7, and 130.9; ten methylenes at δ_C 19.5, 23.9, 25.9, 26.5, 27.9, 31.2, 37.0, 38.1, 39.8, and 62.6; and six methyl carbons at δ_C 16.4, 16.9, 18.1, 19.2, 21.3, and 28.7. The ^{1}H-, ^{13}C-NMR data (table 1) and ESI-MS were found to match with those of quinovic acid-28-*O*-*β*-D-glucopyranosyl ester.[3] The positions of the functional groups were confirmed by using HSQC and HMBC spectra. The HMBC correlations from H-23 (δ_H 0.95)/H-24 (δ_H 0.78) to C-3 (δ_C 79.7)/C-4 (δ_C 40.3)/C-5 (δ_C 56.6) were indicated the positions of oxygenated group at C-3, two methyl groups at C-4. The multiplicity of the oxygenated proton at δ_H 3.13 (dd, J = 4.5, 11.0 Hz) indicated the *β*-configuration of oxygenated group at C-3. The HMBC correlations from H-25 (δ_H 0.99) to C-1 (δ_C 39.8)/C-5 (δ_C 56.6)/C-9 (δ_C 48.1)/C-10 (δ_C 38.2); from H-26 (δ_H 0.90) to C-7 (δ_C 38.1)/C-8 (δ_C 40.8)/C-9 (δ_C 48.1)/C-14 (δ_C 57.4) supported for the

Table 1: NMR data for compounds **1**, **2** and reference compounds

C	**1**			**2**		
	δ_C*	δ_C[a]	δ_H (mult., *J* in Hz)	δ_C#	δ_C[a]	δ_H (mult., *J* in Hz)
1	39.8	39.8	1.05 (m)/1.71 (m)	39.1	39.8	1.05 (m)/1.73 (m)
2	27.9	27.9	1.59 (m)/1.65 (m)	26.1	26.7	1.75 (m)/1.95 (m)
3	79.7	79.7	3.13 (dd, 4.5, 11.0)	88.2	90.4	3.07 (dd, 4.5. 11.0)
4	40.2	40.3	-	36.5	40.0	-
5	55.8	56.6	0.74 (d, 11.5)	55.6	56.7	0.77 (d, 11.5)
6	18.9	19.5	1.36 (m)/1.53 (m)	18.5	19.4	1.37 (m)/1.52 (m)
7	37.6	38.1	1.22 (m)/1.65 (m)	37.5	37.9	1.23 (m)/1.66 (m)
8	40.8	40.8	-	38.9	40.8	-
9	47.4	48.1	2.25 (dd, 5.5, 11.5)	47.3	48.0	2.25 (dd, 5.5, 11.5)
10	37.4	38.2	-	40.2	38.0	-
11	23.5	23.9	1.94 (m)/1.96 (m)	23.4	23.9	1.94 (m)/1.97 (m)
12	129.6	130.9	5.64 (br s)	129.6	130.9	5.64 (br s)
13	133.3	133.4	-	133.3	133.3	-
14	56.0	57.4	-	56.8	57.4	-
15	26.2	25.9	1.74 (m)/2.06 (m)	25.5	25.8	1.77 (m)/2.06 (m)
16	25.2	26.5	1.77 (m)/2.11 (m)	26.0	26.5	1.78 (m)/2.10 (m)
17	49.0	49.9	-	49.0	49.8	-
18	54.7	55.4	2.30 (d, 11.0)	54.7	55.3	2.31 (d, 11.0)
19	39.1	40.0	0.94 (m)	37.6	40.3	0.95 (m)
20	37.5	38.1	1.04 (m)	39.1	38.3	1.04 (m)
21	30.3	31.2	1.28 (m)/1.47 (m)	30.3	31.2	1.30 (m)/1.48 (m)
22	36.5	37.0	1.63 (m)/1.72 (m)	37.0	37.0	1.62 (m)/1.73 (m)
23	28.6	28.7	0.95 (s)	28.1	28.7	0.94 (s)
24	16.6	16.4	0.78 (s)	16.8	17.0	0.81 (s)
25	16.7	16.9	0.99 (s)	16.6	16.9	1.00 (s)
26	18.2	18.1	0.90 (s)	19.2	19.2	0.91 (s)
27	178.1	179.0	-	178.0	179.0	-
28	176.6	178.0	-	176.5	178.0	-
29	19.3	19.2	0.92 (d, 6.0)	18.1	18.1	0.93 (d, 6.0)
30	21.2	21.3	0.94 (d, 6.0)	21.2	21.5	0.95 (d, 6.0)
		28-*O*-Glc		**3-*O*-Rha**		
1′	95.7	95.6	5.39 (d, 8.0)	104.2	104.4	4.73 (br s)
2′	74.2	73.9	3.33 (m)	72.5	72.5	3.84 (brd, 2.5)
3′	78.0	78.3	3.42 (t, 9.0)	72.9	72.5	3.65 (dd, 2.5, 9.0)
4′	71.2	71.3	3.36 (m)	74.1	74.1	3.40 (m)
5′	78.9	78.6	3.37 (m)	69.8	69.9	3.72 (m)
6′	62.5	62.6	3.69 (dd, 5.5, 12.0) 3.82 (dd, 2.0, 12.0)	18.7	17.8	1.25 (d, 6.5)
				28-*O*-Glc		
1″				95.7	95.6	5.40 (d, 8.0)
2″				74.2	74.0	3.34 (m)
3″				79.0	78.3	3.42 (t, 8.0)
4″				71.3	71.2	3.36 (m)
5″				79.3	78.6	3.37 (m)
6″				62.4	62.6	3.68 (dd, 5.5, 12.0) 3.82 (dd, 2.0, 12.0)

