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Two novel resin glycosides isolated from *Ipomoea cairica* with α -glucosidase inhibitory activity

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[ABSTRACT] In the present study, two new compounds from *Ipomoea cairica* were identified and demonstrated to have α -glucosidase inhibitory activity. They were isolated by column chromatography on silica gel and sephadex LH-20 and finally purified by prep-HPLC, with their structures being elucidated by spectroscopic methods, such as 1D- and 2D-NMR and HR-TOF-MS, and chemical methods. Compounds 1 and 2, named cairicoside A and cairicoside B, were evaluated for α -glucosidase inhibitory activity by the MTT method, with the IC₅₀ values being 25.3 ± 1.6 and 28.5 ± 3.3 µmol·L⁻¹, respectively.

[KEY WORDS] *Ipomoea cairica*; Resin glycoside; Simonic acid A; α-Glucosidase

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Introduction

The structure of resin glycosides is composed with a long chain fatty acid aglycone and oligosaccharide, and some modifiable fatty acids are acylated in different sites in the oligosaccharide ^[1]. Many different kinds of resin glycosides have been found from genera of the family Convolvulaceae, including *Ipomoea* ^[2], *Operculina* ^[3], *Quamoclit* ^[4], and *Merremia* ^[5]. Various activities have been reported, including cytotoxicity^[5] and inhibition of multidrug efflux pumps ^[6].

Ipomoea. cairica (L.) Sweet (Convolvulaceae) is an invasive species widely distributed from tropical to subtropical regions, and used as a folk medicine all over the world ^[7-9]. Several kinds of compounds have been found. Some resin glycosides have been found in *I. cairica* ^[10]. In order to find other different lactone site of the resin glycosides in *I. cairica*, as a part of our ongoing chemical studies on the resin glycosides from *Ipomoea* species, a chemical investigation of *I. cairica* has been undertaking in our laboratory. Some plants from convolvulaceae family are

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shown to have significant inhibition against α -glucosidase or anti-diabetic activity ^[11-12]. In this paper, we present the results from elaboration of the structures of two new compounds and the evaluation of their α -glucosidase inhibitory activities.

Results and Discussion

Compound 1 was obtained as a white, amorphous powder, and the molecular formula $C_{69}H_{110}NaO_{26}$ as determined with HR-TOF/MS at m/z [M + Na]⁺ 1 377.786 6 (Calcd. as 1 377.718 3) $[M + Na]^+$. Its IR spectrum gave peaks of hydroxyl $(3 424 \text{ cm}^{-1})$, carbonyl (1 737 cm⁻¹), and aromatic (1 640 cm⁻¹) groups. Alkaline hydrolysis of compound 1 afforded simonic acid A (compound 3) and organic acid fractions, which were identified as isobutyric acid methyl ester ($t_{\rm R} = 3.60 \text{ min}$) m/z101 [M]⁺ (28), 88 (100), 57(70), 41 (35), *n*-decanoic acid $(t_{\rm R} = 12.37 \text{ min}): m/z \ 172 \ [M]^+ (4), \ 155 \ (5), \ 143 \ (30), \ 129 \ (5),$ 87 (59), 74 (100), 55 (18), and transcinnamic acid methyl ester ($t_{\rm R}$ =13.29 min) m/z 162 [M]⁺ (40), 131 (100), 103 (66), 77 (32) by GC-MS experiment. Acid hydrolysis of compound 3 afforded the monosaccharides mixture, which was further derivatized and detected with GC-MS experiment. In the acid hydrolysate of operculinic acid A, methyl ester L-rhamnose, and D-glucose were confirmed by comparison of their retention times of their derivatives with those of authentic L-rhamnose ($t_R = 30.14 \text{ min}$) and D-glucose ($t_R = 31.65 \text{ min}$) derivatives prepared in the same way, respectively.