[a)] Measured in CD_3OD, *δ_C of quinovic acid 28-*O*-*β*-D-glucopyranosyl ester in pyridine-d_5 [3], #δ_C of 3-*O*-*α*-L-rhamnopyranosylquinovic acid 28-*O*-*β*-D-glucopyranosyl ester in pyridine-d_5 [5].

location of two methyl groups at C-8 and C-10. The position of double bond at C-12/C-13 was confirmed by the HMBC correlations from H-12 (δ_H 5.64) to C-9 (δ_C 48.1)/C-14 (δ_C 57.4)/C-18 (δ_C 55.4). The HMBC correlations from H-29 (δ_H 0.92) to C-18 (δ_C 55.4)/C-19 (δ_C 40.4)/C-20 (δ_C 38.1); from H-30 (δ_H 0.94) to C-19 (δ_C 40.4)/C-20 (δ_C 38.1)/C-21 (δ_C 31.2) supported for the location of two methyl groups at C-19 and C-20. Thus, the aglycone of **1** was assigned as an ursane-12-ene skeleton. The HMBC correlations from H-15 (δ_H 2.06/1.74) to C-27 (δ_C 179.0); from H-16 (δ_H 2.11/1.77)/H-18 to C-28 (δ_C 178.0) indicated the presence of two carboxylic groups at C-14 and C-17. The ^{13}C-NMR

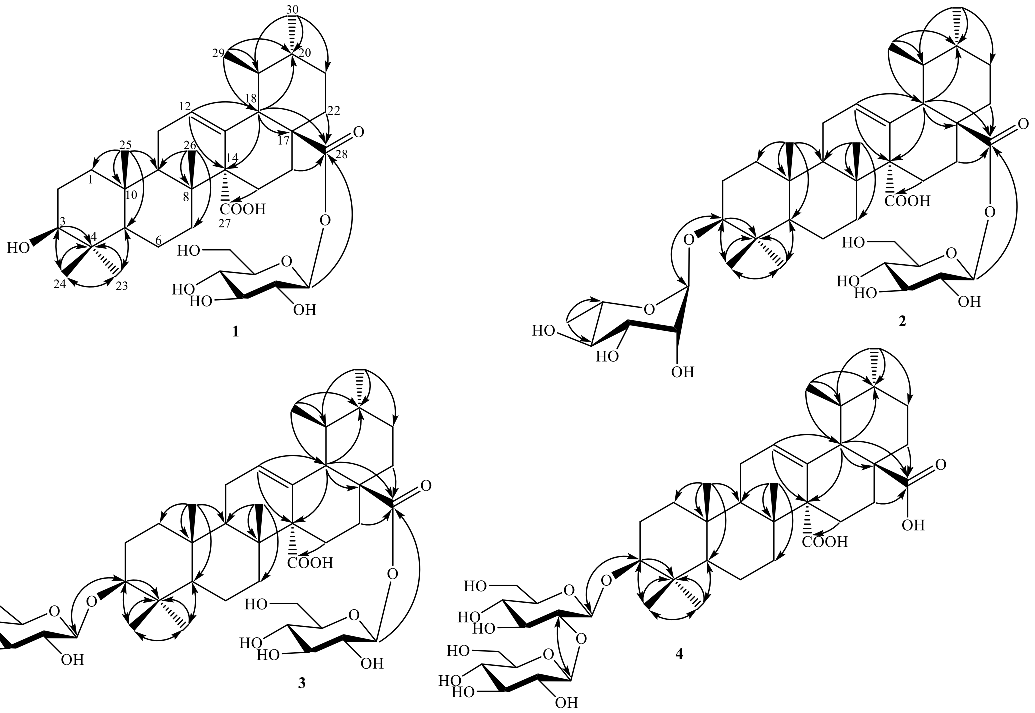


Figure 2: The key HMBC correlations of compounds **1-4**

data (δ_C 95.6, 73.9, 78.3, 71.3, 78.6, and 62.6) and the multiplicity of glc H-1′ [δ_H 5.39 (d, J = 8.0 Hz)] suggested the sugar moiety as *β*-D-glucopyranosyl. In addition, the sugar position at C-28 of aglycone was confirmed by the HMBC correlation between H-1′ (δ_H 5.39) and C-28 (δ_C 178.0). Consequently, the structure of **1** was determined to be quinovic acid 28-*O*-*β*-D-glucopyranosyl ester. Compound **1** was reported from *Mussaenda* genus for the first time.

Compound **2** was isolated as a white amorphous powder. The ^{1}H-NMR spectrum of **2** exhibited the resonant signals: one olefin proton at δ_H 5.64 (br s); four singlet methyl signals (δ_H 0.81, 0.91, 0.94, and 1.00); three doublet methyl signals [δ_H 0.93 (3H, d, J = 6.0 Hz), 0.95 (3H, d, J = 6.0 Hz), and 1.25 (3H, d, J = 6.5 Hz)]; two anomeric protons [δ_H 4.73 (br s), and 5.40 (d, J = 8.0 Hz)]. The ^{13}C-NMR of **2** revealed the signals of 42 carbons, including eight non-protonated carbons at δ_C 38.0, 40.0, 40.8, 49.8, 57.4, 133.3, 178.0, and 179.0; seventeen methines at δ_C 38.4, 40.3, 48.0, 55.3, 56.7, 69.9, 71.2, 72.5×2, 74.0, 74.1, 78.3, 78.6, 90.4, 95.6, 104.4, and 130.9; ten methylenes at δ_C 19.4, 23.9, 25.8, 26.5, 26.7, 31.2, 37.0, 37.9, 39.8, and 62.6; and seven methyl carbons at δ_C 16.9, 17.0, 17.8, 18.1, 19.2, 21.5, and 28.7. Thus, compound **2** were found to be a ursane-12-ene glycoside.[5] The ^{1}H- and ^{13}C-NMR data of **2** were similar to those of **1** except for an addition of one sugar moiety. In addition, this sugar moiety with ^{13}C-NMR data (δ_C 104.4, 72.5, 72.5, 74.1, 69.9 and 17.8) and multiplicity of H-1′ [δ_H 4.73 (br s)] suggested the presence of *α*-L-rhamnopyranosyl moiety. The *α*-L-rhamnopyranosyl position at C-3 of aglycone was confirmed by the HMBC correlation from H-1′ (δ_H 4.73) to C-3 (δ_C 90.4). Based on the above evidence and comparison the NMR and ESI-MS data of **2** to those reported in the literature,[5] the structure of **2** was elucidated to be 3-*O*-*α*-L-rhamnopyranosylquinovic acid 28-*O*-*β*-D-glucopyranosyl ester (glycoside A). Compound **2** was also reported from *Mussaenda* genus for the first time.