The ¹H data of **1** (Table 1) exhibited two *trans*-coupled olefinic protons at $\delta_{\rm H} 6.54$ (d, J = 16.0 Hz, H-2 of Cna) and 7.81 (d, J = 16.0 Hz, H-3 of Cna), and a multiplet due to five



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	Position -	1		2	
	Position -	δ_{c}	δ _H	δ _c	$\delta_{\rm H}$
Glc	1	104.3	4.98 d (7.2)	104.3	4.92 d (7.2)
	2	81.8	3.87*	81.8	3.88*
	3	76.3	4.17*	76.3	4.18*
	4	71.3	4.19 dd (9.0, 9.0)	71.4	4.13 dd (9.0, 9.0)
	5	77.8	3.90*	77.8	3.84*
			4.51-4.53*		4.41*
	6	62.6	4.29*	62.6	4.32*
Rha	1	99.0	5.65 br s	98.5	5.58 br s
	2	73.4	6.13 br s	71.6	6.13 br s
	3	69.6	5.14 dd (9.6, 3.0)	69.5	5.07 dd (9.6, 3.0)
	4	78.2	4.28*	79.2	4.23*
	5	68.1	4.47*	68.1	4.32*
	6	19.2	1.56 d (6.0)	19.2	1.59 d (6.0)
Rha' Rha''	1	98.5	6.20 br s	99.0	6.19 br s
	2	73.0	6.09 br s	72.4	6.02 br s
	3	79.3	4.71 dd (9.0, 2.4)	79.7	4.65 dd (10.0, 2.4)
	4	80.2	4.27*	80.2	4.21*
	5	68.1	4.30*	68.0	4.48 [*]
	6	18.4	1.64 d (6.0)	18.4	1.64 d (6.0)
	1	103.4	6.04 br s	103.3	5.97 br s
	2	69.8	5.00 br s	72.3	4.77 br s
	3	73.3	5.95 dd (3.0, 10.0)	73.0	5.89 dd (3.0, 10.0)
	4	73.4	6.11 t (10.0)		
	5	68.5	4.56*	73.4 68.5	6.13 dd (10.0, 10.0) 4.30*
	6	17.6		17.6	1.43 d (6.0)
			1.36 d (6.5)		
Rha	1	104.3	5.70 br s	104.4	5.63 br s
	2	72.4	4.85 br s	69.8	4.95 br s
	3	72.3	4.54*	72.3	4.47*
	4	73.4	4.26 [*]	73.2	4.19 [*]
	5	70.6	4.37*	70.5	4.41*
	6	18.6	1.58 d (6.0)	18.7	1.61 d (6.0)
Ag	1	173.2		173.0	
	2	34.2	2.44 m; 2.34 m	34.3	2.40, m; 2.28, m
	11	82.6	3.90*	82.6	3.90*
	16	14.0	0.88 t (7.0)	14.0	0.84 t (7.0)
Cna	1	166.2		166.2	
	2	118.1	6.54 d (16.0)	118.1	6.61 d (16.0)
	3	145.3	7.81 d (16.0)	145.3	7.87 d (16.0)
	1′	134.4		134.5	
	2' and 6'	128.4	7.43 m	128.4	7.50 m
	3' and 5'	129.1	7.33 m	129.0	7.38 m
	4'	130.6	7.33 m	130.6	7.38 m
Deca	1	172.7		172.7	
	2	34.2	2.47 m	34.0	2.30 m
	12	14.0	0.85 t (7.0)	14.0	0.80 t (7.0)
Iba	1	176.2			
	2	34.0	2.67 m		
	3	19.0	1.16 d (7.0)		
	3'	18.7	1.15 d (7.0)		
Hexa	1			173.2	
	2			34.3	2.43 m
	6			13.8	0.67 t (7.0)

 Table 1
 ¹³C and ¹H NMR data for compounds 1 and 2 in Pyridine-d₅

Chemical shifts marked with an asterisk (^{*}) indicate overlapped signals and all assignments are based on ¹H-¹H TOCSY experiments; Abbreviations: Glc: glucose; Rha: rhamnose; Ag: 11-hydroxyhexadecanoyl; Cna: *trans*-cinnamoyl; Deca: *n*-decanoyl; Hexa: *n*-hexanoyl; Iba: isobutyryl



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