The ^{1}H and ^{13}C NMR spectra of **3** (tTable 2) showed similar with those of **1** except for an addition of sugar moiety. The sugar moieties in compound **3** were confirmed to be two *β*-D-

Table 2: NMR data for compounds **3**, **4** and reference compounds

C		3			4	
	δ_C*	δ_C^{a}	δ_H (mult., *J* in Hz)	δ_C#	δ_C^{a}	δ_H (mult., *J* in Hz)
1	39.0	39.3	1.04 (m)/1.70 (m)	40.0	40.0	1.07 (m)/1.71 (m)
2	26.8	27.0	1.71 (m)/1.94 (m)	26.5	27.2	1.75 (m)/1.98 (m)
3	88.7	90.7	3.16 (dd, 4.5, 11.0)	91.4	91.4	3.19 (dd, 4.5, 11.0)
4	36.4	40.1	-	40.4	40.3	-
5	55.8	56.9	0.76 (d, 11.5)	56.9	56.9	0.77 (d, 11.5)
6	18.6	19.3	1.35 (m)/1.54 (m)	19.3	19.3	1.36 (m)/1.55 (m)
7	37.5	38.0	1.22 (m)/1.65 (m)	37.7	37.7	1.24 (m)/1.65 (m)
8	39.8	40.8	-	40.8	40.7	-
9	47.2	48.0	2.23 (dd, 5.5, 11.0)	48.0	48.0	2.25 (dd, 5.5, 11.0)
10	37.5	37.8	-	38.1	38.0	-
11	23.4	23.9	1.94 (m)/1.96 (m)	23.9	23.9	1.92 (m)/1.99 (m)
12	129.6	130.9	5.64 (br s)	130.3	130.2	5.62 (br s)
13	133.2	133.2	-	133.5	134.1	-
14	56.8	57.3	-	57.3	57.4	-
15	25.5	25.8	1.76 (m)/2.06 (m)	26.4	25.8	1.77 (m)/2.06 (m)
16	26.0	26.4	1.77 (m)/2.10 (m)	25.8	26.6	1.68 (m)/2.05 (m)
17	49.0	49.8	-	49.5	49.6	-
18	54.7	55.3	2.31 (d, 11.0)	55.6	55.6	2.28 (d, 11.0)
19	40.2	40.2	0.99 (m)	40.0	40.4	1.00 (m)
20	39.7	38.2	1.03 (m)	38.4	38.4	1.02 (m)
21	30.3	31.1	1.29 (m)/1.47 (m)	31.3	31.3	1.28 (m)/1.45 (m)
22	37.0	37.0	1.61 (m)/1.72 (m)	37.0	37.8	1.62 (m)/1.66 (m)
23	28.0	28.5	1.04 (s)	28.5	28.4	1.07 (s)
24	17.0	17.1	0.85 (s)	19.1	17.0	0.86 (s)
25	16.6	16.9	0.99 (s)	16.9	16.9	1.00 (s)
26	19.2	19.2	0.90 (s)	18.2	19.1	0.92 (s)
27	178.0	179.1	-	182.0	179.2	-
28	176.5	178.0	-	179.2	179.2	-
29	18.1	18.1	0.92 (d, 6.0)	17.0	18.2	0.92 (d, 6.0)
30	21.2	21.5	0.94 (d, 6.0)	21.5	21.5	0.94 (d, 6.0)
3-*O*- Glc				**3-*O*- Glc**		
1′	106.0	106.6	4.33 (d, 7.5)	104.5	105.4	4.44 (d, 7.5)
2′	75.8	75.6	3.21 (dd, 7.5, 9.0)	81.1	81.1	3.59 (m)
3′	78.9	77.6	3.26 (m)	78.5	78.5	3.58 (m)
4′	71.3	71.2	3.31 (m)	71.9	71.9	3.40 (m)
5′	78.2	78.2	3.34 (m)	77.7	77.7	3.28 (m)
6′	63.1	62.5	3.69 (dd, 5.5, 12.0	63.1	63.1	3.64 (dd, 5.5, 12.0)
			3.85 (dd, 2.0, 12.0)			3.84 (dd, 2.0, 12.0)
28-*O*-Glc				**2′-*O*-Glc**		
1″	95.7	95.6	5.40 (d, 8.0)	105.4	104.5	4.69 (d, 8.0)
2″	74.2	73.9	3.34 (m)	76.3	76.3	3.24 (m)
3″	79.3	78.2	3.43 (t, 9.0)	78.4	77.9	3.37 (m)
4″	71.9	71.6	3.36 (m)	71.6	71.6	3.30 (m)
5″	78.7	78.5	3.37 (m)	77.9	78.3	3.25 (m)
6″	62.4	62.8	3.69 (dd, 5.5, 12.0)	62.9	62.8	3.67 (dd, 5.5, 12.0)
			3.82 (dd, 2.0, 12.0)			3.86 (dd, 2.0, 12.0)

a) Measured in CD_3OD; *δ_C of 3-*O*-*β*-D-glucopyranosylquinovic acid-277.98-*O*-*β*-D-glucopyranosyl ester in pyridine-d_5 [5], #δ_C of quinovic acid-3-*O*-*β*-D-glucopyranosyl(1→2)-*β*-D-glucopyranoside in CD_3OD.[6]

glucopyranosyl by ^{13}C-NMR data (δ_C 106.6, 78.2, 77.6, 75.6, 71.6, 62.8; 95.6, 78.5, 78.2, 73.9, 71.2, 62.5) and multiplicity of H-1′ [δ_H 4.33 (d, J=7.5)] and H-1″ [δ_H 5.40 (d, J = 8.0)]. Furthermore, the positions of sugar moieties at C-3 and C-28 were confirmed by the HMBC correlations from H-1′ (δ_H 4.33) to C-3 (δ_C 90.7); from H-1″ (δ_H 5.40) to C-28 (δ_C 178.0). Based on tthe NMR and ESI-MS data analysis, and comparison the NMR data of **3** to those reported in the literature, the structure of compound **3** was elucidated to be 3-*O*-*β*-D-glucopyranosylquinovic acid 28-*O*-*β*-D-glucopyranosyl ester (glycoside B).[4] This compound was reported by Zhao and co-authors from the *Mussaenda pubescens*.[7]

The ^{1}H- and ^{13}C-NMR data of compound **4** were similar to those of **3** (table 2). The difference in structure between **4** and **3** is the position of *β*-D-glucopyranosyl moieties. The sugar linkage of **4** was confirmed as *β*-D-glucopyranosyl-(1→2)-*β*-D-glucopyranoside by the observation of the HMBC correlation between glc H-1″ (δ_H 4.69) and glc C-2′ (δ_C 81.1). In addition, this sugar at C-3 of aglycone was proved by the HMBC correlation from glc H-1′ (δ_H 4.44) to C-3 (δ_C 91.4). Moreover, careful analysis and comparison the NMR and ESI-MS data of **4** with those reported in the literature,[6] the structure of **4** was determined to be quinovic acid 3-*O*-*β*-D-glucopyranosyl(1→2)-*β*-D-glucopyranoside. Compound **4** was reported for the first time from *Mussaenda* genus.

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